

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JULY 9, 1926.

I.—GENERAL; PLANT; MACHINERY.

Influence of rate of stirring on reaction velocity. F. C. HUBER and E. E. REID (Ind. Eng. Chem., 1926, 18, 535—537).—The rate of reaction is approximately a linear function of the rate of stirring in reactions such as the ethylation of benzene by ethylene, the oxidation of sodium arsenite by oxygen, etc., where the reaction of the dissolved gas is rapid but the solution of the gas is slow, and therefore affected by the stirring. Reactions such as the catalytic hydrogenation of cottonseed oil or a hydrocarbon are approximately linear functions of the rate of stirring when a certain rate has been reached; in this case, a certain rate of stirring is necessary to ensure uniform distribution of the catalyst and thus promote hydrogenation. Reactions such as the saponification of ethyl benzoate or the hydrolysis of benzyl chloride not affected by the rate of stirring.

B. W. CLARKE.

PATENTS.

Furnaces for heating at low temperatures. F. PUENING (E.P. 228,148, 14.1.25. Conv., 23.1.24).—A large quantity of gas is reciprocated over the goods to be heated by an apparatus comprising a water-sealed bell moving up and down within a similarly shaped container; at each stroke the reciprocating gas acquires slight increments of heat by admixture with products of combustion, similar amounts of excess gas being exhausted to atmosphere. One bell-piston may serve several heating chambers, in which case the stroke of the bell is lessened when heating chambers are cut out for repairs.

B. M. VENABLES.

Kiln. H. F. GRUETZEMACHER, JUN., Assr. to GLENCOE LIME & CEMENT Co. (U.S.P. 1,580,483, 13.4.26. Appl., 8.10.23).—A combustion chamber adjacent to the lower part of the kiln is provided with means for supplying fuel and air thereto and for preheating the air. The air is passed through a heating chamber within the walls of the kiln above the combustion chamber and thence into the combustion chamber through a conduit external to the kiln. Means are provided for controlling the velocity of circulation of the air and for otherwise controlling its temperature.

H. HOLMES.

Refractory brick for furnace linings. O. ARAM (U.S.P. 1,576,021, 9.3.26. Appl., 14.9.22).—A surface coating, consisting largely of metallic aluminium, is applied to exposed surfaces of the refractory lining, the durability of which is thereby prolonged.

F. SALT.

Absorption refrigerating apparatus. SIEMENS-SCHUCKERTWERKE G.M.B.H. (E.P. 229,332, 13.2.25. Conv., 14.2.24).—In an absorption refrigerating system the difference in pressure between the generator side and the absorption side of the apparatus is utilised by means of an injector to cause movement of the gas in the low-pressure side from the evaporator to the absorber. This permits a wider choice of inert carrier gas; for example, air at about atmospheric pressure may be used for this purpose, with the advantage that small leaks are comparatively harmless.

B. M. VENABLES.

Evaporating process and apparatus. Soc. GÉN. D'ÉVAPORATION PROC. PRACHE & BOUILLON (E.P. 242,259, 16.10.25. Conv., 31.10.24).—The liquid to be evaporated is heated and evaporated by passing counter-current in contact with a stream of cold air, the bulk of the liquor being returned through an indirect heat interchanger and preheater to the evaporator. Fresh make-up liquor is added to the return circuit, and the liquor when sufficiently strong is drawn off from the bottom of the evaporator. The warm moist air from the evaporator is propelled by a fan to another contact apparatus where it gives up its sensible and latent heat to a counter-current stream of cold water, the air returning to the evaporator and excess water being drawn off. The heat thus given to the water is transferred back again to the liquor in the above-mentioned heat interchanger, and additional heat to compensate for losses and to provide for the temperature drops is given to the liquor in the preheater, which is heated by live steam or other external means. In the case of liquors which do not deposit crystals the evaporator may consist of a contact tower filled with coke or other filling; if crystals are deposited during evaporation the air may be passed over the surface of the liquor in a vat; and if the material to be evaporated is pasty, the air may be passed through the interior of a rotating drum, the exterior surface of which picks up the material to be evaporated or dried. To transfer the heat of condensation back to the liquor, instead of a simple water circuit, apparatus similar to a refrigerating apparatus, comprising vaporiser, compressor, and condenser, may be used, the necessary external energy being supplied as power to the compressor.

B. M. VENABLES.

Apparatus for heating and boiling liquids. BLAIR, CAMPBELL & MCLEAN, LTD., and R. WEBSTER (E.P. 250,762, 25.4.25).—In apparatus for heating liquids by steam, comprising a number of units of twin concentric pipes forming annular chambers for

the steam, the units are arranged to be held together and to the steam supply header solely by means of a nut on the inner tube. B. M. VENABLES.

Multiple-effect evaporative separation. H. K. MOORE, Assr. to BROWN Co (U.S.P. 1,582,066-7, 27.4.26. Appl., [A] 16.10.22, [B] 30.11.23).—(A) In a multiple-effect evaporating process the liquor flows from an intermediate effect through progressively hotter effects and then through effects that are progressively cooler. (B) Liquor which would become viscous if completely concentrated in the cooler effects, is supplied hot to a heat interchanger and cooled to the temperature of an intermediate effect and is passed through that and a series of cooler effects. From the coolest effect the liquor is reheated to the temperature of the effect next hotter than the starting point by means of the heat interchanger, and then passes through a series of hotter effects, being heated while passing to each of these effects by heat taken from the liquor leaving the hottest effect of the series. B. M. VENABLES.

Evaporator. J. A. PAUL (U.S.P. 1,580,323, 13.4.26. Appl., 29.4.21).—The open end of a cylindrical evaporator drum is closed by a cylindrical curved spring metal cover in such a way that only the edges are adapted to yield when the internal pressure exceeds a predetermined amount. H. HOLMES.

Heat treatment of materials for the extraction of volatile constituents or moisture therefrom. W. R. HUME (E.P. 250,302, 29.12.24).—The material is fed to the interior of a rotating drum against the walls of which it is held by centrifugal force. The drum is subjected to external heat and if desired is also heated internally by steam or electricity. Various devices for feed inlets, product outlets, and liquid seals are described. B. M. VENABLES.

Rotary drums for cooling or otherwise treating materials by air currents. J. S. FASTING (E.P. 250,318, 8.1.25).—A rotary drum, e.g., a cooler connected to a rotary cement kiln, is provided with metal cylinders, rings, bands, plates, or the like, having a large surface relative to their weight, connected to the inner surface of the drum by chains permitting a limited amount of free movement, and arranged within the drum to attain the maximum amount of transference of heat from air to the material or *vice versa*. B. W. CLARKE.

Measurement of [flow of] heat. A. BLACKIE (E.P. 250,416, 29.5.25).—The apparatus comprises a heat-receiving vessel containing air or other fluid connected to a capillary orifice and a pressure gauge or manometer. The rate of flow of heat into the vessel is calculated from the reading of the manometer when equilibrium is reached between the expansion of the air and its outflow through the orifice. The face of the vessel which receives the heat is preferably blackened and the other faces may be protected by vacuum or water jackets. The vessel itself may form its own pressure gauge on the aneroid principle. B. M. VENABLES.

Tubular mills for grinding or crushing. O. BOUZIN (E.P. 250,504, 28.12.25).—A grinding mill of the type where a number of separate cylindrical grinding chambers rotate about a common axis, is provided with feed and outlet chambers common to all the cylinders, the feed chamber being provided with a scoop for each cylinder. B. M. VENABLES.

[Gyratory] crushers. ALLIS-CHALMERS MANUF. Co., and R. C. NEWHOUSE (E.P. 250,749, 3.4.25).—In a gyratory crusher of the type which has a conical crushing head and shaft, depending from a universal bearing at the top and oscillated by an eccentric at the bottom, the eccentric is driven at high speed direct from a motor at the top of the apparatus, through a rotating shaft within the hollow gyrating shaft. The gyrations are so rapid that the pieces of material being crushed never have time to come to rest. To prevent harmful effects of vibrations the whole crusher and motor is suspended freely by means of three ropes. B. M. VENABLES.

Pulveriser mill. A. KELLER (U.S.P. 1,580,610, 13.4.26. Appl., 25.1.24).—A grinding ball within a grinding ring co-acts with a pusher member having a constantly shifting and rolling contact with the ball. H. HOLMES.

Pulveriser. T. L. MCKAIN (U.S.P. 1,580,620, 13.4.26. Appl., 24.5.22).—A cylindrical drum is provided with rotary beaters mounted axially and extending close to but not in contact with its inner surface. The lower part of the lower half of this surface is perforated, the remaining portions of the lower half are smooth and non-perforated, and the whole of the upper half is corrugated to provide longitudinal ribs. H. HOLMES.

Pulverising mill. F. I. RAYMOND, Assr. to RAYMOND BROS. IMPACT PULVERIZER Co. (U.S.P. 1,582,538, 27.4.26. Appl. 25.6.23).—A pulverising mill of the ring and roller or similar type is provided with an inlet for material above the grinding zone, and air is admitted below the grinding zone in such a manner that it first moves outwards and then upwards and inwards, passing through the grinding zone and separating the material as it falls therefrom. B. M. VENABLES.

Pulverising mill. C. B. STRACHAN, Assr. to STRACHAN TUBE MILL Co. (U.S.P. 1,583,324, 4.5.26. Appl., 25.5.25).—The grinding elements comprise a number of cylinders of different diameter assembled loosely and coaxially within a rotating cylindrical casing. Cylindrical spaces for entry and exit of material are left between the ends of the mill and perforated inner diaphragms which serve to retain the grinding cylinders in place. B. M. VENABLES.

Carrying out chemical reactions. E. BUHTZ (E.P. 250,722, 26.2.25).—The substances which are to react together are liquefied and supplied, preferably through concentric conduits, to a substantially horizontal disc rotating at a low speed. The disc may be dished or funnel-shaped, may be provided with corrugations or rings to increase the surface and

provide strong agitation, and may work in conjunction with a smaller disc which may also rotate.

B. M. VENABLES.

Controlling operations of solvent-recovery plants. G. G. OBERFELL (U.S.P. 1,580,157, 13.4.26. Appl., 18.4.25).—The vapour pressure of a liquid mixture is determined by bringing a definite volume of air into contact with the mixture and measuring the increase of volume of the air due to its saturation with the vapour.

H. HOLMES.

Drying apparatus. G. J. PEZOLD (U.S.P. 1,580,164, 13.4.26. Appl., 28.1.24).—The cylindrical wall of a rotary drum is provided on its opposite ends with flanges projecting inwards, and hollow trunnions are arranged on heads secured to the flanges. An inner shell, spaced from the wall, is secured at its opposite ends to the flanges, and the space between the shell and the wall constitutes a heating chamber. Transverse partitions, spaced from the heads, are secured within the shell, and the space between one partition and the adjacent head forms a conduit for the heating fluid which passes into the heating chamber through perforations in the adjacent part of the shell.

H. HOLMES.

Rotary drying apparatus. C. MILLER (U.S.P. 1,581,359, 20.4.26. Appl., 7.3.25).—A rotating vertical cylindrical container is supplied with air from a fan at the bottom, the stream of air being divided by a perforated plate into several currents. The air is exhausted through several outlets at the top.

B. M. VENABLES.

Anti-freezing solution. E. H. CROFOOT, Assr. to LARROWE CONSTRUCTION Co. (U.S.P. 1,581,179, 20.4.26. Appl., 16.1.20. Renewed 18.9.25).—An anti-freezing solution for radiators consists of the waste liquor from sugar extraction processes.

A. R. POWELL.

Separation of liquid mixtures by distillation. E. MERCK (E.P. 244,036, 29.4.25. Conv., 8.12.24).—See F.P. 595,821; B., 1926, 176.

Annular furnace with revolving hearth for continuous working. L. HONIGMANN (U.S.P. 1,583,974, 11.5.26. Appl., 20.5.24).—See E.P. 237,376; B., 1925, 742.

Separation of the solid constituents of liquids containing them. H. N. MCLEOD (U.S.P. 1,585,300, 18.5.26. Appl., 28.6.24).—See E.P. 248,081; B., 1926, 392.

Means for reducing air currents in centrifugal machines. K. J. SVENSSON and K. A. P. NORLING (U.S.P. 1,584,488, 11.5.26. Appl., 3.8.23).—See E.P. 202,616; B., 1924, 736.

Drying process and apparatus. T. RIGBY (U.S.P. 1,585,511, 18.5.26. Appl., 13.2.22).—See E.P. 182,157; B., 1922, 800 A.

Manufacture of porous masses for storing gases. GAS ACCUMULATOR Co. (UNITED KINGDOM), LTD. From AUTOGEN GASACCUMULATOR A.-G. (E.P. 242,241, 8.9.25).—See G.P. 420,150; B., 1926, 230.

Means for supplying heated air to furnaces and the like. A. CROWCROFT (E.P. 251,318, 14.8.25).

[Regulating supply of air to] furnaces. E. ROUCKA (E.P. 228,924, 6.2.25. Conv., 7.2.24).

Heat exchange apparatus. R. SELIGMAN (E.P. 251,064, 13.3.25).

[Device for cleaning] centrifugal separators. DE LAVAL CHADBURN Co., LTD., and A. C. BAMFORD (E.P. 251,738, 18.3.25).

Vacuum-producing apparatus. [Water-jet gas ejector.] G. SOKOLOW-WICHNEVSKY (E.P. 251,858, 12.10.25).

Centrifugal separator (E.P. 250,319).—See II.

Crucible furnaces (E.P. 238,562).—See VIII.

Electrical gas cleaning (E.P. 250,499).—See XI.

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

Dependence of the form of the flame in a combustion flue on various factors. P. STOLLER (Gas-u. Wasserf., 1926, 69, 374–378).—The influence of (1) form of air nozzle, (2) increased draught, (3) gas velocity and form of burner orifice, and (4) amount of excess air on the form of flame in a vertical combustion chamber, 3.7 m. high and 0.1 m.² cross-section, has been studied by varying one factor at a time and determining the temperature distribution in the chamber and regenerator. A rectangular air nozzle gives a more regular temperature distribution than a "whirling" nozzle, but a somewhat lower temperature in the combustion chamber and a higher temperature in the regenerator. Increased draught lowers the maximum temperature and moves its position nearer to the base of the chamber. A more regular temperature distribution is attained by varying the cross-section of the gas and air apertures so that the gas velocity and air velocity are nearly the same. At the same time a rise of temperature occurs in the regenerator, and the efficiency of the combustion is lowered. With increasing excess of air the height of the flame decreases.

A. B. MANNING.

Gases and the carbonisation of fuels. P. LEBEAU (Bull. Soc. chim. Belg., 1926, 35, 47–74).—Extended researches on the gases liberated on the heat treatment of various fuels at temperatures up to 1000° are summarised. Starch, certain sugars, casein, and amygdalin were also examined (cf. B., 1923, 877 A, 964 A; 1924, 206, 585, 700, 775; 1925, 88, 744).

W. CLARK.

Active charcoal. H. CHAUMAT (La Nature, 1926, 149–155; Chem. Zentr., 1926, I., 2891).—The absorptive power of charcoal is greater the freer it is from certain hydrogen compounds; it is greater also, when comparing different absorbed substances, the greater the heat effect associated with the absorption. The absorption is not in

accord with Henry's law. Activation is produced by treatment with sulphuric acid, zinc chloride, or phosphoric acid, of which the last is the most efficient. The phosphorus formed on heating the charcoal to 600° with phosphoric acid reacts with the hydrogen compounds, producing hydrogen phosphide. The excess of phosphoric acid is removed by washing, the charcoal dried, and ignited at 1200°; it is then washed with hydrochloric acid and finally dried at 300°. The charcoal so treated contains only 0.2 instead of 4.5% H, and can absorb 65–70% of its weight of benzene.

A. B. MANNING.

Determination of the porosity of coke, refractory bricks, and rammed moulding sand.

H. ESSER and E. PIVOVARSKI (*Stahl u. Eisen*, 1926, 46, 565–567).—An apparatus for the rapid determination of the true and apparent volume of a porous body is described. The principle of the method is similar to that of Washburn and Bunting (*B.*, 1922, 710A; cf. Pressler, *B.*, 1924, 381). The air in a glass container is rarefied by sucking a portion into a measuring burette, and after introducing the sample the operation is repeated to the same diminished pressure in the container. The difference between the quantities of air measured in the burette in the two operations multiplied by the quotient, barometric pressure/pressure in container after withdrawal of air, gives the volume of the substance. The apparent volume is found in the same apparatus by the mercury displacement method. From these figures the porosity is calculated as usual.

A. R. POWELL.

Liquid purification of coal gas and the recovery of sulphur. C. HARNIST (*Chim. et Ind.*, 1926, 15, 506–513; cf. Berthelot, *B.*, 1926, 36).—The American, Claus, Burkheiser, Feld, and other processes for the purification of coal gas and the recovery of sulphur therefrom are summarised and critically compared. The American process, in which the hydrogen sulphide is absorbed in a solution of sodium carbonate which is regenerated by the passage of a current of air, makes recovery of the sulphur difficult owing to the very low concentration of hydrogen sulphide in the air current. The modification of the process in which the alkaline solution contains suspended ferric oxide, is unsuccessful in practice owing to the difficulty of dealing with the precipitated material. The recovery of sulphur by the Claus process is not yet a commercial success. Although the Feld process, in which the hydrogen sulphide and ammonium sulphide are oxidised by sulphur dioxide to a mixture of polythionates and sulphur, the polythionates being subsequently converted into ammonium sulphate, has yet to overcome many practical difficulties, it appears to be the most promising as a basis for future developments.

A. B. MANNING.

Determination of paraffin hydrocarbons. W. STEUER (*Gas- u. Wasserf.*, 1926, 69, 417–419).—An improved method of determining methane and ethane consists in burning the hydrocarbons mixed

with oxygen over red-hot platinum or palladium. The methane-ethane mixture is measured in one Hempel burette, the oxygen in a second. The two burettes are connected by 10 cm. of transparent quartz capillary tubing containing 3 cm. of platinum or palladium wire. The gases are mixed by passing the oxygen into the other burette, the quartz tube and contained catalyst are heated by a full Bunsen flame, and the mixture is passed over the catalyst slowly in order to avoid explosion. The catalyst is then raised to a bright red heat, and the gas mixture passed over it repeatedly until combustion is complete. The amounts of methane and ethane are calculated from the total contraction and the carbon dioxide formed. Palladium induces combination more rapidly and at a lower temperature than platinum. Ethane burns at a lower temperature than methane. For this method, rapidity, accuracy, and facility of operation are claimed.

R. A. A. TAYLOR.

Ultra-violet spectroscopy of flames of motor fuels. G. L. CLARK and W. C. THEE (*Ind. Eng. Chem.*, 1926, 18, 528–531).—The spectra of detonation flames in an internal-combustion engine give a characteristic banded structure, readily distinguishable from the spectra of explosion and combustion. Lead tetraethyl causes some dispersion of the banded spectrum and a shortening of the ultra-violet region; butyl nitrite (a detonation inducer) accentuates the ultra-violet spectrum. Characteristic lead lines are prominent when lead tetraethyl is used, and show that the process of decomposition of the lead tetraethyl molecule is the important factor, the combination with bromine introduced as ethyl bromide taking place subsequently. The persistence of these lead lines with only minute traces of lead tetraethyl present affords the most delicate test for the presence of this compound. Copper lines from the plug spark appear as emission lines when lead is present in the vapour, but as absorption lines in its absence. The study of the spectra of the flames in the internal-combustion engine promises to be a satisfactory method of elucidating the reaction mechanisms under varying conditions.

B. W. CLARKE.

Wurtemberg oil shales. II. F. C. GAISSER and H. BADER (*Chem.-Ztg.*, 1926, 50, 277–280).—A rapid method of extracting the organic matter from oil shale consists in stirring it into 10–15 times its weight of fused sodium hydroxide, heating for 5–10 min., extracting the fused mass with water, and treating the insoluble material with hydrochloric acid. The organic residue is collected and dried at 105° for analysis in the usual way. From Wurtemberg oil-shale the product so obtained consisted of 19.6% of a voluminous, finely divided, brown powder containing 63.92% C, 7.18% H, 2.15% N, 12.83% O, and 7.4% S; the shale is therefore more of the nature of a coal-shale than of a true oil-shale. Distillation of this type of shale yields 7.8% of tar, 3.3% of water, 85% of "coke," and 3.9% of gas, but if the organic matter is first separated as above and then distilled the yield of tar is 8.5% and that of coke 8.3% (containing 22% of ash). The gases

obtained in the latter operation are rich in hydrogen sulphide and the tar contains a large proportion of sulphur compounds.
A. R. POWELL.

Effect of pressure and temperature on total volume of partially vaporised Midcontinent crude [petroleum]. R. E. WILSON and H. G. SCHNETZLER (Ind. Eng. Chem., 1926, 18, 523—525).—Variations in the water content of the crude oil produce large differences in the ratio of volume of vapour+liquid to volume of cold liquid over a wide range of temperatures and pressures. The shape of the curves obtained by plotting the results indicates solubility of water in the oil. At moderate pressures and temperatures above 150°, the volume of vapour produced is so large that it must be allowed for in calculations concerning design of rectifying stills.
B. W. CLARKE.

Bergin benzine and Bergin oil. E. LÁSZLÓ (Petroleum, 1926, 22, 421—425).—A mixture of 100 kg. of Italian lignite with 67 kg. of topped coal tar and 4 kg. of "Lux" material was "berginised" in a 50-litre bomb by the continuous method, with the addition of 5 kg. of hydrogen. The finely-ground lignite was mixed with the tar to form a paste. The lignite contained 13.8% of water, 8.45% of ash, and 0.8% of sulphur. Hydrogenation took place at 470° under a pressure of 140—150 atm. The expansion gases were freed from benzine by activated charcoal, yielding 8 kg. of benzine. 20 kg. of gas were formed and 139 kg. of "runnings," and the loss was 9%. The benzine was water-white, had an odour of petroleum benzine, and d 0.7303. 84.6% distilled over from 50° to 120°, and the benzine was easily refined. The relation between b.p. range and d corresponded to that of American benzine. The lower fraction obtained by distilling the "runnings" had d 0.990 and contained 12.5% of phenols, being similar to a low-temperature tar oil. The wax content was 1% to 1.5%. The residue was very viscous, and on re-distilling 47% of hard asphalt remained, indicating a total asphalt content of about 60%, but the ash content was small. A third fraction of the "runnings" consisted of water (29.5 kg.) containing 7.83 g. of ammonia per litre. This only corresponded to 19% of the total nitrogen present in the raw material, the rest having escaped as ammonia in the expansion gases.
H. MOORE.

Nature of the protoparaffin waxes in petroleum. A. SACHANEN (SACHANOV) and M. BESTUSCHEV (Petroleum, 1926, 22, 484—486).—Protoparaffins were precipitated by ethyl alcohol from an isoamyl alcohol solution of Grosny petroleum, refined with acid, and treated in benzene solution with floridin; 3% of wax with m.p. 51.5—53.5°, and 3% with m.p. 45—47° were obtained. A third sample of protoparaffin wax was obtained from the precipitate from the inner surface of a pipe line. This, after refining, had m.p. 75—77°. The solubility of these samples in light benzene, machine oil, benzene, and isoamyl alcohol was investigated, and the results are given in tables and graphs. The protoparaffins dissolve as true crystalline substances, and there is no difference between them and pyroparaffins,

observed differences of behaviour being due to the accompanying substances in crude oils and residues.
H. MOORE.

Adsorption of asphalt from mineral oils or residues by hydrosilicate earths. H. HERBST (Petroleum, 1926, 22, 424).—1000 g. of residue from kerosene distillation were diluted with 250 g. of kerosene. The mixture had an asphalt content of 8%, and was filtered through a layer of mixed powdered and granular "Silit" 15 cm. thick, weighing 580 g., at 60°. After 48 hours filtrate commenced to pass, and 95 g. were collected in a further 48 hours, being light-yellow and free from asphalt. A second fraction, 65 g. free from asphalt, was collected, and after three days a further 150 g., dark in colour and containing 7% of asphalt. Thus the "Silit" was exhausted after adsorbing only 2.5% of its weight of asphalt. The first fraction of filtrate was of slightly lower density than the succeeding fractions.
H. MOORE.

Influence of rate of stirring on reaction velocity. HUBER and REID.—See I.

Determination of small quantities of hydrogen in gaseous mixtures. LEBEAU and MARMASSE.—See VII.

PATENTS.

Plant for drying fuels, together with installation for electrical precipitation of dust. SIEMENS-SCHUCKERTWERKE G.M.B.H. (G.P. 425,499, 27.11.21).—Propagation of ignition from the precipitator to the drying chamber is prevented by the cooling and baffling action of a fan installed between the two.
J. S. G. THOMAS.

Burning pulverised fuel. H. KREISINGER, J. ANDERSON, and J. E. BELL, Assrs. to COMBUSTION ENGINEERING CORP. (U.S.P. 1,581,351, 20.4.26. Appl., 30.12.20).—A stream of pulverised fuel is projected downwards into a combustion chamber from which the outlet for flame is at the top, so that the flame takes a U-shaped course. Air is admitted parallel to, and on each side of, the fuel jet, and an additional slower current of air, drawn in by the natural draught of the furnace, is admitted between the descending and ascending legs of the U in order to control the depth of the U.
B. M. VENABLES.

Carbonisation of coal, peat, wood, and the like. C. B. WINZER and P. A. BROWN (E.P. 250,661, 14.1.25).—The material is charged into chambers which move continuously in a cycle in a circular or straight muffle or oven. The charge is heated progressively and the distillation products are drawn off in stages. The carbonised residue finally reaches an unheated zone in the plant where it is cooled and discharged. In the heated zone the heating gases are caused to follow a helical path around the chambers. The charge may be introduced into the chambers in basket containers.
R. A. A. TAYLOR.

Treatment [distillation] of carbonaceous materials. A. M. A. STRUBEN (E.P. 250,699, 26.1.25).—Before distillation the material is mixed

with a substance which has a boiling point higher than that of the least volatile product required to be distilled. A suitable retort for the process consists of a horizontal drum fitted with paddles and other necessary appurtenances, including a condensing system and a still for fractionating the distilled products and recovering the added material.

R. A. A. TAYLOR.

Standpipe apparatus [for carbonisation plant].

J. VAN ACKEREN, Assr. to KOPPERS Co. (U.S.P. 1,581,556, 20.4.26. Appl., 13.12.23).—The top of the ascension pipe from a carbonisation chamber is connected with a discharge pipe which curves over and downwards and leads into a pipe, slightly inclined to the horizontal, which enters the tar main below its longitudinal axis. At the junction of the discharge pipe and the inclined pipe is a valve which moves upwards on to a seating at the end of the discharge, in order to shut off communication between the ascension pipe and the tar main. In the discharge pipe is a spray-nozzle, directed towards the junction of the discharge and inclined pipes, thus playing on the valve.

R. A. A. TAYLOR.

Lignite distillation apparatus J. E. NEUMAIER, Assr. to LIGNITE PRESSED COAL Co. (U.S.P. 1,582,297, 27.4.26. Appl., 1.2.22).—An inclined revolving cylinder is mounted on bearings in a head at each end. Heating means are provided and the material is introduced through an inlet in one head, passes via independent passages through the inclined cylinder, and out through the other head. The heads are mounted on trunnions which permit of variation in the inclination of the cylinder.

R. A. A. TAYLOR.

Distilling oven. O. LASCHE and C. GEISSEN, Assrs. to KOHLENVEREDLUNG GES. (U.S.P. 1,582,650, 27.4.26. Appl., 15.11.23).—The oven consists of a vertical cylinder with a transversely corrugated wall. It is heated from the inside and enclosed in an outer jacket, and a layer of material to be distilled is formed on the outside of the corrugated cylinder.

R. A. A. TAYLOR.

Carbonisation, distillation, and gasification of fuels. C. A. A. M. ROUX (F.P. 600,472, 2.10.24).—The central portion of the fuel mass is gasified and the radiant heat of the process utilised to heat the outer portion of the charge.—R. A. A. TAYLOR.

Water-gas from liquid hydrocarbons. M. BRUTZKUS (E.P. 250,289, 10.11.24; cf. E.P. 155,776 and 144,915, B., 1922, 735 A).—The reaction is carried out in the cylinder of a Diesel type engine, in a four-stroke cycle. In the first stroke the cylinder fills with superheated steam, e.g., at 400° and 1 atm., and this is compressed in the second stroke to about 31 atm., the temperature thereby rising to 1200°. At the beginning of the third stroke (expansion) the liquid hydrocarbon, e.g., residue from naphtha distillation, coal tar, etc., is injected into the cylinder together with sufficient air to burn 10–15% of it. During the stroke the remainder of the hydrocarbon reacts with the steam to form water-gas rich

in hydrogen, which, during the fourth stroke, escapes through the exhaust into the gas-holder.

A. B. MANNING.

Gaseous fuel. J. HARRIS (U.S.P. 1,581,441, 20.4.26. Appl., 2.6.23).—A mixture of 10–50 pts. of acetylene and 90–50 pts. of coal gas by volume is claimed as a fuel for cutting and welding.

R. A. A. TAYLOR.

Manufacture of methane. SOC. D'ETUDES MINÈRES ET IND. (F.P. 590,744, 15.2.24).—Mixtures of carbon monoxide and hydrogen, after a preliminary heating, are led over asbestos or pumice coated with rhodium black.

A. COULTHARD.

Debenzolisation of gas. COMP. POUR LA FABR. DES COMPTEURS ET MATÉRIEL D'USINES À GAZ (F.P. 596,498, 9.4.25).—The wash oil is allowed to circulate while air is excluded.

R. A. A. TAYLOR.

Electric flue gas testing apparatus. G. SOKOLOV-WICHNEVSKY (E.P. 250,478, 12.10.25).—A gas-testing apparatus of the katharometer type is provided with a tubular cooler which at its lower end is connected to a water jet pump, whilst a gas filter and the katharometer cell containing gas and air chambers are arranged at its upper part.

J. S. G. THOMAS.

Liquid fuel. E. M. FLORES (E.P. 249,577, 28.10.24).—Carbureting substances such as crude petroleum or a petroleum fraction 150–220°, or products of the distillation of coal, are added to fermented musts, and, also, if desired, denaturing substances and/or a solution of boric acid in methyl and/or ethyl alcohol or acetone, the mixture being kept for a time and distilled once to produce the fuel. The vapours from distillation may be dehydrated by passing over a drying agent, and the distilled fuel may be enriched by treatment with acetylene or other gases.

H. MOORE.

Alcohol [motor] fuel. R. DE SARIGNY (E.P. 250,019, 8.4.25).—A fuel suitable for internal-combustion engines consists of alcohol 50%, benzol 12%, petrol 34%, ether 3%, and a denaturant 1%, by volume. The denaturant may consist of pyridine 25% and mineral oil 75%.

S. BOWMAN.

Motor fuel oil. F. L. DIETZ (U.S.P. 1,573,307–8, 16.2.26. Appl., [A] 16.11.21, [B] 2.2.22. [B] Renewed 18.7.25).—A fuel for internal-combustion engines consists of (A) low-temperature tar, with the addition of a mixture of alcohol and a hydrocarbon both boiling at 79°, to prevent its solidification; or (B) crude tar from the distillation of coal up to a temperature of 600°, and alcohol.

H. MOORE.

[Composition for] prevention of fuel knock. T. MIDGLEY, JUN., Assr. to GEN. MOTORS CORP. (U.S.P. 1,571,862, 2.2.26. Appl., 18.9.23).—The composition consists of aniline, unsaturated gasoline, and a blending agent.

A. J. HALL.

Recovering oil from oil-bearing sands. D. C. COLLIER (U.S.P. 1,570,205, 19.1.26. Appl., 8.4.21).

—The material is thoroughly agitated with a mixture of water and crushed carbonaceous matter, *e.g.*, coal. The oil and coal become agglomerated and float on the water while the sand settles out. The mixture of oil and coal may be used as a fuel, or the oil may be separated by distillation.

R. B. CLARKE.

Removing water from oils or other liquids.

A. LOZAI (E.P. 239,833, 9.7.25. Conv., 10.9.24).—In a heat exchanger consisting of vertical tubes with a heating chamber supplied with steam, the tops of the tubes are provided with openings to form weirs. A jacketed head is connected to the heat exchanger and heated by the fluid used in the interior of the latter. A vacuum pump assures the flow of the vapours evolved in the tubes counter to the flow of the liquid film through the tubes. The lower ends of the tubes are obliquely notched to prevent a film forming across them. Additional vertical pipes form a connexion between the head and a receptacle below the exchanger to lower the pressure in the latter and facilitate the downward flow in the pipes. When water is eliminated from oil in this apparatus the steam is drawn off vertically and does not come into contact with the dehydrated oil.

H. MOORE.

Separating the phases of emulsions, particularly of petroleum, by means of an electric current. J. H. C. DE BREY, Assr. to NAAML. VENNOOTS. BATAAFSCHE PETROLEUM MAATSCH. (U.S.P. 1,570,209, 19.1.26. Appl., 11.4.24).—The essential feature of the process consists in the application of a pulsating alternating current, the peak voltage of which is at least double the effective voltage. Petroleum can be freed from small quantities of water in this manner.

R. B. CLARKE.

Treating [dehydrating] apparatus for crude oil. H. L. EDWARDS (U.S.P. 1,578,273, 30.3.26. Appl., 29.3.24).—A longitudinal pipe containing heating sections, closed transversely, but traversed by oil tubes longitudinally, serves as a conduit for the oil. Heating fluid is supplied by a pipe to the sections. Intermediate sections are unobstructed and allow settling out of the water, for which outlets are provided.

H. MOORE.

Electrical dehydrator [for crude oil]. D. K. CASON, JUN. (U.S.P. 1,579,515, 6.4.26. Appl., 16.8.24).—The apparatus consists of a series of conical plates spaced apart at gradually increasing distances from top to bottom. Alternate plates are earthed and the intervening ones connected to a high-tension circuit.

S. BOWMAN.

Dehydrating oil. H. C. EDDY, Assr. to PETROLEUM RECTIFYING Co. OF CALIFORNIA (U.S.P. 1,580,591, 13.4.26. Appl., 26.8.24).—Water and solid impurities are separated from an oil emulsion by treating it to agglomerate the fine water particles, allowing the liquid to stratify, and removing the driest portion. A wetter portion is passed through

water to wash out water and suspended impurities, and the supernatant oil is removed. H. MOORE.

Distilling petroleum oils and other liquids under high vacuum. O. Y. IMRAY. From ZIELEY PROCESSES CORP. (E.P. 248,513, 24.12.24).—In a process for the distillation of the heavier fractions of petroleum to produce saleable lubricating oils without subsequent refining, the oil moves downward through inclined conduits in a shallow stream under a pressure of 50 mm. of mercury or less, being subjected to increasing heat. Instantaneous evaporation of the oil and free egress of the vapours prevent cracking or coking in the conduits. A header supplies oil to the conduits, and exit headers are separately connected to separating towers and thence to fractional condensers, jacketed for a cooling medium. The condensers are connected to duplicate receiving tanks, for convenience in withdrawing the product. To facilitate the removal of vapour each conduit is provided with an internal conduit perforated with small holes, which allow the passage of oil or vapour, but tend to break up any froth formed. The ends of the internal conduit may be partially closed by plugs. The vacuum pump is preferably of the ejector type. The vapour outlets throughout the system are of large capacity, and intercommunicate, to promote equalisation of the pressure.

H. MOORE.

Cracking liquid or readily fusible organic complexes [tars and heavy oils] by means of an electric arc. C. LONGHI (E.P. 248,830, 10.12.24).—To obtain gases rich in acetylene and also carbon black a submerged arc is used and the material acted upon is kept at a relatively low temperature by circulation through a refrigerating device. The materials used are tars and crude and heavy oils generally. The gases produced consist of hydrocarbons of the paraffin series and a large proportion of acetylene. The sulphur and nitrogen present are converted into hydrogen sulphide and hydrocyanic acid and may be separated from the other products, thus allowing the utilisation of substances of high nitrogen or sulphur content. For the generation of the arc mono-phase, two-phase, three-phase, or direct current may be used. The furnace is gas-tight and constructed of metal, and the arc may be continuous or disruptive. The liquid contents of the furnace are circulated through a refrigerator by means of a pump. The carbon or metal electrodes may be hollow for the conveyance of gaseous or liquid substances into the arc zone. The depth of the liquid layer above the arc has a great influence on the proportion of acetylene produced.

H. MOORE.

Cracking process [blending]. J. T. SHEVLIN. From UNIVERSAL OIL PRODUCTS Co. (E.P. 249,604, 29.12.24).—An improvement on the Dubbs process (cf. E.P. 160,236; B., 1921, 292 A). To obtain lighter products a heavy hydrocarbon oil is subjected to a cracking temperature in a heating coil, and thence passes to an expansion chamber and to a dephlegmator. The oil and condensate from the

dephlegmator are returned, together with an independent supply of charging stock, to the cracking coil. The pressure in the dephlegmator may be above atmospheric, but below that in the cracking coil or conversion chamber. More than one dephlegmator may be used, the vapours travelling through them in obstructed paths, fresh charging stock being introduced where desired and the non-vaporised residue being returned to the cracking coil. The non-condensable gas may be returned to the cracking zone to assist in the conversion.

H. MOORE.

Treatment of hydrocarbons. E. GOLTSTEIN (E.P. 249,895, 30.9.24).—Petroleum and similar hydrocarbons are subjected to the action of light of short wave-length such as is obtained by the use of a mercury vapour lamp. The treatment may be applied to either the liquid or vapour phase during distillation, the effect being polymerisation and consequent change in the physical characteristics of the distillates. For example, using a quartz mercury vapour lamp at 60 volts and 6 amp. the viscosity (Engler) of a Russian mineral oil was increased from 6.4 to 7.8 in $\frac{1}{2}$ hr. S. BOWMAN.

Pyrogenesis of petroleum. E. M. CLARK, Assr. to STANDARD DEVELOPMENT Co. (U.S.P. 1,578,802, 30.3.26. Appl., 3.7.19).—Two separate bodies of heavy oil are confined under pressure in a digester. To one heat is applied, and the vapours pass into the second body, which is cooled to promote condensation of the desired products. The oil displaced from the second body is fed to the first body, until the second body becomes of substantially lower boiling point than the first body, conversion being thus effected at a relatively low pressure.

H. MOORE.

Apparatus for converting higher boiling-point hydrocarbon oils into lower boiling-point oils. W. M. McCOMB (U.S.P. 1,579,554, 6.4.26. Appl., 2.3.21).—Conversion of heavy oils into lighter oils is effected by means of a vertical spiral coil, the lower turns of which have an increased inclination to accelerate flow of oil. The coils are grouped for electrical heating.

S. BOWMAN.

Treating [cracking hydrocarbon] oil. G. EGLOFF (U.S.P. 1,579,601, 6.4.26. Appl., 26.4.17. Renewed 27.9.22).—An externally heated still is provided with means for producing an electric arc within the charge of oil in the still. The oil is thus heated to a cracking temperature and the vapours are passed through a reflux atmospheric condenser, the reflux returning to the still and the uncondensed gases passing to another condenser connected with a receiver.

S. BOWMAN.

Apparatus for distilling hydrocarbons N. F. W. HAZELDINE (U.S.P. 1,580,372, 13.4.26. Appl., 9.5.22).—A tank is divided by a horizontal partition into two chambers. An inner shell within the upper chamber provides a jacket for cooling fluid. A riser extends upwards from an opening in the partition and projects through the bottom wall

of the inner shell. The oil, to be distilled is supplied to the lower chamber, which is provided with a steam coil. Condensed liquid may be withdrawn at a point beneath the outlet of the riser.

H. MOORE.

Desulphurising petroleum oils. J. B. RATHER, Assr. to STANDARD OIL Co. OF NEW YORK (U.S.P. 1,580,531, 13.4.26. Appl., 9.5.23).—Hydrogen sulphide is dissolved in the oil, which is then mixed with a mixture of sodium hydroxide and litharge, or sodium plumbite. Lead sulphide is produced, suspended in the oil, and promotes reaction between the sulphur compounds of the oil and the plumbite solution, rendering the oil sweet to the "doctor test" and non-corrosive.

H. MOORE.

Centrifugal separating apparatus [for treating mineral oils]. P. P. HARVEY and H. J. HOLFORD (E.P. 250,319, 8.1.25).—A centrifugal separator which is specially suitable for oils containing waxy matter is provided with a number of vertical tangential (almost radial) vanes in the lower part of the bowl, and above these, with a number of deflecting plates of dish or conical shape. The material is fed to a space left among the vanes at the centre of the bowl at the bottom and is separated into its constituents while rising between the radial vanes, the circular deflectors then serving to guide the products to appropriate outlets. Any waxy matter is largely deposited on the radial vanes, hence a considerable quantity can accumulate before cleaning is necessary.

B. M. VENABLES.

Penetrating oil and lubricant. H. N. COPTHORNE, Assr. to ILLINOIS GRAPHITE Co. (U.S.P. 1,580,233, 13.4.26. Appl., 5.6.24).—Deflocculated graphite is suspended in a distillate of $d^{15.5}$ 0.843 obtained by cracking pentadecane at a distilling range of 220–350°.

H. MOORE.

Treatment of waste products [acid tar] from acid treatment of petroleum oil. R. A. HALLORAN, W. N. DAVIS, and G. A. DAVIDSON, Assrs. to STANDARD OIL Co. (U.S.P. 1,579,607, 6.4.26. Appl., 6.9.23).—The acid tar resulting from the treatment of petroleum with sulphuric acid is heated under a pressure of 50 lb. per sq. in. either with or without the addition of fuel oil. Separation into clean weak acid and an oil suitable for fuel is obtained.

S. BOWMAN.

Winning of technically valuable products from the alkaline waste products resulting on refining mineral, tar, and shale oils. SUDFELT & Co., Assees. of M. GELBKE (G.P. 426,947, 27.4.23).—The waste products, freed as far as possible from water, are distilled *in vacuo* with superheated steam as long as volatile constituents, especially unsulphonated oils, are evolved. The residue is decomposed with dilute sulphuric acid. The distillates may be used as lubricants, while the products from the residue may serve as emulsifying media in place of soap or Turkey-red oil, and for the hydrolysis of fats.

R. A. A. TAYLOR.

Apparatus for distillation of carbonaceous substances. E. R. SUTCLIFFE, AssT. to PURE COAL BRIQUETTES, LTD. (U.S.P. 1,584,487, 11.5.26. Appl., 13.5.18).—See E.P. 166,161; B., 1921, 618 A.

Apparatus for cracking oils. R. C. HOLMES and F. T. MANLEY, AssTs. to TEXAS Co. (U.S.P. 1,583,973, 11.5.26. Appl., 26.6.19).—See E.P. 160,200; B., 1921, 291 A.

Apparatus for quenching coke. F. ALBACH (E.P. 246,497, 22.1.26, and 246,508, 23.1.26. Conv., 22 and 24.1.25).

Burners for liquid fuels. J. Y. JOHNSON. From BADISCHE ANILIN- & SODA-FABR. (E.P. 251,191, 13.11.25).

Production of oil gas. E. C. R. MARKS. From GASIFIER Co. (E.P. 251,675, 7.1.25).

Treating waste liquors containing cyanogen compounds (E.P. 250,824).—See VII.

Ammonia from ammoniacal liquor (G.P. 426,863).—See VII.

III.—TAR AND TAR PRODUCTS.

Isolation of homogeneous pyridine through the perchlorate. ARNDT and NACHTWEY.—See A., May, 525.

PATENTS.

Recovery of phenols and cresols. R. M. CRAWFORD (U.S.P. 1,582,512, 27.4.26. Appl., 16.4.25).—Oil containing phenol and cresols is washed continuously with an alkaline solution containing sufficient alkali to remove the phenol as an alkali phenoxide, and the cresols are subsequently removed by extracting the oil with fresh alkaline solution. L. A. COLES.

Continuous working up of waste acid obtained in the purification of benzol. H. MENZEN (G.P. 426,387, 23.3.24).—Apparatus for the continuous treatment of the waste acid with water, sodium hydroxide solution, and oil, comprises a mixing vessel with a separator below it, storage vessels for the waste acid, sodium hydroxide solution, and hot oil, and a condenser delivering into a separator. L. A. COLES.

Cracking tars (E.P. 248,830).—See II.

Treating alkaline waste products from refining tar oils (G.P. 426,947).—See II.

IV.—DYESTUFFS AND INTERMEDIATES.

Oxidation and constitution of Sulphur Black. U. PERRET (Annali Chim. Appl., 1926, 16, 69—83).—A description of the preparation of the dye, some of its properties, and examination of the self-heating effects, development of acidity, and changes in dyeing properties during isolation in the dry state. Analysis of various samples showed that recent ones were neutral or nearly so, whereas old ones were often considerably acid. The total sodium

and sulphur contents were found to vary considerably, and colours having similar dyeing properties and prepared from the same material may have varying compositions. In some lengthy experiments on drying under various conditions, deterioration nearly always took place. When the dye is suspended in 0.5*N*-sulphuric acid a considerable portion of the sodium in the dye resists the action of the acid, and also old samples are stated to react acid before all the sodium is transformed into sodium sulphate. It is surmised that there may be certain acidic (possibly sulphonic) groups present which are not explained by the usually suggested formulæ for sulphur dyes, the latter being discussed. Three stages of oxidation can be distinguished: (1) during precipitation of the dye (with the formation of leuco-compound) and action of the air on the sodium polysulphide; (2) oxidation of the dye itself with possible transformation of thiol to sulphonic groups; (3) oxidation of the sulphur in the chain to form sulphuric acid. Old samples of the dye were found to be insoluble in sodium sulphide solution and could not be made soluble by re-fusion with polysulphide, as has been stated. Deficiency of polysulphide in preparing the black may lead to the production of sulphites, and not thiosulphates, on air treatment, and give rise to strongly bluish coloured mother liquors, and reasons are given for assuming that the dye is present here as soluble sulphite compounds. S. B. TALLANTYRE.

Fluorescope [for detecting small quantities of fluorescein]. H. MARCELET (Chim. et Ind., 1926, 15, 528; Bull. Soc. Chim., 1926, [iv.], 39, 803—804; Ann. Falsif., 1926, 19, 296—298).—A simple apparatus is described for determining small quantities of fluorescein in solution by comparing the fluorescence of the solution with that of a standard solution. A beam of light is divided by means of inclined mirrors so that half illuminates one tube and the other half the comparison tube. The tubes containing the solutions are illuminated horizontally and are viewed from above. For very dilute solutions the fluorescein is extracted with ether and so concentrated before comparing with the standard. A. B. MANNING.

See also A., May, 508, Nitration of decahydro-naphthalene (NAMETKIN and MADAIEFF-SSITSCHIEFF). 509, 1-Phenylnaphthalene (WEISZ and WOIDICH). 511, Action of sulphur on monochloroanilines. Mechanism of aromatic thionation and structure of sulphide dyestuffs (HODGSON). 512, Catalytic reduction of *a*-nitronaphthalene to *a*-naphthylamine (PARRETT and LOWY). 513, Colouring matters derived from thiocarbodibenzidine (ROSSI and CECCHETTI). 514, Influence of sulphur on colour of azo dyes (PALMER and REID). 515, Identification of phenols by means of the spectroscope (WALES and PALKIN); Colour and constitution. Influence of the methylthiol group alone and in conjunction with the methoxyl group (HODGSON and HANDLEY). 521, Preparation of quinizarin (REYNOLDS and BIGELOW). 522, "Polymethine" dyes and a general dye

formula as basis of a new generalisation (KÖNIG). 526, Preparation and properties of methyliso-quinoline-yellow (PHILLIPS and GOSS). 527, Quinoline Red and related dyes (SCHEIBE and FISCHER). 530, Synthesis of phenyrosinduline (LANTZ and WAHL). 531, Derivatives of 2-phenylbenzothiazole (BOGERT and CORBITT); Polychrome Methylene Blue (MACNEAL and KILLIAN); Dyes from the alkaloids of ipecacuanha (PYMAN; PALKIN and WALES).

Influence of rate of stirring on reaction velocity. HUBER and REID.—See I.

Neocyanine, a new sensitiser for the infra-red. DUNDON, SCHOEN, and BRIGGS.—See XXI.

PATENTS.

Manufacture of diarylamines. O. Y. IMRAY. From SOC. OF CHEM. IND. IN BASLE (E.P. 250,819, 11.9.25).—Nuclear substitution products of diphenylamine are prepared by the action of nuclear substitution products of aromatic hydrocarbons upon alkali metal compounds of aromatic amines, one or both of the compounds containing at least one nuclear substituent in addition to the halogen atom or amino-group. Catalysts, such as copper or its compounds, may be present during the reaction. For example, 140 pts. of sodium are added at 175–180° to 1800 pts. of *o*-toluidine containing 1 pt. of copper powder; after heating to 160–190° until the sodium has disappeared, 750 pts. of *o*-chlorotoluene are added, and the mixture is heated for 24 hrs. at 250–300° in a closed vessel. The *oo'*-ditolylamine obtained is purified by washing with water, followed by fractional distillation *in vacuo*.

L. A. COLES.

Mono-oxamic acids of diaminoanthraquinones. BRITISH DYESTUFFS CORP., LTD., H. M. BUNBURY, and R. ROBINSON (E.P. 250,883, 19.12.24).—Compounds of the type, $\text{NH}_2\text{AQ.NH.CO.CO}_2\text{H}$ (AQ = an anthraquinonyl residue) are obtained by heating mixtures of diaminoanthraquinones, oxalic acid, and water to 105–110°. The products, which contain an amino-group free to take part in condensations and other reactions, *e.g.*, in the preparation of aminodiamthrimides, are dark red to violet powders which are thrown out as gels on the addition of mineral acids to their solution in water or in alkalis; on heating to 180–190° they split off carbon dioxide, yielding formamido-derivatives.

L. A. COLES.

Preparing 1:8-aminonaphtholsulphonic acids. T. GUBELMANN and J. M. TINKER, Assrs. to NEWPORT Co. (U.S.P. 1,573,056, 16.2.26. Appl., 9.5.25).— α -Naphthylaminesulphonic acids containing a sulphonic group in position 8, or their salts, are fused with caustic alkalis in the presence of ammonia, under pressure. Formation of 1:8-dihydroxynaphthalenesulphonic acid is thus avoided. For example, for the preparation of S-acid (1:8-aminonaphthol-4 sulphonic acid) 500 lb. of caustic soda, 500 lb. of caustic potash, and 400 lb. of water are dissolved by heating in an autoclave, and 1000 lb. of the

monosodium salt of α -naphthylamine-4:8-disulphonic acid are added. Ammonia gas is introduced up to a pressure of 100–150 lb. and the autoclave is heated to 190–200° for 3–4 hrs. After releasing the ammonia pressure the mass is dissolved in water and acidified in the usual manner.

R. B. CLARKE.

Reduction of organic compounds with hyposulphites. H. BUCHERER (G.P. 423,029, 1.12.22).—Organic compounds insoluble in water are reduced by treatment with sodium hyposulphite and acetic acid, or other aliphatic monocarboxylic acids, such as formic acid or, particularly in the reduction of mono- or dinitronaphthalenes, stearic or oleic acid. Mixtures of the materials are boiled under a reflux condenser until the compounds have dissolved, or until evolution of sulphur dioxide has almost ceased. Examples are given of the production of acetanilide and sulphanilic acid from nitrobenzene; of α -acetamidonaphthalene and a mixture of two aminonaphthalenesulphonic acids (probably the 1:4- and 1:2-compounds) from α -nitronaphthalene, of 1:8-diaminonaphthalenedisulphonic acid and small quantities of perimidine compounds from 1:8-dinitronaphthalene; and of a condensation product of equimolecular proportions of acenaphthenequinone and acenaphthenone from acenaphthenequinone.

L. A. COLES.

Preparation of diphenylene sulphide. A. SCHÖNBERG (G.P. 426,476, 17.7.23).—Diphenylsulphoxide is treated with halogen-free substances capable of removing water. For example, diphenylsulphoxide is warmed with phosphorus pentoxide until a reaction commences and then left to itself. After cooling, the melt is extracted with benzene. Diphenylene sulphide remains on distilling off the benzene. The yield is not so good as when sodamide or potassamide is used (cf. A., 1924, i., 39).

A. COULTHARD.

Manufacture of trisazo-dyestuffs. O. Y. IMRAY. From FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 251,140, 24.6.25).—See U.S.P. 1,565,344; B., 1926, 185.

Colouring-matter paste. BADISCHE ANILIN- & SODA-FABR., Assees. of E. THEOBALD (U.S.P. 1,584,202, 11.5.26. Appl., 8.6.25).—See E.P. 247,052; B., 1926, 355.

Dyes (F.P. 599,566).—See VI.

Azo dyes (F.P. 600,106).—See VI.

Dyeing artificial silks (G.P. 423,601).—See VI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Chemical analysis of cotton. XI. Absorption of Methylene Blue from buffered solutions. D. A. CLIBBENS and A. GEAKE (J. Text. Inst., 1926, 17, T 127–144).—In a previous paper (B., 1923, 1066 A) it was shown that a measurement of the absorption of Methylene Blue by cotton is profoundly influenced by the presence of traces of acids or alkalis. This is now ascribed to a hydrogen-ion

effect and the test, for analytical purposes, is simplified by having a phosphate mixture in the dye solution so that neutrality ($p_H 7$) is maintained automatically. The effect of hydrogen-ion concentration has been studied particularly with modified celluloses. The absorption by the oxycelluloses (A., 1925, i., 234; B., 1925, 165) falls rapidly as the acidity of the mixture increases. The product obtained by drying sulphuric acid into cotton also absorbs Methylene Blue freely, but in this instance the absorption does not fall as acidity increases. Using a buffered solution for the oxycelluloses and a solution containing free acetic acid for the other product it is thus possible to distinguish between two common types of cellulose modification which may be encountered in bleaching, even when present in the same material. Some experiences of the quinhydrone electrode for determining hydrogen-ion concentrations in Methylene Blue solutions are described.

Experiments on the absorption of Methyl Green and Malachite Green by caustic-scoured cotton and the effect of the alkali present (recorded as ash alkalinity) are also described. With these dyes it was obvious that much of the apparent absorption was due to precipitation of the colourless dye bases by the free alkali present. When the ash alkalinity was reduced to a minimum by washing with acid the absorptions fell into line with those recorded for Methylene Blue, diminishing as the severity of the scouring treatment increased. J. C. WITHERS.

Chemical analysis of cotton. XII. Hydrocellulose. (MISS) C. BIRTWELL, D. A. CLIBBENS, and A. GEAKE (J. Text. Inst., 1926, 17, τ 145—170).—"Hydrocellulose" is defined as any preparation of cotton cellulose (other than esters) the properties of which have been altered to a greater or less extent by the action of acids, but the work described is limited to preparations which still retain the structure of the cotton hair. Such materials have been obtained by steeping cotton in various acids at temperatures between 20° and 100° ("acid steeping") and by drying fabric impregnated with small quantities of dilute acids ("acid drying"). Tensile strength, viscosity (in cuprammonium hydroxide solution), copper number, and Methylene Blue absorption measurements have been made on the products in order to explore the effects of temperature, time, and acid concentration. Simple relations between loss of strength, viscosity, and copper number, taken in pairs, are found to hold between wide limits, at any rate over the range of acid attack likely to occur in technical processes. The relation between loss of strength and viscosity leads to a suggestion that for textile purposes the minimum permissible viscosity of a 2% solution of a cotton material in the cuprammonium solvent shall be 1000 times that of water (or $\log \eta = 1$). Viscosity is most sensitive as an indication of tendering in the early stages, up to the first 20% loss in breaking load, whilst copper number is most sensitive when the material is approaching disruption. A connexion between copper number and viscosity is best brought out by considering the viscosity of the 2% cellulose

solution, η , relative to that of the solvent, η_0 , this being 0.0152 poise. This gives a new term, $\log \eta/\eta_0$, designated \bar{V} , which is connected with copper number, N_{Cu} , for the hydrocelluloses obtained by acid steeping or by acid drying below 70°, in the expression $N_{Cu}\bar{V}^2 = 2.6$. This relation is most useful in distinguishing hydrocelluloses from certain oxycelluloses which resemble them closely in other characteristics. The value 2.6 is fairly constant for hydrocelluloses over the important range represented by a loss in breaking load of 15—75%, but for oxycelluloses the figure is distinctly higher and is not constant. When cotton is attacked by sulphuric acid of increasing concentrations it first suffers a loss of affinity for basic dyes, which is the normal effect of acid attack, but then acquires enhanced affinity. In this respect, therefore, the sulphuric acid product resembles oxycelluloses, but it can be distinguished from these because it absorbs Methylene Blue quite as well from an acid solution as from a neutral one (cf. preceding abstract). The product contains sulphuric acid in a state of combination which is not destroyed by the most drastic alkali boiling. The normal effects of alkali boiling on the hydrocelluloses are loss of weight and considerable drop in copper number, but the material does not become much weaker (unless the loss in weight is great) and the viscosity remains constant. The factors governing the rate of attack have also been explored. The connexion between time and copper number, concentration and temperature being constant, is well shown by the expression $N_{Cu} = K.T^{0.6}$. Hydrochloric and sulphuric acids in equimolar solutions attack cotton at about the same speed. A 10° rise in temperature, over the range 20—100°, increases the copper number about 2.3 times. The effect of hydrochloric acid is greatly accelerated by the presence of much neutral salt, an important effect which must be considered when discussing the action of concentrated solutions of zinc or magnesium chlorides on cotton. J. C. WITHERS.

Chemical analysis of cotton. XIII. Scouring losses. R. G. FARGHER and (MISS) L. HIGGINBOTHAM (J. Text. Inst., 1926, 17, τ 233—246).—Using an experimental kier which was found to reproduce technical conditions satisfactorily, a study has been made of the caustic boil under the extreme range of conditions represented by normal works practice. Typical American, Egyptian, and Indian yarns were tested, and the factors determined were scouring loss, residual wax content, and the final Methylene-Blue absorption and copper number. Various modifications of the water, soda, and lime boils have also been tested on the same yarns. The results are interpreted as indicating the relative efficiencies of pressure, alkali concentration, lime, etc. in scouring and of Methylene-Blue absorption and copper number as control tests. In addition, scouring losses during the normal pressure boil with caustic soda have been determined for a wide range of cottons, the literature on this subject containing so many contradictions. The losses ranged from 6 to 9%, American varieties coming at the lower

limit, Egyptian intermediate, and Indian high. Only one cotton was found outside this range, namely Pima, with a loss of 11.5%, but this variety is abnormal in other respects. About half the material lost cannot be accounted for as wax, nitrogenous material, mineral matter, or other substances removable by cold water or cold dilute acids. The above losses in the normal caustic boil do not represent all the material in cotton which is less resistant than "normal" cellulose to caustic alkali; a further loss of 1–2% occurs if 3% sodium hydroxide is used at 40 lb. excess pressure instead of 1% at 20 lb.

J. C. WITHERS.

Literature of keratin (the principal constituent of wool). J. BARRITT (*J. Text. Inst.*, 1926, 17, T 111–126).—A review dealing with the analysis, constitution, reactions, degradation by hydrolysis and by mild oxidation, uses, and properties of keratin, with bibliography.

J. C. WITHERS.

Rancidity and oxidation of fatty oils in regard to wool lubrication. W. RHYS-DAVIES (*J. Text. Inst.*, 1926, 17, T 220–232).—Technical defects caused by the oxidation of wool oils are discussed and traced mainly to the amount of linoleic acid glyceride in commercial olive oils. The Mackey tester is more trustworthy for judging wool oils than such characteristics as iodine value.

J. C. WITHERS.

Detection and determination of glycerol in cotton cloths and sized yarns. G. SMITH (*J. Text. Inst.*, 1926, 17, T 187–191).—For detection, the material is extracted with alcohol and, after removing fat and zinc and magnesium salts present, the glycerol is benzoylated by the Schotten-Baumann method. The dibenzoate has m.p. 72–73°. The less specific colour tests due to Denigès are also described. Provided that more than 1% is present, the "I.S.M." acetin process is satisfactory for quantitative determinations.

J. C. WITHERS.

Effect of sizes on the elastic behaviour of flax yarns. J. A. MATTHEW (*J. Text. Inst.*, 1926, 17, T 192–205).—Load-extension diagrams of sized flax warp yarns show that the ordinary sizing process has the effect of reducing the extensibility of the yarn by about half. Very little of this effect is due to an adhesive action of the starch, since, under the conditions on the machine, cold water has the same result. The effect is caused by applying tension while the yarn is wet.

J. C. WITHERS.

Effects of humidity on the properties of fabrics, with special reference to the control of humidity during strength tests. R. G. PARKER and D. N. JACKMAN (*J.S.C.I.*, 1926, 45, 47–54 T).—Cotton, linen, wool (ordinary and chlorinated for the production of an unshrinkable finish), silk, Celanese, and viscose fabrics dried over phosphorus pentoxide for 5 days contained 0.8%, 1.0%, 2.0%, 1.1%, 0.1%, and 2.2% of moisture and these regained moisture at similar rates when exposed to a moist atmosphere, there being a tendency for the moisture content to rise with increasing rapidity

as the humidity rises so that their moisture contents at 100% relative humidity become very large and indefinite in amount. At a normal humidity of 70%, typical samples of fabric contained 6% to 12.4% of moisture in the order Celanese, cotton, linen, silk, wool, viscose. The regain of moisture by viscose is similar to that of wool, and chlorination of wool does not appreciably affect its regain. The initial rates of regain of moisture by dry fabrics are very rapid. Fabrics dried over phosphorus pentoxide showed a high rate of absorption of moisture when exposed to an atmosphere of 50% relative humidity, equilibrium being obtained within 1 hr. (cotton), 3 hrs. (Celanese), and 5–10 hrs. (wool, silk, and viscose silk). Cotton and linen showed a slight increase in bursting strength with increasing humidity, the increase for linen being over 25% when wet. Under similar conditions all the other fabrics behaved similarly to one another in sustaining a large and fairly regular decrease in strength with increase in humidity. Wool, silk, and Celanese fabrics are more weakened by boiling for 1 hr. in a 1% solution of sodium oleate than in 1% solution of sodium carbonate.

A. J. HALL.

[Comparison of] tensile strengths of textile fibres under wet and dry conditions. J. OBERMILLER and M. GOERTZ (*Textilber.*, 1926, 7, 163–168, 245–246).—The tensile strengths of (air) dry and wet single textile fibres, before and after extraction of their natural fats and waxes by means of benzene, were determined. Average results for 50 to 100 fibres indicate that the ratios of the wet and dry tensile strengths of cotton, wool, natural silk, cuprammonium ("Adlerseide"), viscose, nitrocellulose, and cellulose acetate artificial silks are 110–120%, 80–90%, 75–85%, 50–60%, 45–55%, 30–40%, and 65–70% respectively, and that these ratios and the tensile strengths of individual fibres are not appreciably affected by extraction of the fat. The tensile strengths of wet and dry fibres and also their ratios are considerably decreased when the dry fibres are exposed to a temperature of 75–100° for 87–119 hrs. For example, the ratios of the wet and dry tensile strengths of American Texas cotton, Indian cotton, merino wool, natural silk (degummed), cuprammonium silk ("Adlerseide"), and "Vistra" silk before and after the heat treatment were 114 and 108.1%, 112.4 and 101.7%, 89.4 and 85.5%, 82.3 and 65.8%, 59.7 and 52.0%, 53.4 and 51.7% respectively.

A. J. HALL.

Weighting and decreasing the lustre of artificial silk. W. BRUCKHAUS (*Kunstseide*, 1925, 7, 260–261).—The lustre of artificial silk is decreased so that it more closely resembles that of natural silk by precipitating barium sulphate within the fibres. For example, artificial silk is steeped in a warm 2–3% solution of sulphuric acid and then directly immersed for 20–30 min. in a 3–5% solution of barium chloride at 70°. The resulting silk absorbs dyes evenly, especially in the absence of salt, and the barium sulphate does not dust out. Artificial silk may be satisfactorily weighted 3–5% by means of barium sulphate but excellent weighting up to about

185% may be obtained by means of the ordinary methods employed for natural silk using stannic chloride, sodium phosphate, and sodium silicate; the resulting silk absorbs dyestuffs evenly.

A. J. HALL.

Spruce wood. H. URBAN (Cellulosechem., 1926, 7, 73—78).—Willstätter and Zechmeister's method of separating lignin from wood is modified by using a mixture of 3 vols. of ordinary concentrated hydrochloric acid (d 1.18) and 1 vol. of phosphoric acid (d 1.7), the wood being treated for about 2 days at 20°. The mixture dissolves cotton wool in $1\frac{1}{2}$ min., and it has the advantages that the lignin is obtained almost free from chlorine, filters readily, and that the inconvenience of handling super-concentrated hydrochloric acid is avoided. Spruce wood, extracted with a mixture of alcohol and benzene, and containing 4.7% OMe, yielded 20—26% of lignin of the composition C 64.0, H 6.1, OMe 15.45, Cl 0.33%, which is expressed by the formula $C_{33}H_{36}O_{12}(OMe)_4$. This is best methylated by suspending 3 g. in 100 c.c. of 45% potassium hydroxide and adding 100 c.c. of methyl sulphate during 10 hrs. with cooling. After one such treatment the MeO-content is 31.3%, and after two treatments the maximum of 32.4% is reached. This corresponds with the introduction of five methoxyl groups, making the formula of the original lignin $C_{33}H_{29}O_6(OH)_5(OMe)_4$, it being assumed that 1 mol. of water is lost. Fusion with potassium hydroxide yields 14% of protocatechuic acid. Cellulose is readily methylated by the above procedure. 2 g. of raw cotton, previously treated with acetone and water, after two methylations with 200 c.c. of 45% potassium hydroxide and 200 c.c. of methyl sulphate, contains 44.7% of OMe, which remains unchanged on further methylation. Methylated cotton retains its structure; it dissolves in chloroform and tetrachloroethane to clear, viscous solutions, but is insoluble in water, alcohol, ether, ethyl acetate, acetone, carbon tetrachloride, and carbon disulphide. Sulphite-cellulose requires nine methylations to reach its maximum of 41.2% OMe. Wood reaches its maximum of 41.0% OMe in five methylations. This, and also methylated wood containing only 20% OMe, yield, on hydrolysis with hydrochloric and phosphoric acids, a methyl-lignin containing 25% OMe. By methylating wood once, hydrolysing, and remethylating the residue a 20% yield of methyl-lignin containing 32.0% OMe is obtained. Although in fully methylated wood the carbohydrate part must be highly methylated, only a small proportion of it can be extracted with chloroform, and the proportion of lignin in the residue is not greatly increased. This suggests combination between the carbohydrate and the lignin. Some of the extracts show a tendency to crystallise. By treating lignin with sodium hydroxide and *p*-toluenesulphochloride two hydroxyl groups (per C_{33}) are readily attacked and a third after repeated treatment; this agrees with the ready methylation of lignin to 24% OMe corresponding with an additional $2\frac{1}{2}$ OMe groups. Methyl-lignin is not affected by *p*-toluenesulphochloride. Wood can be completely dissolved by heating at 75° in acetic anhydride and

zinc chloride and passing chlorine through the mixture; the product is precipitated by pouring into water. After such treatment sulphite-cellulose contains 3% Cl and 44.3% of acetyl, and lignin contains 31.6% Cl and 16.2% of acetyl. Wood then contains 11.0% Cl and 34.4% of acetyl, which agrees with the composition calculated on the assumption that it consists of 70% of cellulose and 30% of lignin.

A. GEAKE.

Wetting spruce wood with calcium and magnesium bisulphite liquors. C. G. SCHWALBE and K. BERNDT (Papier-Fabr., 1926, 24, 250—253).—When wood shavings ($44 \times 2 \times 7$ mm.) are shaken with bisulphite solution, containing 3.60% SO_2 and 0.959% MgO or 1.070% CaO, at 70°, the weight absorbed is 126—137% after 3 hrs. and 140—150% after 8 hrs. There is no significant difference between magnesium and calcium bisulphites. The unabsorbed solution is somewhat poorer in sulphurous acid and in base than the original liquor, even when allowance is made for loss of sulphur dioxide into the space above the liquid (which was filled with carbon dioxide to avoid oxidation) and for dilution by the moisture contained in the wood.

A. GEAKE.

Indicators for the cellulose industry. E. ÖMAN (Papier-Fabr., 1926, 24, 267—270, 285—288, 299—303).—Free sulphurous acid in sulphite liquors may be determined by titration with 0.1N-sodium hydroxide to the stage of hydrogen sulphite or of sulphite. For the former titration the best indicator is bromophenol-blue, which has a marked and sharp colour change at the required p_H (4.2). This titration is comparatively little affected by the other weak acids present, viz., carbonic, acetic, and formic acids, and the amounts of calcium sulphite calculated from the titre are accurate to within 1%. For titration to complete neutralisation thymolphthalein is a better indicator than phenolphthalein. With the latter the colour change takes place at p_H 8.2, instead of 9.3, which corresponds with exact neutralisation; the end point also is not very sharp and depends on the amounts of water and of indicator added. White liquor from sulphate-cellulose manufacture may contain sodium hydroxide, sulphide, carbonate, sulphate, and sulphite, and may be titrated with acid in three stages. At p_H 11 the sodium hydroxide and half the sodium sulphide are neutralised, at p_H 9 half the sodium carbonate is also neutralised, and at p_H 4 the sodium sulphide and carbonate are completely neutralised and also half the sulphite. The best indicators for the three stages are Nile-blue, thymol-blue and bromophenol-blue respectively. Frequently the determination of sodium hydro-sulphide by titration with iodine, and of sodium hydroxide by titration with hydrochloric acid in the presence of Nile-blue, is sufficient. Mixtures of sodium hydrosulphide and sulphite may be analysed by titrating with iodine, followed by titration of the acid thus liberated. The acid liberated is equivalent to the iodine consumed in oxidising sodium sulphite, and to half that consumed in oxidising sodium hydrosulphide. Two successive titrations of the same sample using different indicators are not recommended;

it is preferable to take separate samples for each titration. Titration of the supernatant liquid over a precipitate such as barium carbonate is also liable to yield inaccurate results. A. GEAKE.

Ripening of viscose solutions. J. D'ANS and A. JÄGER (Kunstseide, 1926, 8, 17—19, 43—46, 57—59; Chem. Zentr., 1926, I., 3108).—The number of thiocarbonate groups combined with cellulose may be determined by titration with iodine. The xanthate content diminishes regularly, the velocity increasing with rise of temperature. All carbon disulphide combined with cellulose reacts with iodine. The sensitiveness of a viscose solution to electrolytes depends on the molecular complexity of the cellulose, the number of xanthate groups combined with cellulose, the amount of electrolytes which are always present in viscose solutions, the amount of free sodium hydroxide, and the temperature. In the determination of ammonium chloride ripeness the sodium hydroxide content is important, and the appearance of spontaneous coagulation is not sharply defined. Loss of carbon disulphide molecules does not necessarily imply a diminution in the size of the molecule. In following changes of viscosity the sodium hydroxide content must be the same as that of technical viscose solutions. Viscosity and ammonium chloride ripeness give comparable figures for the condition of viscose solutions. Fresh viscose solution, after mixing with ammonium chloride, generally contains free caustic alkali, the amount of which increases the older the solution. At the coagulation point perfectly fresh viscose solution, containing little alkali, has an excess not of free alkali, but of ammonium chloride. With increasing alkali content of the viscose solution the amount of ammonium chloride used in double decomposition is so great that the cellulose complex is precipitated in a solution which still contains alkali. The stability of viscose solutions towards coagulation increases with increasing xanthate content, and this effect is greater than the influence of sodium hydroxide, especially in fresh solutions. Solutions rich in sodium hydroxide, and containing the same amount of xanthate, have an apparently smaller ammonium chloride ripeness. In very ripe solutions, which almost coagulate spontaneously, the effect of the xanthate content on the ammonium chloride ripeness almost disappears. The number of xanthate groups found by titration with iodine increases with the amount of carbon disulphide used. A. GEAKE.

Increasing the suspension of [paper] coating mixture by the addition of colloids. B. K. STEADMAN (Pulp and Paper Mag., 1926, 24, 307).—Attempts to preserve the suspension of casein coating mixtures by adding other substances of a colloidal nature showed that sodium silicate is effective and fairly satisfactory, but tends to increase the viscosity of the mixture to an undesirable extent. Gum arabic has a similar action, but causes pin-holes to appear in the coating. D. J. NORMAN.

PATENTS.

Washing or otherwise treating wool and other fibrous textile materials. E. C. DUHAMEL, and

COMP. GÉN. DES IND. TEXTILES (E.P. 230,808, 18.2.25. Conv., 11.3.24).—Wool, silk, and other textile materials are passed successively through a series of washing units, each unit being followed by a squeezer. The detergent liquor squeezed from the material leaving each unit flows, with or without partial purification by centrifugal separation of fatty substances and solids, to the adjacent unit containing dirtier liquor. The washing units are very small (about 5 litres capacity) so that they merely ensure the thorough impregnation of the textile material with washing liquor. The period of contact of the washing liquor with the textile materials is thus small, and the detergent power of the liquor is thereby increased. A. J. HALL.

Manufacture of felt. C. and E. PICHARD (E.P. 248,343, 4.2.26. Conv., 28.2.25).—Carroting is effected by treating the hair before or after removal from the skin with a solution of carbamide or derivatives thereof, preferably the nitrate. D. J. NORMAN.

Fabrics or materials and the manufacture thereof. BRIT. CELANESE, LTD., T. C. WOODMAN, and W. A. DICKIE (E.P. 249,946, 10.1.25).—Water-proof and gasproof properties and other special effects may be imparted to fabrics composed of filaments or fibres of esters or ethers of cellulose, either alone or in association with other fibres—animal, vegetable, or artificial—by subjecting the fabric to heat and pressure in order to cause a partial or complete coalescence of the fibres. Plasticising agents such as monomethylxylenesulphonamide facilitate the process, and may either be added to the spinning solution or dissolved in a volatile solvent, which is a non-solvent of cellulose derivatives, e.g., benzol, and sprayed or otherwise distributed over the fabric before the heating operation; 20 pts. of monomethylxylenesulphonamide dissolved in 100 pts. of benzol is a suitable quantity per 100 pts. of cellulose acetate. The temperature, pressure, and duration of the treatment depend on the extent to which it is desired to close up the pores of the fabric; for example, the fabric may be passed slowly between calender rolls at 100—180° under pressures varying from 300 to 600 lb. per sq. in. D. J. NORMAN.

Manufacture of artificial materials [from *N*-substituted cellulose thiourethanes]. L. LILLENFELD (E.P. 248,994, 9.7.25. Conv., 30.5.25. Addn. to 231,806; B., 1925, 985).—Artificial filaments and films of high lustre and of good tensile strength, both in the wet and dry state, are obtained from *N*-substituted cellulose thiourethanes by using strong acids, e.g., sulphuric acid of 25—65% strength, phosphoric acid of 45—70% strength, hydrochloric acid of 20—35% strength, or acetic acid of 70—100% strength, as precipitating agents instead of the weak acids and other coagulants suggested in the original patent. Neutral or acid salts or organic compounds such as glucose or glycerin may be added to the acid bath, the temperature of which may be below, at, or somewhat above (e.g., 30°) the ordinary temperature. The freshly-precipitated threads are plastic and may be stretched before they are fixed by washing out the

acid. Increased tensile strength is imparted by steaming the washed threads either before or after drying. A suitable spinning solution contains 10–16% of an *N*-substituted cellulose thiourethane (prepared as described in E.P. 231,801; B., 1925, 955) in 6–8% caustic soda solution. The spinning bath may then consist of 50% sulphuric acid maintained at the ordinary temperature or at 5–8°.

D. J. NORMAN.

Spinning viscose. J. C. HARTOGS (U.S.P. 1,573,062, 16.2.16. Appl., 30.6.24).—Evolution of hydrogen sulphide from the viscose spinning bath is avoided by adding a ferric salt, which is decomposed to sulphur and ferrous sulphate. The presence of sulphur in the thread prevents it from breaking in the twisting process, while ferrous sulphate increases the lustre of the threads and prevents their adhesion.

R. B. CLARKE.

Cellulose nitrate composition. S. J. CARROLL, Assr. to EASTMAN KODAK Co. (U.S.P. 1,580,189, 13.4.26. Appl., 9.7.24).—A film-forming composition, substantially free from camphor and from high-boiling esters used as substitutes for this, consists of nitrocellulose, monochloronaphthalene, and butyl alcohol.

A. GEAKE.

Artificial silk from viscose. W. MENDEL, Assr. to S. A. NEIDICH (U.S.P. 1,580,843–4, 13.4.26. Appl., 28.5.25).—Viscose is projected (A) into a soft coagulating medium and then into an aqueous phosphate solution, or (B) directly into an aqueous phosphate solution.

A. GEAKE.

Solvents for nitrocellulose and cellulose acetate. PATHÉ CINÉMA (ANC. ETABL. PATHÉ FRÈRES) (F.P. 601,546–7, 30.10.24).—Mixtures of an alcohol with acetone or with the acetic or formic ester of the alcohol are used as solvents for (A) nitrocellulose, or (B) cellulose acetate, for the manufacture of threads, films, varnishes, or artificial leather. In examples nitrocellulose is dissolved in a mixture of 80 pts. by vol. of absolute alcohol and 20 pts. of acetone, or of 80 pts. of amyl alcohol with 20 pts. of amyl acetate.

A. GEAKE.

Manufacture of lignone derivatives and their application. C. F. CROSS and A. ENGELSTAD (E.P. 248,834, 10.12.24).—New lignone derivatives are obtained by the action of acid condensing agents, e.g., hydrochloric acid, either alone or in the presence of phenolic compounds, on lignone or its derivatives, more particularly those sulphonated derivatives resulting from the acid digestion of lignocellulose by the processes described in E.P. 202,016 and 229,002 (B., 1923, 971A; 1925, 312). The new lignone derivatives may be used in the dyeing industry, or as a basis for the manufacture of writing and printing inks. Example: 100 pts. of lignone extract containing about 37 pts. of solid matter are mixed with 10 pts. of hæmatein and 10 pts. of concentrated hydrochloric acid and heated at 95–100° until the mixture occupies about one-half of its original bulk. The resulting product may be used in the dyeing industry in the same manner as

ordinary logwood extract. Writing ink is made, for example, as follows:—A mixture containing 100 pts. of extract (37% of solid matter), 5 pts. of pyrogallol, 2.5 pts. of gallic acid, and 6 pts. of concentrated hydrochloric acid is evaporated at 95–100° to one-half of its original bulk; an equal volume of water is added and in this solution are dissolved successively 10 pts. of crystalline ferrous sulphate and 2 pts. of a soluble blue, such as Nigrosine. The resulting concentrated ink is diluted 7–9 times with water when required for use as a writing ink. The fluidity of the ink may, if desired, be regulated by the addition of a gum. Inks made in this manner tend to become fixed in an insoluble state on drying; if, however, this property is not required, the concentrated lignone extract may be used without previous treatment with condensing agents.

D. J. NORMAN.

Digestion of paper pulp or the like and concentration of the spent lye. J. HOLMES (E.P. 248,926, 13.3.25).—The installation comprises a digester, a closed feed reservoir provided with connexions to the top and bottom of the digester, and a multiple-effect evaporator for concentrating the liquor from the reservoir. The first stage of the evaporator is heated by the steam from the digester and reservoir, succeeding stages by the steam from the preceding stage. Heat exchangers placed in the steam connexions between the stages of the evaporator supply heat to the wash liquor and to fresh caustic liquor. If desired the reservoir may be externally heated. Provision is made for introducing steam direct into the first stage of the evaporator.

D. J. NORMAN.

Gas and heat recovery from [wood pulp] digester blow-off exhaust vapours. L. B. DECKER (U.S.P. 1,576,643, 16.3.26. Appl., 22.6.25).—The exhaust vapour from the digester is passed into a chip bin which is arranged to communicate with the digester inlet.

D. J. NORMAN.

[Sulphite-pulp] digester. J. W. ALSTYNE (U.S.P. 1,579,261, 6.4.26. Appl., 3.10.25).—A digester for making sulphite wood pulp has a conical base fitted at the lower end with a discharge pipe; a steam inlet pipe passes centrally up this discharge vent and extends into the bottom of the digester.

D. J. NORMAN.

Hydrating cellulose fibres [for paper making]. J. A. DE CEW, Assr. to PROCESS ENGINEERS, INC. (U.S.P. 1,580,814, 13.4.26. Appl., 7.6.24).—Fibres are beaten with a solution of an alkali until pliable, and the alkali is then neutralised with aluminium sulphate.

A. GEAKE.

Production of pulp and other products from wood. L. BRADLEY and E. P. MCKEEFE (U.S.P. 1,581,671, 20.4.26. Appl., 3.11.21).—Wood is treated with a hot aqueous acid to convert a part of the water-insoluble constituents into fermentable sugars, without cooking the wood or injuring its pulping properties. After separating the sugars, the wood is subjected to a chemical pulping operation.

A. GEAKE.

Producing paper pulp. E. LAMBERT and W. E. MATTHEWS (U.S.P. 1,582,336, 27.4.26. Appl., 26.8.22).—Wood stock is comminuted, boiled for 1 hr. with sufficient water to cover it and 1 lb. of potassium hydroxide per 100 lb. of stock, washed to remove dirt, and re-ground. A. GEAKE.

Stencil sheets. S. HORII (E.P. 250,798, 10.7.25).—A coating for a stencil sheet of fibrous material consists of a solution of an ester or mixture of esters of polysaccharides, such as starch acetates or cellulose acetates, nitrates, or xanthate in suitable solvents such as alcohol, ether, or amyl acetate, to which naphthenic acid glycerides and other tempering agents such as fatty acids, fats, paraffin, ceresin, or other waxes are added. Such a coating is soft but not sticky, stores well, and does not become hard in cold weather. A. GEAKE.

De-inking paper stock. J. E. PLUMSTEAD, Assr. to JESSUP & MOORE PAPER Co. (U.S.P. 1,576,994, 16.3.26. Appl., 16.11.25).—The disintegrated stock is agitated with an alkali; chlorine is then introduced and the resulting mixture subjected to heat. D. J. NORMAN.

Waterproofing cellulose, paper, and the like. R. WOLFFENSTEIN and A. MARCUSE (G.P. 426,428, 30.5.14).—Cellulose and materials containing it are waterproofed by treatment with thionyl chloride. A. R. POWELL.

Economical disposal of waste sulphite [cellulose] liquor. A. M. THOMSEN, Assr. to CROWN WILLAMETTE PAPER Co. (U.S.P. 1,582,317, 27.4.26. Appl., 13.5.24).—Waste liquor from sulphite-cellulose manufacture is concentrated to a syrup and treated with a porous, combustible material. Impregnation of the material with the syrupy liquid is assisted by the former being hotter than the latter. A. GEAKE.

Manufacture of a non-deliquescent body from sulphite-cellulose [waste] liquor. A. G. BLOXAM. From A.-G. F. ANILIN-FABR. (E.P. 251,019, 22.1.25).—See F.P. 592,119; B., 1926, 188.

Making pulp for paper. B. S. SUMMERS (U.S.P. 1,584,902, 18.5.26. Appl., 22.12.23).—See Can. P. 246,537; B., 1926, 153.

De-inking printed paper. L. E. GRANTON (U.S.P. 1,585,092, 18.5.26. Appl., 26.3.24).—See E.P. 222,160; B., 1924, 977.

Apparatus for drying fabrics, paper, etc. J. J. LYTH (E.P. 251,470, 29.6.25).

Preparing rubber-coated fabrics and apparatus therefor. DUNLOP RUBBER Co., LTD., and W. J. DEXTER (E.P. 251,357, 2.2.25).

Utilising waste heat from gas from sulphur burners etc. (U.S.P. 1,581,511).—See VII.

Combinations of rubber and paper (U.S.P. 1,567,646).—See XIV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Conditions governing the bleaching of wool with hydrogen peroxide. S. R. and E. R. TROTMAN (J. Soc. Dyers and Col., 1926, 42, 154—157).—A determination of the factors which are responsible for the loss of weight and damage to the epithelial scales of ordinary and moderately chlorinated wool fabric during bleaching at 55° by means of solutions of sodium peroxide having a known p_H of 9 to 13. The bleaching solutions were prepared by the addition of sodium silicate to hydrogen peroxide. Bleaching causes no marked increase in the proportion of damaged fibres of ordinary wool but a distinct increase in chlorinated wool, especially when the bleaching liquor has a p_H exceeding 10, the increase being proportional to the p_H . The loss of weight produced by bleaching increases with increase of p_H and is much greater for chlorinated than for ordinary wool. A better white is obtained in the more alkaline bleaching liquors and the best colour, together with the least structural damage to the fibres, is obtained with p_H 10. A mixed indicator containing dinitrodiazoaminobenzene (cf. Brandt, B., 1901, 711) and phenolphthalein or phenolthymolphthalein is suitable for controlling the acidity of bleaching liquors. For example, an indicator containing 5 c.c. of a saturated alcoholic solution of dinitrodiazoaminobenzene and 0.1 c.c. of a 0.4% alcoholic solution of phenolphthalein gives a range of colours: p_H 8 yellow, p_H 9 reddish-yellow, p_H 9.5 redder yellow, p_H 10 orange, p_H 11 red, and p_H 12 deeper red. A. J. HALL.

From "hot-bleach" to "cold-bleach." [Development of the Mohr process of bleaching.] R. MOHR (Textilber., 1925, 6, 909—912).—The Mohr process of bleaching in which cellulosic textile materials (fabric may be treated in open width) are successively bleached under a hydraulic pressure of 2—3 atm. in the same container with a chlorine bleaching solution, soured, then further bleached with a liquor containing a peroxide and afterwards thoroughly washed, is more economical in labour and particularly lime than the usual processes of kiering and bleaching with hypochlorites. Cold or warm liquors may be used; the textile material suffers only a small loss in weight and is uniformly and thoroughly purified so that it is very absorbent and takes up dyes evenly. A. J. HALL.

[Comparison of the] "hot-bleach" and "Mohr-bleach" processes. M. FREIBERGER (Textilber., 1926, 7, 148, 226—227).—The advantages claimed for the Mohr process of bleaching (cf. preceding abstract) are refuted. The Mohr process removes pectic and protein impurities from cotton, but only partially removes fats and waxes. The last-named impurities retard penetration of the cotton by the bleaching liquors so that the use of excess of bleaching agents is necessary; a considerable amount of oxycellulose is thereby formed. Cotton bleached by the Mohr process becomes yellower after extraction with light petroleum and the extracted cotton becomes still yellower when

steamed. The yellowing of the bleached cotton during storage is hindered by the presence of the fats and waxes incompletely removed during bleaching. Cotton fabric suffers a 5% loss of weight during bleaching by the Mohr process whereas the "hot-bleach" process of Thies-Herzig produces a loss of 12%; fabric bleached by the Mohr process thus contains impurities, which are revealed by dyeing it with Alizarin. The Mohr process of bleaching produces an excessive loss of strength and is really but a partial bleach which can be better obtained by the usual methods or in less elaborate plant.

A. J. HALL.

Test for mercerised cotton. H. MENNELL (*J. Text. Inst.*, 1926, 17, τ 247).—The test depends on the fact that mercerised cotton is rendered sensitive to substantive dyes by treatment with sulphuric acid, especially in the presence of formaldehyde. The reagent consists of 320 c.c. of sulphuric acid of *d* 1.6, diluted with 260 c.c. of 40% formaldehyde, and the dye preferred is Chlorazol Sky Blue G.W., used in a boiling bath made alkaline with sodium carbonate. The sample to be tested, and samples known to be mercerised and unmercerised respectively, are steeped in the acid for 2 min., then washed and dyed. If the dye-bath is of such a concentration as to give a 0.1% shade on unmercerised cotton it will give about a 0.8% shade on fully mercerised material, and the degree of mercerisation can be judged from the intermediate shades. Dyed material may be stripped with hypochlorite or hyposulphite before testing.

J. C. WITHERS.

Simple and reliable test for mercerisation. R. W. KINKEAD (*J. Text. Inst.*, 1926, 17, τ 213—219).—The material is stained with Methylene Blue and then boiled with sodium carbonate and a trace of iodine. Details of concentrations, which are varied slightly for different cellulosic fibres, must be observed. If the material has not been mercerised it will remain blue, but if mercerised with caustic alkalis it will become reddish-purple, and the new shade will afford a clue to the experienced observer of the concentration of the alkali employed. The test may be applied to a dyed material after stripping and is not affected if the fibre has been mildly bleached or soaked in acids. If the mercerisation has been carried out by means of acids, however, the colour change will be much less definite, but such materials respond readily to Huebner's iodine tests (*J.S.C.I.*, 1908, 27, 105).

J. C. WITHERS.

Dyeing of wool with indigo. F. PETERHAUSER (*J. Soc. Dyers and Col.*, 1926, 42, 152—154).—Indigosol O (cf. Vaucher and Bader, *B.*, 1924, 864) is superior to indigo since it is easily soluble in water and may be applied to wool by the methods used for acid dyes; it does not oxidise in the dye-bath and allows the accurate matching of desired shades. Its subsequent development by oxidation is not satisfactory when hydrogen peroxide, a persulphate, or atmospheric oxygen is used. Indigosol

OR and O4B are similar soluble derivatives of monobromo- and tetrabromo-indigo respectively.

A. J. HALL.

Influence [on dyeing] of the degree of dispersion of dyes in dye liquors. R. ROESTEL (*Textilber.*, 1926, 7, 228—230).—More uniform and deeper shades are obtained on vegetable, Chardonnet silk, and wool fibres by means of direct, mordant, basic, vat, and sulphur dyes when a dispersing or wetting-out agent, particularly Tetracarnite (Sandoz), is added to the dye liquors. Chardonnet silk is satisfactorily and evenly dyed with the insoluble bases of basic dyes such as Chrysoidine G extra, Rhoduline Heliotrope, and Methylene Blue RR dispersed in aqueous solutions containing Tetracarnite, and under similar conditions the shade obtained on wool by means of the base of Brilliant Green A extra is brighter than, and as fast to rubbing as, that obtained by dyeing wool in the usual manner in the presence of acetic acid. The presence of Tetracarnite assists the dyeing of cotton with Alizarin more than similar additions of ammonia or Turkey-red oil. Benzo Fast Red L dyed on cotton is not appreciably affected by treatment with boiling aqueous solutions containing 10% of sodium carbonate or 33% of Tetracarnite or 10% of Turkey-red oil and 10% of sodium carbonate, but under similar conditions dyeings of Columbia Blue 3G are considerably stripped. Benzo Fast Red L and Patent Blue are readily removed from wool by treatment with a 50% solution of Tetracarnite but are much less affected by similar treatment with aqueous solutions containing sodium carbonate or Glauber's salt and sulphuric acid.

A. J. HALL.

Dyeing materials used for the manufacture of buttons and the like. FLEMMING (*Deuts. Färber-Ztg.*, 1925, [2]; *Textilber.*, 1926, 7, 175—176).—For the production of satisfactory shades on horn, the material is steeped overnight in a 10% solution of sodium carbonate, then washed in water, mordanted by immersion for 6—8 hrs. in a solution containing one or more of the salts of copper, iron, and chromium, and afterwards dyed in a cold solution containing a suitable oxidation fur dye (*e.g.*, *p*-phenylenediamine and hydrogen peroxide); mordanting may be effected before or after dyeing and prevents subsequent sublimation of dye from the horn. Ivory is dyed by methods similar to those used for horn but ivory nut materials may be dyed in boiling liquors containing basic, direct, or sulphur dyes. Ivory is also dyed with oxidation colours by the method described above. Galalith (prepared from casein) is preferably dyed in warm solutions containing acid or direct dyes, whereas Bakelite materials (phenol-formaldehyde condensation products) are satisfactorily coloured by means of basic dyes.

A. J. HALL.

Chemical analysis of cotton. Absorption of Methylene Blue from buffered solutions. CLIBBENS and GEAKE.—See V.

Chemical analysis of cotton. Scouring losses. FARGHER and HIGGINBOTHAM.—See V.

PATENTS.

Raw-stock-dyeing apparatus. F. M. and G. W. MORTON (U.S.P. 1,571,863, 2.2.26. Appl., 6.4.25).—The dye-vat has a dye-liquor inlet port in the upper portion of one side wall and a removable curved cover plate to which a perforated dye-liquor distribution plate is attached on its under side. The lower side of the inlet port is inclined inwards and upwards, the upper side being horizontal and the other sides diverging outwards. The edge of the dye-liquor distribution plate is inclined downwards so that it is in line with the upwardly inclined lower side of the inlet port. Dye-liquor entering through the inlet port is deflected upwards by the edge of the distribution plate and afterwards percolates downwards through the plate on to textile material below. A. J. HALL.

Dye vat. F. M. and G. W. MORTON (U.S.P. 1,571,864, 2.2.26. Appl., 26.10.25).—The vertical side walls of the vat are attached by means of an outer flange to a horizontal bottom plate provided with a horizontal dye-liquor outlet port. A horizontal perforated plate in the same plane as the outer flange rests on lugs projecting upwards from the bottom plate, and the latter is inclined downwards from the centre to its outer edges. A. J. HALL.

Producing fast dyeings on wool. BADISCHE ANILIN- & SODA-FABR., Assees. of H. KRZIKALLA, H. KÄMMERER, and J. NÜSSLEIN (U.S.P. 1,579,121, 30.3.26. Appl., 3.8.25).—Wool or other animal fibre is impregnated with a sulphonated azo-dye component containing an amino- or hydroxy-group or both so that it can be coupled on the fibre with a diazo-compound. To obtain a fast colour the resulting dye must be difficultly soluble, hence the number of sulphonic groups must be reduced to a minimum. Chromed and unchromed dyes which will couple may also be employed to impregnate wool. For example, wool is boiled for 1 hr. in a bath containing 1:5-di-[6-sulpho-2-hydroxy-3-naphthoylamido]naphthalene, and is then treated with diazotised *m*-xylylidine when a bluish-red shade fast to washing, milling, and light is developed. T. S. WHEELER.

Dyeing brown shades on [cotton] fibres. COMP. NAT. DE MAT. COL. ET MANUF. DE PROD. CHIM. DU NORD RÉUNIS, ETABL. KUHLMANN (F.P. 595,705, 27.6.24).—Cotton is dyed in brown shades fast to washing, alkali, chlorine, and light by impregnation with a solution containing products obtained by the condensation of formaldehyde and aniline or its homologues in acid solution, being afterwards passed through a solution of caustic soda or sodium carbonate, then washed, oxidised with a dilute solution of sulphuric acid containing a dichromate or persulphate, washed, and soaped. The resulting shades may also be diazotised and developed with β -naphthol or coupled with diazo-compounds. Alternatively cotton is impregnated with a solution containing lactic acid, the above described condensation products, sodium chlorate, sodium acetate,

and copper sulphate or a vanadium salt, then oxidised during 6 hrs. at 40° with a solution of a dichromate; white and coloured resists may be obtained by after-printing with a reducing agent, e.g., a sulphite, bisulphite, or hyposulphite, and suitable dyes. Brown effects are also obtained by printing cotton fabric with a paste containing the condensation products and gum tragacanth thickening, drying, fixing in an alkaline solution, and developing the shade by oxidation with a persulphate; alizarin and vat dyes may be printed and developed simultaneously by oxidation. A. J. HALL.

Dyes and dyeing process. BADISCHE ANILIN- & SODA-FABR. (F.P. 599,566, 8.5.25. Conv., 30.6 and 24.7.24 and 21.1.25).—Fast dyeings on cotton and artificial silks (cellulose acetate silk excepted) capable of being after-treated with zinc chloride, lead acetate, etc., are obtained by direct application in the presence of sodium chloride or Glauber's salt of suitable substantive or non-substantive dyes which have been previously treated with chromium salts, or by the application of suitable dyes in the presence of chromium salts (e.g., fluoride, acetate, oxalate, and formate). Suitable dyes include the chromium compounds of azo dyes prepared from mixtures of 2:5- and 2:8-naphthylaminesulphonic acids and salicylic acid (yellow); from *m*-aminobenzoyl-*p*-aminosulphosalicylic acid and 1:3-acetaminonaphthol-4:6-disulphonic acid (red); and from benzidine, *oo'*-dianisidine, or diaminodiphenylcarbamide and salicylic acid (golden-yellow to yellowish-orange). The chromium compound of the triphenylmethane dye, Chromoxan Blue R, dyes blue. A. J. HALL.

Azo dyes [for cellulose acetate]. BADISCHE ANILIN- & SODA-FABR. (F.P. 600,106, 26.6.25. Conv., 4.9.24).—Cellulose acetate silk is dyed in fast shades from aqueous solutions or suspensions of azo dyes containing one or more monohydroxyethylamino-groups, but no sulphonic acid group. A suitable dye is obtained by coupling diazotised *p*-nitroaniline or 3-nitro-*p*-toluidine with hydroxyethylaniline in acetic acid solution. A. J. HALL.

Formation of pigment azo dyes on vegetable fibres. L. CASSELLA & Co., Assees. of G. KALISCHER and K. KELLER (G.P. 422,467, 12.4.24).—Very deep shades fast to rubbing are obtained by impregnating cotton with azoxy-derivatives (related to azo-derivatives) of arylides of β -hydroxynaphthoic acid in alkaline solution and afterwards coupling with a diazo-compound. A suitable azoxy-compound is obtained by alkaline reduction of the nitroanilide of β -hydroxynaphthoic acid. A. J. HALL.

Dyeing artificial silks manufactured from acidyl celluloses, cellulose ethers, and related products. FARBENFABR. VORM. F. BAYER & Co., Assees. of W. DUISBERG, W. HENTRICH, and L. ZEH (G.P. 423,601, 25.12.23. Addn. to 418,940; cf. E.P. 225,862; B., 1925, 583).—Azo dyestuffs suitable for dyeing cellulose acetate silk are prepared by coupling diazotised amines or their substituted

derivatives with aralkylarylamines or their substituted derivatives containing carboxylic or sulphonic acid groups. For example, yellow, bordeaux, and orange-red dyes are obtained by coupling 5-nitro-2-anisidine and benzyl-*o*-sulphanilic acid, 2:4-dinitroaniline and 4-sulphobenzyl-2-toluidine, and 2:4-dinitroaniline and benzylanthranilic acid respectively.

A. J. HALL.

Manufacture of effect threads. FARBENFABR. VORM. F. BAYER & Co., Assees. of G. RUDOLPH (G.P. 423,602, 27.3.24. Addn. to 407,834; B., 1925, 240).—Fabrics capable of being dyed in more than one colour are woven from ordinary silk and from silk yarn having a diminished affinity for dyestuffs produced by treatment for 2 hrs. with tannic acid and then for 1 hr. with tin salts in the presence of formaldehyde.

A. J. HALL.

Finishing and ornamentation of textile materials. J. HUEBNER (E.P. 250,283, 10.10.24).—Cellulose is deposited upon or within textile materials composed wholly or partly of animal fibres, such as wool or silk, by printing, padding, or stencilling such materials with suitable cuprammonium solutions of cotton or other cellulosic substance and afterwards treating them with a precipitating agent, such as hydrochloric acid or caustic soda, the residual copper being removed simultaneously or by a subsequent process; the textile materials are dyed or printed before or after the deposition of cellulose. For example, fabric is printed with a 2% cuprammonium solution of bleached cotton (30 g. of copper per litre of solution), dried (optional), soured in hydrochloric acid of *d* 1.05, whereby the cellulose is deposited and copper removed, and then dyed preferably without previous drying. The effects produced are very close imitations of coloured woven fabrics (cf. E.P. 227,480; B., 1925, 240).

A. J. HALL.

Lignone derivatives (E.P. 248,834).—See V.

Starch preparations (E.P. 244,708).—See XVII.

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

Recovery of selenium from lead chamber slimes. W. STAHL (Chem.-Ztg., 1926, 50, 280).—The most economical process for treating the slimes which accumulate in the lead chamber process of making sulphuric acid consists in heating them in cast iron pots with strong sulphuric acid and a little sulphur trioxide, the fumes evolved being returned to the chamber circuit. The reaction product is extracted with dilute sulphuric acid, the insoluble residue separated in a filter-press and smelted for the recovery of lead, and the solution treated with sulphur dioxide for the precipitation of selenium. The acid filtrate from the selenium is concentrated and used again in the process until it becomes too foul. Addition of lime to the foul liquor neutralises the acidity and precipitates calcium sulphate, which may be

used for the manufacture of plaster of Paris etc., and the liquor may be safely run to waste.

A. R. POWELL.

Sensitiveness of diphenylamine as a reagent for nitric acid. R. KRAUER (Chimica e Ind. (Brazil), 1926, 1, 143—144).—The solution of diphenylamine in sulphuric acid used as a reagent for nitric acid gives, after keeping, a positive reaction with pure sulphuric acid. This is probably due to the absorption of traces of nitric acid from the atmosphere of the laboratory. By using crystals of diphenylamine instead of the solution in sulphuric acid as little as 0.00021% of nitric acid can be detected in sulphuric acid. The reaction is intensified by dilution with water.

G. W. ROBINSON.

Constitution of bleaching powder. H. DITZ and B. NEUMANN (Z. Elektrochem., 1926, 32, 231—240).—Polemical between Ditz and Neumann (cf. Neumann and Hauck, B., 1926, 190).

M. S. BURR.

Manufacture of sodium chromate. N. F. YUSHKEVICH (Trans. Inst. Econ. Min. Petr. (Russia), 1925, [13], 1—29; Chem. Abstr., 1926, 20, 1305).—When 37.85% of chrome iron ore (44.6% Cr₂O₃) is roasted with 24.3% of sodium carbonate (98.5% pure) and 37.85% of lime (90% pure), the formation of chromate at 700° is slow, whilst at 1160°, 95% of the chromium is oxidised in 30 min. The heating must be rapid, the stirring continuous, and the temperature of the heating gas must not exceed 1260°.

A. A. ELDRIDGE.

Analysis of lime. J. C. BAILAR (Ind. Eng. Chem., 1926, 18, 389—390).—The determination of calcium oxide in the presence of carbonate, *i.e.*, the available lime, may be carried out by adding to a well hydrated portion of the sample, either excess of iodine and titrating with sodium thiosulphate, or excess of zinc chloride and titrating with sodium hydroxide using an indicator consisting of phenolphthalein and alizarin-cyanine-green C. The methods are quicker than the well-known sucrose method, but the iodine method is expensive when many determinations are required. The author considers that the zinc chloride method is the most satisfactory for the sugar industry.

G. T. PEARD.

Stabilisation of hydrogen peroxide solutions and of perborates. KARSTEN-SALMONY (Chem.-Ztg., 1926, 50, 280—281).—A review of the patent literature.

A. R. POWELL.

Determination of traces of carbon monoxide. H. DAVIES and H. HARTLEY (J.S.C.I., 1926, 45, 164—168 T).—Iodine pentoxide, prepared by the chloric acid method, is employed and the apparatus and procedure adopted are described. The pentoxide is shown to be stable over the temperature range (120—150°) used in the determination, but, owing to the carrying over of oxy- or hydroxy-compounds of iodine, generally a small positive "blank" is found, even when air free from carbon monoxide is passed over heated iodine pentoxide. The procedure which results in a "blank" not exceeding 1—2 parts per

million is detailed, and the importance of using dehydrated material is emphasised. A special form of absorption vessel for completely absorbing and retaining the iodine liberated during a determination, is described and the method of titration is dealt with.

Determination of small quantities of hydrogen in gaseous mixtures. P. LEBEAU and P. MARMASSE (Compt. rend., 1926, 182, 1086—1087).—The determination of a small percentage of hydrogen in a mixture of gases may be carried out accurately by passing the mixture over silica gel (previously heated at 150° in a vacuum) cooled to the temperature of liquid air. The only gases not fixed by the gel under these conditions are hydrogen, helium, neon, and traces of methane, nitrogen, oxygen, and carbon monoxide. This mixture is then analysed by the method previously described (Lebeau and Damiens, A., 1913, ii, 253, 349: B., 1913, 126, 186, 277). H. J. EVANS.

Influence of rate of stirring on reaction velocity. HUBER and REID.—See I.

PATENTS.

Producing commercial hydrochloric acid. H. A. GALT, Assr. to PITTSBURGH PLATE GLASS CO. (U.S.P. 1,581,436, 20.4.26. Appl., 23.3.23).—A concentrated solution of calcium chloride is treated with sulphuric acid of such strength as to produce precipitated calcium sulphate; after leaving the mixture to react, it is filtered to obtain a concentrated solution of hydrochloric acid. H. ROYAL-DAWSON.

Utilising waste heat from gas [from sulphur burners etc.]. H. CLEMM and A. SCHNEIDER, Assrs. to ZELLSTOFF-FABR. WALDHOF (U.S.P. 1,581,511, 20.4.26. Appl., 3.4.22).—The heat of gases from sulphur burners and pyrites furnaces is transmitted to a heat-exchanging device through which fresh sulphite lye is forced under pressure, the lye being then conveyed to an apparatus for boiling cellulose. The whole of the system containing fresh lye, whether heated or not, is completely closed, thus retaining a certain pressure therein in order to avoid any loss of sulphur dioxide and any noxious change of the lye.

H. ROYAL-DAWSON.

Treating waste liquors containing cyanogen compounds. J. DENIS (E.P. 250,824, 18.9.25).—The liquor is treated with lime water and ferrous sulphate, and then with carbon dioxide to precipitate iron ferrocyanide as a blue sludge, which is filtered off. The filtrate is treated with barium hydroxide or the like to precipitate the excess of carbonic acid, the precipitate carrying down the last traces of iron ferrocyanide. When the liquors contain phenols and hydrocarbons these are separated by their difference of density before or after adding the reagents mentioned in the first stage of the process and are conveyed into a closed reaction chamber into which is passed a current of air or other gas, which is carburetted by the phenols and hydrocarbons and may be utilised as fuel gas. H. ROYAL-DAWSON.

Obtaining alkali thiosulphate from solutions containing alkali sulphide. F. RÜSBERG, Assr. to RHENANIA VEREIN CHEM. FABR. A.-G. (U.S.P. 1,567,755, 29.12.25. Appl., 28.5.25).—Solutions containing sodium sulphide are treated with a gas containing 32% by vol. of sulphur dioxide in the presence of sodium sulphite. H. ROYAL-DAWSON.

Manufacturing sodium fluoride from silicofluorine compounds. W. SIEGEL (U.S.P. 1,581,819, 20.4.26. Appl., 9.5.25).—Potassium silicofluoride is treated with an alkaline potassium compound; after separating the silicic acid formed, the potassium fluoride so obtained is further treated with a sodium compound to transform the potassium fluoride into sodium fluoride, which is then separated from the potassium compound. H. ROYAL-DAWSON.

Manufacture of anhydrous chlorides. J. GOHIN (F.P. 601,612, 4.11.24).—Solid chlorides are sublimed in one or two stages without condensation in a chamber provided with a lining resistant to the action of hot chlorine, in such manner that the sublimate commences to condense only at the end of the apparatus. J. S. G. THOMAS.

Separation of barium and radium salts. I. BACHILOV (F.P. 601,938, 7.8.25).—The solution of the double salt of barium and radium is crystallised after addition of salts containing the same ion as the solution, and which do not form salts soluble in the solvent employed, with the barium and radium salts present. J. S. G. THOMAS.

Production of alumina and potassium salts from leucitic rocks. F. GIORDANI and U. POMILIO (G.P. 426,832, 10.9.21).—Solution of constituents other than potassium and aluminium in the decomposition of leucite is reduced to a minimum by treating the rock with a small quantity of acid at first, and adding more acid from time to time as solution proceeds, the total quantity of acid added being somewhat less than that required for complete solution of the aluminium and potassium.

L. A. COLES.

Apparatus for expelling ammonia from ammoniacal liquor. BAMAG-MEQUIN A.-G. (G.P. 426,863, 27.8.24).—The apparatus comprises a series of columns for expelling free ammonia, provided with serrated bells having their overflow devices high up, so that the bells lie completely below the level of the liquid, thereby exposing a large surface for evaporation, and a series of columns for expelling fixed ammonia in which intimate admixture of lime with the liquor is effected by the provision of narrow ducts running parallel to the periphery of the bells. The columns are divided into two sections by a sloping partition, the lower section serving as a preheater and storage chamber for the liquor. Before leaving the columns, the gas passes through a chamber in which liquid particles are separated as completely as possible, e.g., by the provision of baffle plates, the separated liquid passing to the previous column.

L. A. COLES.

Decomposition of material containing selenium. MANSFELD-A.-G. FÜR BERGBAU & HÜTTENBETRIEB, and K. WAGENMANN (G.P. 426,669, 22.7.25).—Mixtures of the material with sodium bisulphate, or with sulphuric acid and alkali sulphates, are heated above 700°, and the selenium which distils over is condensed. Anode sludge, before subjection to the above treatment, is dried and heated with sufficient concentrated sulphuric acid to convert into sulphates all the constituents soluble in sulphuric acid.

L. A. COLES.

Production of zirconium oxide. H. S. COOPER and L. P. BENSING, Assrs. to KEMET LABORATORIES Co. (U.S.P. 1,582,126, 27.4.26. Appl., 1.3.20).—Anhydrous zirconium chloride, free from iron, is hydrolysed and heated at gradually rising temperatures.

H. ROYAL-DAWSON.

Production of barium peroxide. A. F. MEYERHOFER (G.P. 426,034, 20.10.23).—Barium phosphate is treated with hydrofluosilicic acid, the precipitated barium silicofluoride separated from the phosphoric acid produced and decomposed by heating into barium fluoride and silicon tetrafluoride, the latter being used for the regeneration of hydrofluosilicic acid whilst the barium fluoride is treated with calcium hydroxide yielding calcium fluoride and barium hydroxide, which latter is then converted into barium peroxide by the usual method.

J. S. G. THOMAS.

Manufacture of hydrogen and other gases. AIRSHIP GUARANTEE CO., LTD., and P. L. TEED (E.P. 250,700, 27.1.25).—In the manufacture of hydrogen from ferrosilicon and caustic soda the residual sludge is continuously discharged into a trap adapted to be raised or tilted to prevent the ingress of air into the generator, while allowing the entire plant to be purged periodically to carry away any air which may leak into the system at other places. Should the pressure in the generator rise above a predetermined limit, the excess gas bubbles through a hydraulic seal into the atmosphere. The gas and steam from the generator pass through a condenser and the condensed water is returned wholly or in part to the generator to keep the strength of the solution more or less constant.

H. ROYAL-DAWSON.

Removing traces of chlorine from air. L. ROSENSTEIN (U.S.P. 1,578,850, 30.3.26. Appl., 19.4.23. Renewed 31.8.25).—Air containing traces of chlorine is passed up a tower containing scrap iron down which a solution of ferrous chloride continuously flows, being withdrawn at the bottom and pumped to the top of the tower. The chlorine rapidly reacts with the iron in presence of ferrous chloride solution forming a further quantity of that substance. At intervals a portion of the circulating solution is removed and fresh iron and water are added.

T. S. WHEELER.

Manufacturing powder of lead suboxide intermingled with powder of metallic lead. G. SHIMADZU (U.S.P. 1,584,150, 11.5.26. Appl., 14.7.23).—See E.P. 218,119; B., 1924, 711.

Manufacturing lead oxide. G. SHIMADZU (U.S.P. 1,584,151, 11.5.26. Appl., 18.4.24).—See E.P. 236,368; B., 1925, 714.

Production of lead acetate. R. PLAÜELN (U.S.P. 1,585,316, 18.5.26. Appl., 20.5.24).—See E.P. 224,199; B., 1925, 283.

Producing substances from boron and carbon. E. PODSZUS (U.S.P. 1,585,412, 18.5.26. Appl., 15.9.23).—See E.P. 204,337; B., 1924, 982.

Ammonia from vinasses (U.S.P. 1,576,427).—See XVII.

VIII.—GLASS; CERAMICS.

Annealing and re-annealing of glass. III., IV. W. M. HAMPTON (Trans. Opt. Soc., 1925-6, 27, 161—179; cf. B., 1925, 207).—Formulae previously deduced were applied to the case of heating glass under constant gradient. The rate of heating did not affect the ratio $(S_0 - S)/S$ (S_0 = strain due to temperature gradient, S = strain relieved by plastic flow) although it altered $S_0 - S$ at a given temperature, whilst changes in the size of the specimen affected both the ratio and $S_0 - S$. The theoretical strain-temperature curve agreed in form with the experimental curve when the coefficient of expansion was taken as variable with temperature. Theoretical analysis indicated that the annealing temperature could only be measured because of the sudden change in the coefficient of expansion in the region where measurable viscous flow is commencing. This temperature was only of use for comparing the annealing requirements of similar glasses, and, because of temperature lag, a standard rate of heating was necessary. The correctness of the stress-temperature equation $df/dt = f^2 k / \eta$ was confirmed in tests at relatively high temperatures and the values of k/η deduced for two glasses at various temperatures. From viscosity measurements, from the rate of sag of beams under load, values of k were found in the two cases. At lower temperatures (300°) the disappearance of birefringence was not a phenomenon of viscosity only but of plasticity, in which the law $df/dt = (f - f_L)^2 k / \eta$ held. The stress was relieved by flow on annealing until it reached a value f_L , a constant depending on the temperature. This conclusion was in agreement with results given by Adams and Williamson (B., 1921, 81A), it explained Twyman's results on chilling and tempering (B., 1923, 1177A), and it correlated the behaviour of glass with the property of clays and paints as studied by Bingham and Green (B., 1920, 495 A). The viscosity-temperature curve for three soda-potash-lead silica glasses gave a point of inflection as log η increased beyond 13.

A. COUSEN.

Progress report on investigation of saggar clays. R. A. HEINDL (J. Amer. Ceram. Soc., 1926, 9, 131—143).—A study of the characteristics of the individual saggar clays used in America is presented. A basis of classification was obtained by determination of the following properties: water of plasticity; volume and linear shrinkage; porosity and modulus

of rupture in the dry state; softening point; strength, porosity, and shrinkage after firing to five different temperatures; resistance to dunting; petrographic and chemical analyses. For the dunting test, small saggars were placed in furnaces at temperatures varying from 350° to 800° for 30 min., then withdrawn quickly and allowed to cool in the air. Most of the saggars fractured at or below 575°. For the purpose of continuing the study, the clays are arranged in five groups in the order of increasing average modulus of rupture and decreasing average porosity. Additional tests to study the effect of repeated firings showed that, after a number of firings under industrial conditions, the general result was an increase in the transverse strength and a decrease in porosity. Petrographic examination, however, indicated that, although the cold modulus of rupture was increased by repeated firings, the increase in the quantity of glass due to this treatment would probably actually weaken the structure at kiln temperatures.

F. SALT.

Refractory materials. D. B. SCHULZ (Feuerfest, 1926, 2, 33—35).—The paper presents the following data:—(1) Curves showing the relationship between composition and refractoriness of alumina-silica and kaolin-silica mixtures, from results by Seger; (2) examples of the composition of good average refractory materials (after Koppers) to illustrate the division of these by Littinsky into quartz (acid), chamotte (basic), and quartz-chamotte; (3) analyses of refractory materials for marine boilers, under the divisions (a) highly aluminous, (b) "dynaxite," (c) normal; (4) the relationship between the composition and softening and fusion points of refractory materials, after Hirsch; (5) the analyses of the ash of three naval coals from Wilhelmshaven; (6) the colour, composition, and m.p. of 11 coal ashes from various localities, by Aktiebolaget Ljungström Angturbin, Stockholm.

A. COUSEN.

Flow of heat in the walls of ceramic kilns. H. WILLMER (Ber. Deuts. Keram. Ges., 1925, 6, 49—62).—A method of determining directly the loss of heat through kiln walls is presented. Five kilns of different types were studied under working conditions. Thermo-elements were placed in holes, 50 cm. deep, bored into the walls at an average height of 1.8 m. above the kiln floor, and about 10 to 20 cm. apart. The outside temperature of the walls was measured by means of surface thermo-elements—small copper discs, to which thermo-elements were soldered. The weight of the kiln setting (saggars and ware) and the coal consumption were also determined. The results obtained indicated that the amount of heat absorbed by the walls, soles, and crowns of round kilns varied—according to the type and size of the kiln, the firing period, and the maximum temperature attained—from 12.8% to 18.9% of the total heat generated from the coal. The corresponding figure for a gas-fired kiln was 25%. An additional 5% was lost by radiation to the atmosphere from a stoneware kiln. These figures correspond roughly with the amount of heat required to heat the whole of the kiln setting including saggars. The degree of penetration

(*S*) is given by the formula: $S = A\sqrt{z}$, where *z* is the firing period in hrs., and *A* a coefficient, which varies for the different kilns from 5.2 to 15.9. To test the effect of insulation, the middle portion of the door of an earthenware biscuit kiln was constructed of bricks made from diatomaceous earth, temperature measurements being taken as before. From the results it is calculated that, with a completely insulated kiln, a saving of 7% in fuel would have been effected.

F. SALT.

Absorption of sulphur dioxide from kiln gases by ceramic ware. F. G. JACKSON (J. Amer. Ceram. Soc., 1926, 9, 154—173).—A study was made of two clays, one practically free from sulphur, the other containing pyrites. Draw trials were taken from a laboratory kiln and from industrial kilns at suitable temperature intervals. The laboratory kiln was an exact replica in miniature of a round down-draught kiln and measured 15 in. in diam. 10 g. were removed from the surface of each test brick and analysed for water-soluble sulphates and bases. The results of the tests showed that greatly increased concentration of sulphur in the kiln atmosphere produced only slight increase in the absorption of sulphur by the ware. Hence, variations in the sulphur content of the coal, within industrial limits, can have little effect on the amount of sulphur absorbed by the ware. The greatest absorption took place at about 500°, the time taken to reach this temperature having little effect. Under extreme conditions of exposure to sulphur gases only a small proportion of the bases present in the clays was converted into sulphates. When a coal containing only 0.5% of sulphur was used as fuel, less than 8% of the available sulphur was absorbed by the ware. Decomposition of the sulphates at higher temperatures could not be noticeably affected by the law of mass action, since the concentration of sulphur in the kiln gases is low under all circumstances.

F. SALT.

Properties of potters' flints and their effects in white-ware bodies. E. E. PRESSLER and W. L. SHEARER (Tech. Papers U.S. Bur. Standards, 1926, 20, [310], 289—315).—A study was made of the physical and chemical properties of 15 American and 2 French flints and of the firing behaviour of typical vitreous and semi-vitreous white-ware bodies containing these flints. Grading by air elutriation showed that, with four exceptions, more than 50% of the material consisted of particles less than 0.02 mm. diam., and about 99% of particles less than 0.10 mm. diam. The sp. gr. of the French flints was lower than that of the American and was reduced more by calcination. Calcination to cone 9 caused the development of much cristobalite, cryptocrystalline flints showing a greater proportion than quartz flints. The effect of heat treatment on flints and on pulverised bodies was studied by reference to alundum powder as standard. The resistance of bodies to thermal shock was tested by quenching specimens at 600° in air (expansion due to α - β quartz inversion at about 573°) and at 200° in water at room temperature. The thermal expansion near the quartz inversion point was approximately proportional to the flint content,

other factors remaining constant. Bodies containing crypto-crystalline flint showed greater expansion in the cristobalite inversion range (220—275°) than quartz flint bodies, and less at the quartz inversion point. Extreme fineness of the flint in bodies caused a slightly higher rate of expansion in the cristobalite inversion range and a lower rate at the quartz inversion point. The thermal shock tests indicated that crypto-crystalline flints promote resistance in the temperature range near the quartz inversion point, whereas quartz flints increase the resistance in the cristobalite range. Crypto-crystalline flints do not lower the maturing temperature of a body, but tend to render it more susceptible to overfiring. Finely-ground quartz flint lowers the maturing temperature of a body without affecting the vitrification range.

F. SALT.

Relationship between the constitution and the properties of porcelain. R. RIEKE (Ber. Deuts. Keram. Ges., 1925, 6, 144—153).—The structure of a fired porcelain body usually presents the appearance of a glassy (felspathic) matrix, in which crystals of mullite ($3\text{Al}_2\text{O}_3, 2\text{SiO}_2$) and quartz are more or less finely distributed, and which is permeated with minute pores due to air bubbles. The mullite and quartz crystals may vary both in number and size. The properties of ordinary hard porcelain are mainly determined by the nature of the matrix, which usually forms much more than one-half of the body. Its composition may vary as follows: 78—86% SiO_2 , 11—8% Al_2O_3 , 10—6% K_2O , omitting minor ingredients. The composition: 86% SiO_2 , 8% Al_2O_3 , 6% K_2O represents the optimum theoretical conditions, when all the quartz and 10—15% of the mullite have been dissolved. The nearer a porcelain approaches this optimum in composition, the better its physical properties. The quantity, size, and distribution of the mullite crystals have a marked effect upon the mechanical properties. These crystals should preferably exhibit a fine-grained formation, the structure then having the appearance of a felted network of needles. The methods by which the desired body structure can be produced and existing porcelains improved are discussed in broad outline. The quality of the matrix is affected by the kind of felspar used, its varying content of potassium, sodium, lime, silica, etc. The addition of small amounts of lime, magnesia, zinc oxide, etc. reduces the viscosity of the matrix and promotes the formation of large mullite crystals, thus increasing the translucency of the fired body, but also increasing the coefficient of expansion and hence reducing the resistance to abrupt changes of temperature. In general, the quartz in the body should be visible under the microscope only as small grains. To achieve this, the raw quartz must be ground as fine as possible and the ware must be fired for a long time at a high temperature. The above additions may also be made to accelerate the solution of the quartz in the matrix. The degree of mullite development is influenced by the type of kaolin used; relatively coarse-grained types, *e.g.*, English china clay, tend to promote the formation of large mullite crystals and thus produce more translucent bodies than the

fine-grained, plastic varieties. Mullite has been identified in bodies fired to 1200°, but it develops rapidly with rising temperature. F. SALT.

Translucency of porcelain. R. RIEKE and K. SAMSON (Ber. Deuts. Keram. Ges., 1925, 6, 189—201).—The translucency of nineteen porcelains, varying in composition between the limits of 40—60% of clay substance, 15—35% of quartz, and 15—35% of felspar, was determined with a photometer (*cf.* Steger, Ber. Deuts. Keram. Ges., 1921, 2, 9). The refractive index of the matrix of hard porcelain is 1.48, of mullite crystals 1.64, and of quartz crystals 1.54. Hence, mullite has a greater effect upon translucency than quartz. In general, the translucency of porcelain is increased by raising the firing temperature, and by increasing the felspar content at the expense of the quartz, the clay-substance content remaining constant. The highest translucency values were obtained with bodies containing 40—50% of clay substance, 15—30% of quartz, and 30—35% of felspar. Repeated firing to the same temperature reduced the translucency by increasing the mullite formation. The well-known Seger porcelain (low-clay-substance content) was much more translucent when fired to cone 12 instead of cone 10. Bodies prepared with kaolins of different origin exhibited marked variations in translucency; *e.g.*, the substitution of china clay for Sedlitz kaolin improved the translucency considerably. Varying the type of raw quartz used also affected the translucency. Best results were obtained with Norwegian vein quartz, which was converted almost entirely into cristobalite after one firing in a porcelain oven at cone 15. The more finely ground the quartz, the better the translucency. The effect of pegmatite, when substituted for felspar and sand, varied considerably, some varieties giving improved results, others the reverse. The addition of small amounts of lime or magnesia increased the translucency; iron oxide had the opposite effect. Preliminary fusion of the felspar with the quartz proved very effective; by fritting together these materials, so that all the quartz was dissolved, and using the resulting frit to prepare porcelain bodies with Sedlitz kaolin and English china clay, the translucency figures were 312 and 370 respectively (Seger porcelain 230).

F. SALT.

Impurities [discolorations] in fired porcelain. HACKELOER-KÖBBINGHOFF (Ber. Deuts. Keram. Ges., 1925, 6, 236—242).—The causes to which discolorations in fired porcelain are due are discussed. Pyrites is frequently present in kaolins having a high percentage of sand (*i.e.*, insufficiently washed). The composition of the original rock and the mode of kaolinisation largely determine whether, and in what form, pyrites is present. Felspar may contain micaceous impurities; iron-containing biotite and chloride micas in particular must be carefully eliminated. Water used in grinding, or in the making operations, if not fresh, may be contaminated with filamentous algæ, minute iron-secreting organisms, the cells of which cause "iron spots" on the fired ware. Rust may be introduced into the body from

iron piping, whether galvanised or not. Grinding pans should be lined, and the lining cemented, with material containing only traces of iron. German "Findlings" quartzite is not suitable. Excessive weathering, or ageing, of porcelain body mixtures also leads to the rapid development of filamentous algæ. Compressed air, used for cleaning biscuit ware, may absorb moisture contaminated with iron from the pumps. In the firing of body mixtures which have been insufficiently ground, deposition of carbonaceous matter frequently occurs during the reducing period. F. SALT.

Elutriation process for electro-corundum. H. VIERHELLER (Sprechsaal, 1926, 59, 113—114, 128—130; Chem. Zentr., 1926, I., 2957).—The addition of 0.04% of sulphite-cellulose waste lye of 33—35° B. (*d* 1.3—1.32) to water prevents the flocculation of the corundum, and enables the elutriation of corundum dust finer than 0.09 mm. to be carried out (cf. G.P. 399,725 and 414,909; B., 1924, 1014).

Microscopical investigation of opacity phenomena in enamel frits. A. OTREMBIA (Keram. Rund., 1926, 34, 67—70, 88—90, 107—109).—The opacifying effects of fluorine compounds in enamels, without the addition of tin oxide, were studied microscopically. Frits varying widely in composition were melted and allowed to cool in the air—not quenched in water, as is the usual practice. Fluorine was introduced in varying proportions in the form of fluorspar, natural and artificial cryolite, and sodium silicofluoride. The frits were heated to 740° and 950°, in order to study the effect of temperature. To produce an opacifying effect, fluorspar required a high temperature, or, alternatively, the use of a frit containing a high percentage of fluxes. Its opacifying effect is limited; when present in large proportion, it raises the melting point of the frit. The opacity produced is probably due to the formation of the fluorides of aluminium and of the alkalis. Natural cryolite produced greater opacity at the lower temperature than did fluorspar at the higher, the effect in this instance being due in part to the presence of minute bubbles formed by the volatilisation of fluorine or silicon fluoride. Artificial cryolite proved to be a more effective opacifier than natural cryolite. In some cases, sodium silicofluoride produced a more uniform and greater opacity at low temperatures than artificial cryolite, provided that enough felspar was present to supply the necessary alumina and alkalis. F. SALT.

Determination of porosity of refractory bricks. ESSER and PIVOVARSKY.—See II.

PATENTS.

Continuous [glass] tank furnace. E. T. FERNGREN, Assr. to LIBBEY-OWENS SHEET GLASS Co. (U.S.P. 1,581,338, 20.4.26. Appl., 27.10.23).—A furnace for supplying molten glass to a pair of glass-working machines consists of a melting tank connected to a pair of parallel refining tanks by passages narrower than the refining tanks. The enclosed heating space above each refining tank overlaps the outer sides of the tanks, and is supplied

with heat from a chamber above the melting tank, so that a greater portion of the heat is directed to the outer portions of the tank. B. W. CLARKE.

Crucible and like furnaces. T. TEISEN (E.P. 238,562, 14.8.25. Conv., 14.8.24. Addn. to 194,819).—In crucible furnaces, *e.g.*, for glass, of the type described in the chief patent (B., 1923, 460 A) each recuperator is split into two units and the glass-pit is arranged between the two units. The glass-pit may be divided by a partition which may be continued upward to divide the collecting flue for waste gases, and may even be continued through the chamber floor into the crucible chamber. If each section of the recuperator is provided with an independent damper more satisfactory working of the recuperator system is possible. C. A. KING.

Removal of iron skin from waste products of glass manufacture. A. GESNEL (F.P. 601,440, 31.7.25).—Waste products of glass manufacture are treated in a bath containing a cold dilute solution of one or more mineral acids, *e.g.*, hydrochloric, sulphuric, or nitric acid, and hydrofluoric acid or a mineral, *e.g.*, fluorspar or cryolite, yielding fluorine or hydrofluoric acid. J. S. G. THOMAS.

Manufacture of enamelled objects. M. J. DAVIDSEN (E.P. 248,514, 24.12.24).—A cement, rich in alumina and poor in silica and lime, is mixed with not more than 15% of sand or Portland cement, or both, sufficient water for setting being added. Objects made from the mixtures are allowed to harden in air or water and subsequently enamelled and fired in the usual way. F. SALT.

Enamelling [cadmium and zinc]. H. C. PIERCE and C. H. HUMPHRIES, Assrs. to UDYLLITE PROCESS Co. (U.S.P. 1,583,006, 4.5.26. Appl., 5.6.24).—The surfaces are treated with a solution of an arsenate and then enamelled in the usual way. F. SALT.

Opacifying enamels. VER. CHEM. FABR. KREIDL, HELLER & Co. (Austr. P. 102,118, 13.5.23. Conv., 16 and 18.5.22).—To prevent an enamel bubbling when over-fired a quantity of anhydrous material rich in alumina, *e.g.*, felspar, clay, or kaolin, which has previously been heated above 1000°, is added to the opacifier during grinding, in addition to the usual quantity of clay. A. R. POWELL.

Refractory material. GROSVENOR SCIENTIFIC PRODUCTS, LTD., and B. TURNER (E.P. 250,354, 2.2.25).—A refractory material is prepared from chrome ore (most suitably Rhodesian), crushed to pass 0.1 inch mesh and suitably graded, with refined china clay and sodium or potassium silicate. Preferably, 16 pts. by weight of ore are dry-mixed with 1½ pts. of clay and the mixture is moistened with ½ pt. of aqueous silicate (2SiO₂:1Na₂O), *d* 1.6. The mixture is pressed in a mould, then removed, and fired. A. COUSEN.

Refractory article. A. J. JACKMAN, Assr. to VESUVIUS CRUCIBLE Co. (U.S.P. 1,577,124, 16.3.26. Appl., 2.8.24).—Articles made chiefly of graphite

and a vitrifiable ceramic bond are fired at a temperature above the vitrification point of the bonding material.

F. SALT.

Tunnel kiln. H. W. WEBER and A. O. SCHLEIFARTH, Assrs. to RUSSELL ENGINEERING CO. (U.S.P. 1,575,470, 2.3.26. Appl., 27.11.25).—Hot air and gases in the preheating zone are passed through an inlet into a flue arranged transversely across the top wall. A fan at one end induces longitudinal circulation in the flue and discharges the hot gases into a vertical flue in the side wall, whence they are returned to the lower portion of the preheating zone.

F. SALT.

Continuously-operated kiln. C. SMITH (U.S.P. 1,578,559, 30.3.26. Appl., 23.2.23).—A kiln for burning and drying brick and the like consists of a vertical drying shaft, up which the material is conveyed, communicating near the top by means of a flue with a vertical burning shaft connected with a fire-box.

B. W. CLARKE.

Ceramic material. R. T. STULL, Assr. to STULL PROCESS CO. (U.S.P. 1,576,558, 16.3.26. Appl., 20.3.24).—Light-coloured bricks are made from a mixture composed of a natural, highly colloidal, secondary kaolin, deficient in alkali and alkaline-earth fluxes, and a pegmatite.

F. SALT.

Cast or moulded articles [of titanium-containing slag]. F. A. DE SILVA (U.S.P. 1,579,093, 30.3.26. Appl., 9.4.25).—The molten slag obtained in the direct production in the electric furnace of titanium steel from titaniferous iron ores contains principally titanous acid and titanium carbide and when cooled, particularly if the cooling takes place in moulds under pressure, forms an extremely hard, resistant and practically infusible substance suitable for use as a lining for furnaces, in the manufacture of crucibles, etc. For roofing purposes and the like it is coated on asbestos when molten.

T. S. WHEELER.

Artificial aluminous abrasive. H. A. RICHMOND and R. MACDONALD, JUN., Assrs. to GEN. ABRASIVE CO. (U.S.P. 1,583,179, 4.5.26. Appl., 9.8.24).—The operating temperature in the production of crystalline alumina from an aluminous ore is lowered by the addition of an alkaline-earth to the furnace charge.

F. SALT.

Refractory brick for furnace linings (U.S.P. 1,576,021).—See I.

IX.—BUILDING MATERIALS.

Lime-silica index as measure of cement quality. T. MERRIMAN (Eng. News. Rec., 1926, 96, 648—650).—The lime-silica index is the ratio of the available lime, *i.e.*, the total lime less that in combination with the alumina, ferric oxide, and sulphuric acid, to the available silica, *i.e.*, total silica less insoluble residue. Assuming that tricalcium silicate is the most desirable constituent of Portland cement, a high index should indicate a cement of

good quality. Actually, owing to incompleteness of combination of the calcium silicates during manufacture, a high lime-silica index is accompanied by a relatively low strength at 7 days. This shows that the burning operation has been badly carried out and that a large proportion of inert or underburned material is present. In order to ensure that the maximum cementing value is obtained, it is necessary to specify both the strength and the lime-silica index of the cement.

B. W. CLARKE.

Rapid determination of lime in cement and raw materials for cement. H. KÜHL and F. KLASSE (Zement, 1926, 15, 181—184; Chem. Zentr., 1926, I., 2958).—0.3 g. of the ignited material is treated with 5 c.c. of fuming nitric acid on the water bath, and after evaporating the excess of nitric acid, an excess of powdered oxalic acid crystals is added and the mixture heated gradually in an electric furnace, finally keeping at 500—550° for 20—30 min. After cooling, the material is transferred to a calcimeter.

B. W. CLARKE.

Fineness of particles of cement, especially iron Portland cement. A. GUTTMANN (Zement, 1926, 15, 164—168, 185—187, 200—203; Chem. Zentr., 1926, I., 2955).—The finest particles in cement have generally a higher percentage of lime and gypsum than the larger particles. Of two iron Portland cements, the one which was the finer by sieve analysis, showed the lower strength, but use of a 10,000-mesh sieve showed that the other had the larger proportion of very fine particles. Sedimentation in the Bauer apparatus is necessary for the quantitative determination of the finest particles.

B. W. CLARKE.

Endurance of flooring materials. C. H. GEISTER (J. Amer. Ceram. Soc., 1926, 9, 126—130).—The durability and the sanitary properties of all types of flooring materials were investigated on the basis of the following tests: resistance to abrasion, absorption of water, indentation, resistance to chemicals and stains, and deterioration from age. The results of the tests, which are tabulated, show clearly the superiority of ceramic tiles over other materials.

F. SALT.

PATENTS.

Manufacture of aluminous cements, coloured or white. U. B. VOISIN (E.P. 248,282, 14.10.25. Addn. to 243,876; B., 1926, 129).—The aluminous cement mass, very finely ground and very intimately mixed, is roasted for a long period (6 to 8 hrs.) at a temperature well below the softening point (1000—1100°). White cement is produced by the use of white bauxite and pure lime.

F. SALT.

High-temperature cement. P. G. WILLETS, Assr. to HARTFORD-EMPIRE CO. (U.S.P. 1,573,888, 23.2.26. Appl., 13.11.25).—A refractory cement is prepared from barium sulphate, silica, and sodium silicate.

F. SALT.

Manufacturing cements [plaster] from overburnt gypsum, overburnt gypsum waste, or

natural anhydrite. P. P. BUDNIKOV and M. E. LEWIN (G.P. 320,957, 26.1.24).—A hard-setting plaster is produced, without the necessity of employing any simultaneous or subsequent heat treatment, by mixing the raw material intimately with the appropriate amount of acid alkali sulphate, *e.g.*, sodium hydrogen sulphate or potassium hydrogen sulphate, or neutral alkali salt together with the requisite amount of sulphuric acid. J. S. G. THOMAS.

Induration or minerlisation of organic matter. C. D. BURNEY (E.P. 249,899, 25.10.24).—Fibrous organic material, either moistened, or washed with a weak solution of an alkali or of an acid, is treated with a dry hygroscopic chemical which reacts exothermically with water, such as dry caustic soda preferably in flakes, or dry caustic lime, or magnesium chloride, and mixed, while heat is still being evolved, with cement, mortar, and the like to produce on setting a light form of concrete which can be moulded under pressure. B. W. CLARKE.

Coloured granulated slag and method of manufacture. W. VAN DE MARK, Assr. to VULCAN LOUISVILLE SMELTING CO. (U.S.P. 1,582,318, 27.4.26. Appl., 23.5.24. Renewed 25.2.26).—A granulated material, suitable for covering roofing felt, is obtained by smelting the residue from brass or copper smelting furnaces with a calcium compound to reduce its iron content, and pouring into water. A. GEAKE.

Preservation of stone. A. P. LAURIE (U.S.P. 1,585,103, 18.5.26. Appl., 16.8.24).—See E.P. 221,342; B., 1924, 911.

Rotary drums for cooling materials (E.P. 250,318).—See I.

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

Testing cast-iron [previous to casting]. P. WOLFF (Stahl u. Eisen, 1926, 46, 560—564).—Before casting a charge of cast-iron a small wedge-shaped casting should be made in a sand mould. The Brinell hardness of this casting after removing the outer layer gives sufficiently accurate indications of the quality of a charge once it has been standardised against analyses and tensile tests on similar material. The fracture of the test-piece also provides information as to the probable structure of the castings that will be obtained. The sharp end of the wedge will indicate the depth of the white hard layer that may be expected and the upper end of the wedge the nature of the grey interior of the casting. The tensile strength of the metal is given approximately by the equation, $T = (H - 40) / 6$, where H is the Brinell hardness. A. R. POWELL.

Influence of prolonged heating on the crystallisation of combined carbon in cast-iron. O. WEDEMEYER (Stahl u. Eisen, 1926, 46, 557—560).—A series of large-scale tests in a coal-fired reverberatory furnace has confirmed Pivovarski's observation (B., 1925, 805) that prolonged heating at temperatures below 1500° increases the carbide content of

cast iron. The increase is much more marked in the case of cast-iron made from soft iron than in that made from hæmatite iron; thus, overheating for 3 hrs. at 1295° increased the combined carbon in the first case from 1.12 to 2.57% and in the second case only from 0.8 to 1.00%. A. R. POWELL.

Behaviour of aluminium towards iron at high temperatures. R. IRMANN (Z. Metallk., 1926, 18, 121—122).—The amount of iron dissolved by molten aluminium increases with rise of temperature but depends on the nature of the iron, a high silicon and/or carbon content decreasing the solubility of the iron. Thus, aluminium at 800° dissolves in 1 hr. 1% of iron from a steel containing 0.12% C, but none from grey cast-iron with or without the casting skin; at 900°, 2.5% of iron is dissolved from the steel, 0.5% from cast-iron with the skin removed, and nothing from cast-iron with the skin, and at 1000° the amounts dissolved are 6%, 2.5%, and 1% respectively. The solubility of a cast-iron in aluminium decreases hyperbolically with the content of combined carbon and linearly with the silicon content. Cast-iron crucibles are therefore the most suitable for melting aluminium especially if the inner surface is coated with aluminium and then heated to 1000° before use. Steel crucibles similarly treated do not give such good results, but both cast-iron and steel crucibles that have been heated until a thin layer of ferrosferic oxide is formed inside yield only traces of iron to aluminium that is melted in them. A. R. POWELL.

Tensile properties of metals [steel] at high temperatures. T. D. LYNCH, N. L. MOCHEL, and P. G. McVETTY (Proc. Amer. Soc. Testing Materials, 1925, 25, II, 5—26; Chem. Abstr., 1926, 20, 1202).—The elastic properties of medium-carbon steel decrease with rise of temperature (up to 500°), the ductility is minimal at about 250°, whilst the tensile strength is minimal at 100° and maximal at 275°. Nickel steel (5% Ni) was similar, but exhibited better ductility. "Stainless" iron showed much less effect with rise of temperature. Cast manganese bronze showed no change in elastic properties up to 260°, but a drop in tensile strength. Previous heat treatment of the medium-carbon steel reduced the final rate of extension under stress; manganese bronze elongated more than steel in the long-time tests. At a constant high temperature the stress varied linearly with the logarithm of the time to cause fracture. Medium-carbon steel stressed above the proportional limit for 200 hrs. at 400° showed at the normal temperature decreased ductility, increased strength, and increased elastic properties, except the modulus. A. A. ELDRIDGE.

Elastic limit and the cold and hot shortness [of steel]. P. LUDWIK (Z. Ver. Deuts. Ing., 1926, 70, 379—386; Chem. Zentr., 1926, I., 3176—3177).—The changes in the elastic limit of electrolytic iron and ingot steel during ageing and at a "blue heat," as well as the "blue brittleness" and other phenomena of the hardening of steel, are shown to be due to changes in the solubility of the minor consti-

tments in the steel just as is the case with the hardening of aluminium alloys during ageing. The cold brittleness, the dependence of the notched bar impact test figures on the shape of the notch, the velocity of the blow, and the temperature and the possibility of rupture before the beginning of elastic flow in tensile and impact tests are caused by changes in the slip and shear resistance of the metal. The shear strength of steel is decreased during pickling owing to adsorption of hydrogen in the surface layers of the metal, but this has no effect on the elastic limit or tensile strength. The brittleness induced in steel by contact with alkali hydroxide solution is caused by a reduction in the resistance to shear caused by hydrogen adsorption, and an increase in the slip resistance caused by ageing.

A. R. POWELL.

Moduli of elasticity and rigidity and their change caused by magnetisation in different kinds of steel. K. HONDA and T. TANAKA (Sci. Rep. Tôhoku, 1926, 15, 1—37).—The moduli of elasticity and of rigidity of carbon steels decrease with an increase of the carbon content and also after annealing and quenching, but they are increased after magnetisation of the steel. The elastic constants of cobalt-iron alloys are increased by magnetising except in the case of alloys containing 35—55% Co, the elastic constants of which decrease in weak magnetic fields. Similar changes take place on magnetising nickel-iron alloys. The elastic moduli of cobalt are reduced by magnetisation. In all cases the effect of a magnetic flux is three times as great on the rigidity modulus as on the elastic modulus.

A. R. POWELL.

Properties of high-silicon structural steels. (Stahl u. Eisen, 1926, 46, 493—503).—Tests carried out on low-carbon steels containing about 1% Si, manufactured at various foundries in different types of furnaces and under different rolling conditions, confirm previous work on the influence of silicon. The tensile strength and, in higher degree, the yield point, properties markedly affected by the shape of the test-piece, are increased without diminution of the percentage elongation and reduction in area of the test-piece.

L. M. CLARK.

Primary crystallites in chrome-nickel steels. F. LEITNER (Stahl u. Eisen, 1926, 46, 525—533).—Specimens of chrome-nickel steels cast at different temperatures were etched to show the structure, and mechanical tests were made. The formation of large crystallites is disadvantageous, leading to local accumulation of impurity and formation of holes with corresponding injury to mechanical properties. These properties can be improved by suitable heat treatment.

L. M. CLARK.

Mechanical properties of titanium steel. K. TAMARU (Sci. Rep. Tôhoku, 1926, 15, 73—80; cf. B., 1925, 593).—Addition of 0.3% Ti to plain carbon steels increases the tensile strength and reduction in area, but decreases the elongation. Owing to the tendency to the formation of titanium nitride when the steels are melted in air, better

mechanical properties are obtained by melting *in vacuo* or under a low pressure of hydrogen. The yield point of titanium steel decreases with an increase in the carbon content whereas the elongation is hardly affected.

A. R. POWELL.

Cementation of ferrous and cuprous alloys by tungsten, molybdenum, and tantalum. J. LAISSUS (Compt. rend., 1926, 182, 1152—1154; cf. B., 1925, 635; 1926, 278).—The cementation of an iron-carbon alloy containing 0.15% of carbon, by finely powdered ferromolybdenum (C 1.86% Mo 71.85%) or ferrotantalum (C 1.00%, Ta 29.26%, Si 1.96%) has been studied between 800 and 1200°. Micrographical examination shows the existence in the interior of a solid solution bounded by a brilliant external layer, the thickness of which increases with rise of temperature and with the duration of treatment. Copper and brass have been similarly treated with tungsten, molybdenum, and tantalum; cemented layers are formed which increase in thickness with rise of temperature.

F. G. SOPER.

Corrosion of steels in the atmosphere. W. G. WHITMAN and E. L. CHAPPELL (Ind. Eng. Chem., 1926, 18, 533—535).—Test specimens enclosed in a galvanised box were sprayed intermittently with water for periods of 15 min. at intervals ranging from 30 min. to 24 hrs., the cycle of operation being regulated automatically. The rate of corrosion compared well with that of specimens in actual field tests. The rate of corrosion varied with the time of the cycle of operation, and appeared to depend on the actual time the specimen was wetted. With chromium steels, the corrosion decreased rapidly as the chromium increased from 8 to 16%.

B. W. CLARKE.

Resistance to corrosion of Thomas and Siemens-Martin steels containing copper. K. DAEVES (Stahl u. Eisen, 1926, 46, 609—611).—The differences in the conclusions arrived at by the Amer. Soc. for Testing Materials and those of O. Bauer (cf. B., 1921, 393 A) from data collected by the Verein deutscher Eisenhüttenleute and the Materialprüfungsamt in Berlin-Dahlem, are discussed. To test whether these were due to climatic conditions or to fundamental differences between German and American steels, experiments have been carried out in which ten specimens of each of various kinds of steel containing varying amounts of copper up to 0.5% have been exposed to the air without regard to the district, and the amount of corrosion determined by weighing the plates before exposure and again after removing the rust. The results are expressed in curves in which the loss in weight is plotted against the % of copper in the steel. In agreement with American results, amounts of copper up to 0.25% are found very effective in retarding corrosion in all the classes of steel tested. The improvement is most marked in the case of Thomas steel. Siemens-Martin steel made by the crude ore process occupies a middle position but Siegerlander material (high natural copper content) is almost as good as Thomas steel. The influence of copper when the steel is

exposed to earth corrosion is not so marked, but again Thomas steel is the more resistant.

A. COULTHARD.

Dilatometric anomaly of paramagnetic nickel-chromium alloys. Alloy for dilatation pyrometry. P. CHEVENARD (Compt. rend., 1926, 182, 1281—1283; cf. B., 1925, 674).—Industrial nickel-chromium alloys (5—10% Cr, 2% Mn) exhibit an anomaly in the dilatation curve near 550°, which is imperfectly reversible. The anomaly is almost completely suppressed in a new alloy ("pyros") in which the manganese content has been increased and iron and tungsten added (82% Ni, 7% Cr, 5% W, 3% Mn, 3% Fe). An expression for the dilatation of "pyros," with the constants determined, is given.

R. A. MORTON.

Surface films in the cathodic polarisation of metals. E. LIEBREICH (Korrosion u. Metallschutz, 1926, 2, 38—41; Chem. Zentr., 1926, I., 2965—2966).—As is the case with chromium, iron, and mercury, there is a potential range in the feeble cathodic polarisation of nickel and aluminium in which the metal has a much greater tendency to dissolve in the electrolyte with the formation of surface films of basic salts. A similar phenomenon appears to take place during the slow natural corrosion of these metals, and is probably the cause of the feeble alkalinity observed by Wurstemberger in the liquid immediately in contact with a corroded metal surface. The facts are explained as follows: during cathodic polarisation the charged hydrogen first formed at the cathode is adsorbed either by the metal or by the solution, leaving a film of solution containing an excess of hydroxyl ions which react with the metal to form basic salts or hydroxide; as the polarisation increases the increasing quantities of hydrogen produced reduce these salts again to metal, after which the hydrogen is evolved in bubbles. A. R. POWELL.

Rapid determination of small quantities of palladium. F. C. ROBINSON (Inst. Min. Met., May, 1926. Advance copy, 3 pp.).—Small quantities of palladium may be determined colorimetrically in the acid used in parting the gold-silver-platinum-palladium bead obtained by cupellation. A metal ratio of 33 Ag : 10 Au : 1 (Pt+Pd) is necessary for satisfactory parting of the bead and, in cases where nitric acid is used, the platinum should not exceed 10 times the weight of the palladium, and in no case more than 50 mg. per 100 c.c. of acid. Sulphuric acid answers equally well for the parting and has the advantage that it does not dissolve platinum. The standard palladium solution contains 1 mg. Pd per c.c., and is made by dissolving the requisite amount of metal in 1 : 1 nitric acid, and evaporating with sulphuric acid in case a sulphate solution is required.

A. R. POWELL.

Production of a regular structure in the recrystallisation of [cold-rolled] copper. W. KÖSTER (Z. Metallk., 1926, 18, 112—116).—When severely cold-worked copper is annealed the new crystals are oriented in such a way that the twinning planes are at an angle of 45° to the direction of

rolling and deep etching shows that the new crystals have a cube face in the plane of rolling and a normal to the cube in the direction of rolling. The recrystallised grains are extended further in the direction of rolling than in the direction perpendicular to it. The regularity of the recrystallised structure decreases with the amount of cold work to which the metal was previously subjected, but the orientation of the new structure is independent of the annealing temperature, time of annealing, and rate of heating. The tensile strength, ductility, and pliability of recrystallised sheet are greatest at an angle of 45° to the direction of rolling, and the tensile strength is at a minimum at an angle of 22.5° to the direction of rolling.

A. R. POWELL.

Determination of aluminium oxide in aluminium metal. W. H. WITHEY and H. E. MILLAR (J.S.C.I., 1926, 45, 170—174 T).—The only method found satisfactory consisted in heating the metal in dry gaseous hydrogen chloride, and determining the residual oxide. Experiments made on foil of various thicknesses indicate that the oxide is confined to a minute surface layer, but that the proportion of oxide in thin foil is not, as might be expected, larger than on thick sheet. By reducing oxides of lead and iron by fusion with molten aluminium and analysis of the resulting ingots cast from the skimmed metal, it is shown that the alumina produced during the reaction, although formed under most favourable conditions for solution in the metal, is almost completely expelled; in other words, molten aluminium is incapable of dissolving its oxide and any oxide originally present in the metal is probably confined to a minute surface layer. A method of determining carbon in aluminium by dissolving the metal in a mixture of sulphuric and hydrochloric acids and burning the evolved gases is described.

Complexity of the phenomena of quenching of certain alloys. A. PORTEVIN and P. CHEVENARD (Compt. rend., 1926, 182, 1143—1145; cf. A., 1921, ii., 510; 1923, ii., 166).—Two general cases are distinguished in the quenching of alloys. In the first the alloy has a γ state stable at high temperatures, and an α state stable in the cold; any treatment which refines the structure of α increases the hardness of the alloy. In the second case, there exists in addition a state λ in labile equilibrium with the γ state. Evidence for this view is adduced from a dilatometric study of an aluminium-bronze, sudden cooling causing the formation of the λ state which is attended by an expansion. F. G. SOPER.

Tensile strength of metals and alloys under a statical load. G. WELTER (Z. Metallk., 1926, 18, 75—80, 117—120).—Rods of different cross-sections of brass, copper, aluminium, and steel were subjected to a load above the elastic limit, but below the breaking strain, for prolonged periods until fracture ensued. Coarse-grained $\alpha+\beta$ -brass withstood loads up to within a few kg. per sq. mm. of the breaking strain almost indefinitely, but little more than the load required to stretch the metal beyond the elastic limit sufficed to cause fracture of fine-grained brass

of similar composition within a few hours or days. Copper and aluminium failed in the course of several months under loads just above the limit of proportionality, whereas steel withstood loads almost equal to the tensile strength indefinitely. Round bars in all cases were stronger than notched bars and bars with a rectangular notch were stronger than those with a wedge-shaped notch. The importance of these results in testing metals for constructional work is pointed out. A. R. POWELL.

Fatigue of metals by direct stress. P. L. IRWIN (Proc. Amer. Soc. Testing Materials, 1925, 25, II., 53—65; Chem. Abstr., 1926, 20, 1203).—The endurance limit obtained for steels and for manganese bronze by direct stress is the same as that obtained by flexural stress. A. A. ELDRIDGE.

Granulation of slags and metals. B. BOGITCH (Compt. rend., 1926, 182, 1221—1223).—In the granulation of slags and metals by quenching, dangerous explosions may occur if too much molten metal is entrained in the molten slag. A jet of compressed air introduced below the water surface serves to retard and break up the molten stream, and to prevent the formation of a solid sheet over the surface of the vessel. Experiments with an iron-magnesium silicate slag and a molten metal, using air compressed to 1 kg., showed that quenching may safely be carried out in smaller vessels, and from a less height than is usual. The hot water produced may also be utilised. J. GRANT.

Rôle of sodium sulphate in the refining of mattes. B. BOGITCH (Rev. Mét., 1926, 23, 193—198).—See B., 1925, 995.

See also A., May, 486, **Action of brass on cuprous chloride** (MASING and MIETHING); **Gold in mercury** (VENATOR). 488, **Reaction between manganese and iron sulphide** (HERTY, JUN., and TRUE). 489, **Preparation and properties of metallic yttrium** (THOMPSON, HOLTON, and KREMERS). 490, **Precipitation of tellurium from alkaline sulphide solutions and its separation from heavy metals and from selenium** (BRUKL and MAXYMOWICZ). 491, **Determination of potassium** (DELAVILLE and CARLIER; WIKUL; LE BOUCHER); **Determination of small amounts of silver in presence of lead** (DONATH); “**Citarin**” as a quantitative reagent (VANINO and GUYOT); **Determination of calcium** (FOOTE and BRADLEY); **Compounds of diphenylthiocarbazon with metals and their use in analysis** (FISCHER); **Determination of cadmium in copper** (BLAZEY). 492, **Determination of manganese, zinc, and cobalt** (STREBINGER and POLLAK); **Volumetric determination of chromium and manganese** (REINITZER and CONRATH).

See also pages 522, **Determination of porosity of rammed moulding sand** (ESSER and PIVOVARSKI). 549, **High-frequency induction furnaces** (WEVER); **Flames of atomic hydrogen** (LANGMUIR). 550, **Arc welding with atomic hydrogen** (WEIMAN and LANGMUIR); **Arc welding in hydrogen and other gases** (ALEXANDER); **Influence of cathodic**

hydrogen on strength of steel (ALEXEJEV and POLUKAROV).

PATENTS.

Electrodepositing iron from minerals. M. OKOCHI and M. HANAOKA, Assrs. to ZAIDAN HOJIN RIKAGAKU KENKYUJO (U.S.P. 1,582,423, 27.4.26. Appl., 2.4.24).—Iron oxide ores, such as magnetite sands, yellow ochre, and bog iron ore, are reduced to spongy iron at 1000°, and a mixture of this iron and material containing a sulphide of iron is made in the anode in an acid electrolyte. A. R. POWELL.

Manufacture of acid-resistant iron-silicon alloys. H. FISCHER (G.P. 426,113, 26.11.19).—Copper is added to the alloys to lower their content of silicon. A. COULTHARD.

Casting aluminium and aluminium alloys and moulds for use therein. E. STRASSER (E.P. 244,441, 18.11.25. Conv., 13.12.24).—Sand moulds for casting aluminium alloys are made of a mixture of “green” sand and a metal or metal compound, such as copper filings or roasted copper sulphide ores. The moulds are used damp so as to ensure as rapid a chilling of the casting as possible. Aluminium-zinc alloys cast in this manner and aged for 14 days show superior mechanical properties to similar alloys cast in the ordinary sand moulds and heat-treated. A. R. POWELL.

Separation and purification of platinum and other platinum metals from platiniferous ores and substances. L. D. HOOPER (E.P. 250,726, 6.3.25).—Platinum is removed from platiniferous materials by treating them with carbon monoxide and chlorine at 250—600° in the presence of a catalyst such as metallic sodium, whereby carbonyl compounds of platinous chloride are volatilised. These may be purified by recrystallisation from carbon tetrachloride. Palladium is recovered from the residue from the platinum extraction by treatment with chlorine and carbon monoxide at 300—650° in the presence of ammonium fluoride or chloride or of hydrogen chloride, whereby similar palladous chloride carbonyl compounds are volatilised. By subjecting the residue from this treatment to carbon monoxide at 300° under 400 atm. pressure ruthenium dicarbonyl is formed and may be extracted with alcohol from the remainder of the mass. The metals are recovered from the carbonyl compounds by heating, with or without the presence of water. A. R. POWELL.

Refining copper-nickel matte. N. V. HYBINETTE, Assr. to ANGLO-CANADIAN MINING AND REFINING Co., LTD. (U.S.P. 1,577,422, 16.3.26. Appl., 13.1.21).—A solution of copper and nickel sulphates, obtained from a later stage of the process, is treated with bessemerised copper-nickel matte to obtain a purified nickel sulphate solution for electrolysis, and a mixture of matte and cement copper which is roasted to remove sulphur. The roasted product is leached with sulphuric acid to obtain copper sulphate solution which is electrolysed for copper and the regeneration of sulphuric acid for further leaching of roasted matte. The nickeliferous residues from leaching are reduced and

dissolved in sulphuric acid regenerated during the previous nickel electrolysis and the solution is treated with a fresh amount of matte as before.

A. R. POWELL.

Electrolytic precipitation of copper. F. LAIST and F. F. FRICK, Assrs. to ANACONDA COPPER MINING Co. (U.S.P. 1,580,614, 13.4.26. Appl., 23.10.25).—In a process involving the leaching of the ore with an acid solution, the electrolysis of the leaching solution to precipitate the copper and regenerate the acid, and the use of this acid for leaching more ore, the leaching solution is also treated with a reagent capable of precipitating iron.

H. HOLMES.

Extraction of copper from ores and the like.

F. M. MURDOCH (Austral. P. 16,878, 14.3.24).—Finely ground copper ores, concentrates, or residues are roasted, if necessary, and leached with a solution of calcium, magnesium, or ferric chloride containing sodium chloride, while a current of sulphur dioxide is passed through the mixture. After filtration copper is recovered from the solution by precipitation with calcium or magnesium hydroxide.

A. R. POWELL.

Precipitation of copper from impure solutions of copper sulphate by electrolysis.

G. BOSSIÈRE (G.P. 426,448, 30.8.24. Conv., 4.3.24).—The electrolysis is carried out in two stages: in the first, when the copper content of the solution is high, pure copper is deposited at the cathode; the second, with the solution weak in copper, is carried out in presence of sulphur dioxide, which is reduced to hydrogen sulphide at the cathode causing the precipitation of the remaining copper and other metals, such as arsenic, as sulphides.

A. COULTHARD.

[White] gold alloy. T. P. SHIELDS, Assr. to SHIELDS & MOORE (U.S.P. 1,580,443, 13.4.26. Appl., 15.5.24).—A white gold alloy contains more than 70% Au, together with more than 15% of nickel and iron, the iron content being more than half the nickel content. A small amount of a deoxidising metal which forms an oxide of the type MO_2 is added to the molten alloy before casting.

A. R. POWELL.

Alloy. Gold alloy. T. P. SHIELDS, Assr. to SHIELDS & MOORE (U.S.P. 1,580,444—5, 13.4.26. Appl., [A] 20.5.25 and [B] 6.7.25).—An intermediate alloy for use in making gold alloys consists of (A) 1—10 pts. of chromium, 24—50 pts. of copper, 10—30 pts. of zinc, and up to about 15 pts. of silver, or (B) 0.1—2 pts. of chromium, 24—50 pts. of copper, and 10—18 pts. of zinc.

A. R. POWELL.

Electrodepositing chromium and preparing baths therefor. C. G. FRNK, Assr. to CHEMICAL TREATMENT Co., INC. (U.S.P. 1,581,188, 20.4.26. Appl., 19.12.25).—Chromium is electrodeposited from solutions of chromic acid by reducing the chromic acid at the cathode in the presence of a catalyst and of a hydrogen film.

A. R. POWELL.

Making metallic chromium. W. E. S. STRONG, C. E. PARSONS, and S. PEACOCK, Assrs. to METAL RESEARCH CORP. (U.S.P. 1,581,698, 20.4.26. Appl., 19.2.25).—A mixture containing chromic oxide, a sodium compound containing oxygen, and carbon is smelted in a blast furnace in such a way that the metallic sodium first liberated reduces the chromic oxide to metallic chromium.

A. R. POWELL.

Extracting arsenides from ores. J. F. SANDERS (U.S.P. 1,581,475, 20.4.26. Appl., 22.4.25).—Ores contaminated with arsenic are finely ground and agitated with carbon disulphide until the arsenic compounds are dissolved.

A. R. POWELL.

Recovery of values from ores. G. D. VAN ARSDALE, H. W. ALDRICH, and W. G. SCOTT, Assrs. to INSPIRATION CONSOLIDATED COPPER Co. (U.S.P. 1,581,479, 20.4.26. Appl., 7.9.23).—In the leaching of ores the residual solids from the first percolation are washed on the counter-current principle to obtain a solution suitable to be added to a rich liquor for the usual treatment. The remaining values are recovered by circulating liquid through the residues and removing the values from the solution after each washing by any method which permits of almost complete recovery.

A. R. POWELL.

Uniting metals [with copper or its alloys]. G. D. BAGLEY, Assr. to ELECTRO-METALLURGICAL Co. (U.S.P. 1,582,024, 27.4.26. Appl., 29.5.26).—Metals of the chromium group may be joined to copper by interposing a nickel layer between the two metals and causing one surface of the nickel to alloy with the chromium group metal and the other surface with the copper.

A. R. POWELL.

Reducing metal sulphides. J. W. BECKMAN, Assr. to BECKMAN-LINDEN ENGINEERING CORP. (U.S.P. 1,582,157, 27.4.26. Appl., 22.5.22).—Metal sulphides may be reduced to the corresponding metal by fusing them with a mixture of a basic oxide, a flux, and carbon.

A. R. POWELL.

Method of operating blast furnaces. R. D. LANCE (F.P. 601,159, 20.10.24).—The process consists in replacing a part or the whole of the solid fuel by gaseous fuel and the enrichment of the furnace gases as they enter the recuperators with a suitable fuel gas.

C. T. GIMINGHAM.

Increasing the oxygen content of air blasts for use in metallurgical processes. A. WAGNER and K. THOMAS (G.P. 426,738, 20.9.22).—The proportion of oxygen is increased by passing the air through porous tubes, through which the nitrogen diffuses more rapidly than the oxygen.

L. A. COLES.

Manufacture of metals and alloys in the electric furnace. E. A. A. GRÖNWALL (F.P. 601,350, 27.7.25).—The process involves the use of one or more vertical electrodes and the introduction of the material to be heated, in a finely divided form, through openings in the walls near the lower end of the electrodes.

C. T. GIMINGHAM.

Smelting metals and ores. SOC. DES PROD. MÉTALLURGIQUES CONSTANT-BRUZAC (F.P. 601,187, 23.10.24).—The carbon dioxide produced by combustion of the fuel is allowed to act on the material to be smelted. A. COULTHARD.

Chill-cast alloy resistant to the action of acids and alkalis. R. SILLER (G.P. 426,835, 16.11.24).—Small quantities of molybdenum, tungsten, nickel, and cobalt are added to the mixture for producing the alloy, the proportions of the molybdenum and tungsten, and of the cobalt, exceeding that of the nickel. L. A. COLES.

Hard alloys for use in the manufacture of tools. SIEMENS & HALSKE A.-G., Assees. of B. FETKENHEUER (G.P. 427,074, 25.3.22).—The alloys are obtained by adding tungsten carbide to molten metals or alloys; e.g., 1–20% is added to a molten alloy containing 1 pt. of chromium and 1–2 pts. of cobalt. L. A. COLES.

Hard alloys which contain silicon and carbon in addition to other substances. SIEMENS & HALSKE A.-G., Assees. of B. FETKENHEUER (Austr. P. 102,286, 18.6.24. Conv., 18.6.23).—Rods containing silicon carbide are dipped into the melt, and heating of the melt is continued until the desired amounts of silicon and carbon have been taken up. A. COULTHARD.

Silver-copper alloys of high silver content. G. A. SCHEID'SCHE AFFINERIE (Austr. P. 102,287, 28.7.24).—The alloys contain silicon or tin or tin-silicon or silicon-bronze to produce the required grade of malleability. A. COULTHARD.

Smelting of ores of antimony, arsenic, and mercury. OESTERR. BAMAG-BÜTTNER-WERKE A.-G., and R. JAHN (Austr. P. 102,293, 11.2.25).—The ground ores are roasted by external heating and the volatilised metal oxides passed through a dust settling chamber kept at a temperature above their precipitation point. The purified vapours are then led to a reduction flame, and the metals so produced are separated by fractional cooling. A. COULTHARD.

Concentrating ores and other materials. E. C. R. MARKS. From ELLIS FLOTATION Co., INC. (E.P. 251,171, 12.10.25).—See U.S.P. 1,555,915; B., 1925, 963.

Manufacturing ductile tungsten. A. JUST, Assr. to GENERAL ELECTRIC Co. (U.S.P. 1,585,497, 18.5.26. Appl., 23.2.24).—See E.P. 214,662; B., 1925, 553.

Recovering vanadium compounds from iron ores containing vanadium and titanium. B. P. F. KJELLBERG (U.S.P. 1,583,053, 4.5.26. Appl., 6.5.25).—See F.P. 598,315; B., 1926, 321.

Casting [easily oxidisable] metals. CHEM. FABR. GRIESHEIM-ELEKTRON, Assees. of A. BECK (U.S.P. 1,584,072, 11.5.26. Appl., 16.7.23).—See G.P. 384,137; B., 1924, 388.

Method of manufacturing metal powder. G. SHIMADZU (U.S.P. 1,584,149, 11.5.26. Appl., 30.1.22).—See E.P. 197,031; B., 1923, 725 A.

Means for agitating and stirring molten metal in open-hearth and like furnaces. J. EDWARDS (E.P. 251,513, 28.9.25).

See also pages 524, Gaseous fuel (U.S.P. 1,581,441), 542, Enamelling cadmium and zinc (U.S.P. 1,583,006), 543, Moulded articles of titanium-containing slag (U.S.P. 1,579,093), 544, Coloured granulated slag (U.S.P. 1,582,318).

XI.—ELECTROTECHNICS.

High-frequency induction furnaces. F. WEVER (Stahl u. Eisen, 1926, 46, 533–536).—The energy requirement of high-frequency induction furnaces is less than that of any other type of furnace. A 35-kg. charge can be melted in 1 hr. using 48 kw. A larger choice of lining materials is made available by the increased life-period of furnace linings due to avoidance of local overheating in the process. Since no electrodes are used, charges can be melted *in vacuo* or in an oxidising or reducing atmosphere. The carbon content of alloy steels can therefore be lowered by oxidation, whilst the velocity of slag-reactions which lower the content of phosphorus and manganese is increased by the agitation of the molten charge caused by eddy-currents. L. M. CLARK.

Colloid condenser. A. NODON (Compt. rend., 1926, 182, 1270–1272).—The colloid condenser consists of sheets of aluminium or magnesium protected by ebonite or paraffined paper. The sheets are separated by a meshed insulator such as canvas in which the interstices are filled with a thick paste of colloidal ferric oxide and glycerin. The foils can be connected to an alternating current supply of a few volts to act as an electrostatic condenser of great capacity. Twenty such couples connected in series will support 110 volts a.c. without appreciable loss. A condenser of this type weighing 100–150 g. possesses a capacity of several hundred microfarads. Colloidal oxides of nickel, chromium, and manganese act similarly. The capacity of the colloid condenser decreases rapidly with increasing surface. The arrangement has the advantage that there is little danger of destruction by internal discharges. R. A. MORTON.

Flames of atomic hydrogen. I. LANGMUIR (Science, 1925, 62, 463–464; Chem. Zentr., 1926, I., 1119).—Atomic hydrogen in considerable concentration is produced by a powerful arc discharge between tungsten electrodes in an atmosphere of hydrogen (cf. E.P. 237,898; B., 1926, 198). By blowing a current of hydrogen from a narrow tube into the arc, an extremely hot flame of atomic hydrogen is produced which melts molybdenum and tungsten with ease. In the presence of hydrogen no oxidation of the metal surface can occur; the

process is therefore suitable for welding (cf. E.P. 237,901; B., 1926, 368). A. B. MANNING.

Flames of atomic hydrogen. I. LANGMUIR (Gen. Electric Rev., 1926, 29, 153—159; Chem. Zentr., 1926, I., 2886; cf. preceding abstract).—The abnormally high thermal conductivity of hydrogen at high temperatures (at 3400° it is about 23 times that of nitrogen) is due to dissociation. Assuming the heat of reaction of the change $H_2 \rightarrow 2H$ to be 98,000 cal., the degrees of dissociation at 1000°, 2000°, 3000°, 4000°, and 5000° Abs. are 3.71×10^{-7} , 1.22×10^{-1} , 9.03, 62.3, and 94.7% respectively. Atomic hydrogen is produced by blowing a current of the gas through an arc of high current density (20 amp. at 300—800 volts) between tungsten electrodes. If the stream of gas containing atomic hydrogen is directed against a metal surface, then in consequence of the catalytic action of the surface, the reverse change, from atomic to molecular hydrogen, takes place with the liberation of 98,000 cal. per 2 g. of hydrogen. In this way it has been possible to melt the most highly refractory substances such as tungsten, tantalum, and thorium oxide, and to attain temperatures higher than those of the oxy-acetylene flame. A. B. MANNING.

Arc welding with atomic hydrogen. R. A. WEIMAN and I. LANGMUIR (Gen. Electric Rev., 1926, 29, 160—168; Chem. Zentr., 1926, I., 2886; cf. preceding abstracts).—Langmuir's atomic hydrogen flame has been applied to the welding of metals. All types of alloys can be welded without deterioration of the metal. A. B. MANNING.

Arc welding in hydrogen and other gases. P. ALEXANDER (Gen. Electric Rev., 1926, 29, 169—174; Chem. Zentr., 1926, I., 2886; cf. preceding abstracts).—In the production of atomic hydrogen for welding, the stream of pure hydrogen may be replaced by mixtures of hydrogen with nitrogen, carbon monoxide, or ammonia. Organic compounds, such as methyl alcohol, may also be used for the purpose. A. B. MANNING.

Influence of cathodic hydrogen on the strength of steel. D. ALEXEJEV and M. POLUKAROV (Z. Elektrochem., 1926, 32, 248—252).—Cathodic polarisation of a steel wire in pure sodium hydroxide solution leaves its strength entirely unaffected, but if a small quantity of mercury ions be present the steel may become as much as 20 times more brittle. In 0.1*N*-sodium hydroxide solution the effect increases with the concentration of mercury, reaching a constant value in a solution containing 0.00125 mg. per 100 c.c. The optimum current density for 0.1*N*-sodium hydroxide and 0.0025 mg. of mercury per 100 c.c. is 0.2 amp. per sq. cm. Polished steel becomes more brittle than steel simply cleaned by washing in ether or benzene. The reduction in strength is less at higher temperatures. Variation in the concentration of sodium hydroxide has a considerable influence; in a solution containing 0.0025 mg. of mercury per 100 c.c., at a current density of 0.2 amp. per sq. cm., and temperature 10°, there is a very pronounced maximum of brittle-

ness when the strength of sodium hydroxide is 0.25*N*. In a solution of pure sulphuric acid the strength of the steel remains unchanged and mercury has no effect. The presence of small quantities of arsenic, however, produces a marked brittleness. The effect diminishes with increase of temperature, disappearing completely at 55—60° in 0.1*N*-sulphuric acid containing 0.025 mg. of arsenic per 100 c.c. with a current density of 0.08 amp. per sq. cm. The arsenic may be made ineffective, however, by rubbing the wire with a brush of glass wool during the electrolysis. The phenomenon is probably very complicated, but appears to depend on the formation of hydrogen as a secondary product of the decomposition of sodium amalgam or of arsenic hydride, not on the primary electrolytic formation. Hydrogen has been observed to penetrate steel, but an X-ray examination of the spacings between the atoms of iron suggests that the penetration of the iron by arsenic or sodium would be much more difficult.

M. S. BURR.

Surface films in cathodic polarisation of metals. LIEBREICH.—See X.

PATENTS.

Protecting electric transformer oils against oxidation. BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF COMP. FRANÇ. POUR L'EXPLOIT. DES PROC. THOMSON-HOUSTON (E.P. 212,928, 13.3.24. Conv., 13.3.23).—Oils used in electric transformers for insulating and cooling purposes are protected against oxidation by means of quinol suspended in the oil at or near the surface exposed to the oxidising influence. If desired the quinol may be placed in a porous container fitted into the upper part of the transformer or may be used in the form of an emulsion, either alone or mixed with a substance, e.g., paraffin wax, which is fusible below the working temperature of the transformer and is soluble in the oil. J. S. G. THOMAS.

Conservation of transformer oils. ALLEGEM. ELEKTRIZITÄTS-GES. (G.P. 426,996, 13.4.23).—While the transformer is running, basic or amphoteric oxides, carbonates, or silicates or refining earths, such as fuller's earth, are introduced into the transformer or into a separate device inserted in the oil-circulating system. Acid substances resulting from changes undergone by the oil are thus rendered harmless. R. A. A. TAYLOR.

[Depolariser for] dry batteries or batteries with a solidified electrolyte. SOC. ANON. "LE CARBONE" (E.P. 228,872, 20.1.25. Conv., 7.2.24).—A depolarising electrolyte for dry batteries consists of a coagulum of fecula containing powdered wood charcoal in suspension and an aqueous solution of ammonium chloride. J. S. G. THOMAS.

Electrolytes for secondary batteries. L. F. DUCKER (E.P. 250,448, 24.7.25).—An electrolyte for use in batteries having lead plates consists of a solution containing 1 lb. of a mixture of magnesium

sulphate (53%) and sodium sulphate (47%), 1 quart of commercial sulphuric acid (d about 1.835), in 3 quarts of water. J. S. G. THOMAS.

Double-fluid [electric] cells. L. DARMONT (E.P. 250,803, 30.6.25. Addn. to 241,729).—In a double-fluid cell of the type described in the previous patent, the layer of cement covering the wall of the porous vessel is coated with a pellicle formed of a solution of an alkali oleate or soap, which, reacting with suitable non-alkaline salts, *e.g.*, the ferric chloride of the depolarising solution and/or the zinc chloride of the exciting solution, produces a layer of insoluble fatty compounds forming a semi-permeable zone, which may be reinforced by addition of sodium phosphate, sodium carbonate, etc. to the alkali oleate employed. If desired, albuminous substances, *e.g.*, corn flour, gelatin, etc., may be added to the coating solution.

J. S. G. THOMAS.

Apparatus for continuously cleaning insulators used in electrical gas cleaning and dust-precipitating installations. LODGE-COTTRELL, LTD. From METALBANK & METALLURGISCHE GES. A.-G. (E.P. 250,499, 17.12.25).—The electrical precipitator is provided with a sloping roof forming a passage with diminishing cross-sectional area, conveying the purified gases to a vertical outlet conduit. The bearer supporting the electrodes passes through openings in the walls of the conduit, and rests upon insulators supported by the sloping roof. A chamber enclosing the insulators, built around the sloping roof and the lower part of the conduit, is provided with openings at the top and bottom so that the heating effect of the sloping roof causes a constant current of hot air to pass upwards through the chamber, thereby keeping the surface of the insulators clean and free from moisture.

L. A. COLES.

Electron discharge devices. STANDARD TELEPHONES AND CABLES, LTD. From BELL TELEPHONE LABORATORIES, INC. (E.P. 250,485, 10.11.25).—An electron discharge device comprises a cathode of thermionically active material and a grid produced by intimately introducing into the material of which the grid is mainly composed a material, *e.g.*, aluminium or an aluminium compound, capable of combining with the particles or vapour emitted from the cathode to form a thermionically inactive compound so that gases evolved within the device are fixed. Thus the grid may be formed of an aluminium alloy. The composite grid is subjected to heat treatment whereby the added material is either diffused through the substance of the grid or forms a homogeneous coating. J. S. G. THOMAS.

Cold exhaustion of incandescence electric lamps and the like. D. S. GUSTIN, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,581,731, 20.4.26. Appl., 25.9.23).—Bulbs containing a filament to which a "getter" consisting of a mixture of phosphorus and graphite has been applied are exhausted cold, flushed with oxygen, and again exhausted cold,

after which the filament is flashed to remove the last traces of gas and moisture. L. A. COLES.

High-frequency apparatus and method of heating. I. LANGMUIR, Assr. to GENERAL ELECTRIC Co. (U.S.P. 1,579,009, 30.3.26. Appl., 23.5.24).—When a body to be heated by induction in a high-frequency field comprises a closed conductor such as a cylinder, a much more rapid heating is effected if the body is rendered discontinuous in the direction in which currents are induced. This result can be applied to heating liquids and gases by enclosing in a tube of suitable non-conducting material, such as glass, a number of coaxial discontinuous metallic cylinders which are heated by the action of an alternating field with a frequency above 10,000 cycles per sec. The gas or liquid to be heated is passed through the glass tube. T. S. WHEELER.

Process of making plates for storage batteries using lead suboxide. G. SHIMADZU (U.S.P. 1,584,479, 11.5.26. Appl., 20.2.23).—See E.P. 198,647; B., 1924, 524.

See also pages 523, Fuel drying plant with electrical precipitator (G.P. 425,499). 524, Electric flue-gas testing apparatus (E.P. 250,478). 525, Separating emulsions (U.S.P. 1,570,209); Electrical dehydrator (U.S.P. 1,579,515); Cracking tars and oils (E.P. 248,830). 526, Cracking hydrocarbon oils (U.S.P. 1,579,554 and 1,579,601). 560, Refining sugar juice by chlorination (E.P. 249,191). 561, Decomposing barium saccharate (G.P. 425,170).

XII.—FATS; OILS; WAXES.

China wood [tung] oil. K. H. BAUER (Chem. Umschau, 1926, 33, 53—56).—Pure α - and β -elæostearic acids were heated to 200° in an atmosphere of carbon dioxide for varying lengths of time, the volatile decomposition products being collected and the changes followed by determination of the chemical constants, by hydrogenation of the products, and by molecular weight determinations. Probably anhydride formation from the free α - or β -acid, together with the formation of some other saponifiable compound, occur during the heating, and examination of the volatile products evolved on heating the β -acid indicates that "cracking" may have occurred. The polymerised β -acid may be a mixture of different acids partially of higher and partially of lower molecular weight than elæostearic acid, with different numbers of unsaturated groups. Molecular weights as determined in camphor solution are in all cases about half the corresponding values found in benzene solution, but these determinations give no definite indications of the changes which have occurred. The author contrasts the results obtained with those obtained by Nagel and Grüss (B., 1926, 164) on heating methyl α - and β -elæostearates at different temperatures in carbon dioxide. H. M. LANGTON.

Action of sulphur on linseed oil. G. S. WHITBY and H. D. CHATAWAY (J.S.C.I., 1926, 45, 115—121 t).—To samples of 20 g. of raw linseed oil definite

amounts of sulphur up to 16.5% were added with agitation and exclusion of air, and at temperatures maintained accurately at 160° (or 170° in some cases), samples being withdrawn at intervals prior to setting and the amount of acetone-insoluble fraction determined. In all cases after heating for 45 min. at 160° all the sulphur added had combined with the oil. Results are given for time of setting and for the concentration of acetone-insoluble material at the moment of gelation. The proportion of sulphur in combination with the oil was always higher in an acetone-insoluble than in a corresponding acetone-soluble fraction. In the action of sulphur and heat on drying oils three well-defined stages succeed each other, viz., chemical combination of sulphur with the oil which takes place comparatively quickly, followed by the formation of an acetone-insoluble product in the second stage, a gradually increasing proportion of the oil changing to a more viscous liquid insoluble in cold acetone. This change is apparently polymerisation, the speed of which is greater, the greater the amount of sulphur present and the higher the temperature. Finally, gelation takes place, and it is presumed that during heating the acetone-insoluble material gradually assumes colloidal dimensions and this colloidal material imbibes the acetone-soluble liquid and any portion of the acetone-insoluble liquid still non-colloidal and a gel then appears rather suddenly. The closely related phenomena of the "drying" of oils and of their thickening under the influence of heat are shown to be similar to the setting of oils under the influence of sulphur and heat.

H. M. LANGTON.

Effect of "blowing" on the composition of certain fatty oils. C. H. THOMSON (Analyst, 1926, 51, 177—180).—The effect of "blowing" at different stages has been examined in the case of cotton seed, whale, sperm, and shark oils, the oils being treated on the large scale, in quantities of about 4 tons, at 115°. Samples were taken at different stages and the specific gravity, viscosity at 93° (Redwood), and the usual constants and variables were determined. Methylated ether was the solvent used to extract the unsaponifiable matter. Tabulated results indicate that iodine value and unsaponifiable matter fall during the oxidation, while specific gravity, viscosity, saponification value, refractive index, and free fatty acids (as oleic) rise to a greater or less extent. Although the constants and variables rise or fall simultaneously at each stage they do not do so at the same rate. The rise in saponification value is concurrent with the rise in soluble volatile acids as indicated by the Reichert-Wolny value. The rise in acid value is also concurrent with the fall in unsaponifiable matter, the increase in acid value being apparently partly due to the oxidation of the unsaponifiable alcohols to fatty acids. Ether-insoluble bromides are not produced from blown shark, whale, or sperm oils, though they are given by the untreated oils. There is practically a regular rise in the viscosity and refractive index after the oil reaches a viscosity of 700 sec., and up to 1800 sec., but below 700 sec. the relationship is irregular. The

various changes caused by "blowing" are brought out clearly in a table giving the analysis of the saponified oils. The amount of glycerol produced is not affected but a considerable quantity of apparently neutral organic matter is formed. Similarity in conditions of blowing must be observed to obtain similar results—the changes in composition are in the same direction as the blowing is continued. In the case of sperm oil blowing was also carried out with the addition of 0.1% of cobalt oxide and certain anomalies were noted.

H. M. LANGTON.

Detection of coconut oil in cacao butter and chocolate. J. RUFFY (Ann. Chim. analyt., 1926, 8, 131—134).—Quantities of coconut oil between 1 and 30% may be detected in cacao butter by applying the method suggested by Härtel and Maranis (B., 1924, 571), but certain modifications were found necessary. The Reichert-Meissl value is determined after saponification with 4 g. of glycerin and 2 c.c. of 1:1 potassium hydroxide solution, the 110 c.c. of distillate being filtered into a 300 c.c. flask and boiled for 15 min. with an upright condenser before titrating. The titrated liquid is slightly acidified, made up to 200 c.c., and 100 c.c. are redistilled and titrated (B). If the value of 100 B/R-M value exceeds 60 adulteration of the cacao butter is to be suspected, 1—2% of coconut oil raising it to about this figure. For milk chocolate the index value must be over 80 before adulteration is assumed.

D. G. HEWER.

Lime pip oil. S. C. MARSHALL and M. S. SALAMON (Analyst, 1926, 51, 237—238).—A crude reddish-brown oil of bitter flavour obtained from West Indian lime pips had the following characteristics: $d^{15.5}$ 0.925, n_D^{40} 1.465, free acidity as oleic acid, 2.8%, saponification value 193.5, iodine value (Wijs, 1 hr.) 109, unsaponifiable matter 0.8%, clouding point in Bellier's test 17.5°. The fatty acids showed n_D^{40} 1.4565, neutralisation value 198, titre 35.8°. The alkali-refined oil (pale yellow and free from bitterness) had $d^{15.5}$ 0.921, n_D^{40} 1.4645, and clouding point in Bellier's test 18.4°.

D. G. HEWER.

Chemical reactions in fat hardening. W. NORMANN (Z. Deuts. Oel- u. Fett-Ind., 1926, 46, 193—195).—Mainly a discussion of the general reactions involved and the behaviour of the nickel-kieselguhr catalyst. Graphs are given for a number of oils showing the progress and completion of hardening, the fall in iodine value and corresponding rise in m.p., and also the fall in iodine value together with progressive fall in refractive index (Zeiss butyrefractometer). For judging the progress of hardening in a particular case the determination at intervals of the refractive index of the oil is the quickest and is better than a method involving the measurement of the amount of hydrogen consumed. An accompanying table shows the amount of hydrogen in cub. m. at 0° and at 20° required to saturate completely 1000 kg. of the commoner fatty acids and fatty oils. In practice oils are seldom hardened till completely saturated. Owing to loss of hydrogen and to side reactions the amount

of hydrogen consumed in a particular hardening is in practice 10–20% in excess of the amount as calculated from the fall in the iodine value.

H. M. LANGTON.

Determination of salt in soap. J. DAVIDSOHN (Chem. Umschau, 1926, 33, 89–90).—Mainly a review of the various methods of determining common salt in soap with particular reference to the method due to Bennett (B., 1921, 741 A). The method gives useful results, but it is preferable to filter off the precipitate of magnesium soap before titrating.

H. M. LANGTON.

Influence of rate of stirring on reaction velocity. HUBER and REID.—See I.

Rancidity and oxidation of fatty oils. RHYS-DAVIES.—See V.

New value in examination of fat mixtures. VAN RAALTE.—See XIX.

PATENTS.

Conversion of neutral or nearly neutral triglycerides, mutually or with other esters. C. VAN LOON (E.P. 249,916, 30.12.24).—Neutral or nearly neutral glycerides are heated for such a time that an interchange of the acid radicals takes place; substances are added which promote catalytically the esterification of free fatty acids and free alcohols, or the action of free alcohols or free acids on triglycerides. For example equal parts of neutral coconut oil and ethyl stearate with the addition of 2% of tin powder were stirred at a temperature of 250°. A slow current of carbon dioxide, to inhibit oxidation, was passed over the mixture and after 1½ hrs. this current was accelerated in order to distil off the more volatile ethyl esters of the coconut oil fatty acids formed during the reaction. A practically neutral distillate of saponif. value 245 (calc. for ethyl stearate 180, and for ethyl laurate 246) was obtained, indicating conversion of the ethyl stearate into ethyl esters of coconut oil fatty acids. In the absence of a catalyst no lower ethyl esters could be isolated even after prolonged heating. Other metals, in addition to tin, and compounds of metals and also organic sulphonic acids may be used as catalysts.

H. M. LANGTON.

Manufacture of a detergent. C. W. FULTON and H. W. HUTTON (E.P. 249,912, 6.12.24).—At least two oils, fatty acids, or resins ordinarily saponifiable are sulphonated. Unsulphonated soap-making oils are then emulsified with the acidulated oils and caustic alkali is added to saponify the soap-making oils and neutralise the acidulated oils. For example coconut oil (1½ pts.) is sulphonated with 25% of its weight of sulphuric acid of d 1.84, and castor oil (1 pt.) is separately sulphonated with 25% of its weight of sulphuric acid. The acidulated oils washed with salt or fresh water, are emulsified together with 3 pts. of unsulphonated soap-making oil, such as cottonseed oil. The whole is warmed and treated with sufficient caustic soda to produce a compound sulphonated soap stock.

H. M. LANGTON.

Soaps and detergent preparations and manufacture thereof. R. VIDAL (E.P. 250,108, 30.9.25. Addn. to 228,390).—Soaps and similar preparations obtained as described in the chief patent (B., 1925, 251) are mixed with aqueous solutions of alkali hypochlorites. For example the pasty soap from 60 pts. by wt. of an aqueous solution containing 33% of castor oil in alkali combination, and 20 pts. of free fatty acid (oleic acid, copra acids, etc.) is added gradually to 80 pts. by wt. of an aqueous solution of sodium hypochlorite at 10°B. (d 1.07). The resulting preparations are easily soluble in water to a clear solution.

H. M. LANGTON.

Combining gases and vapours with liquids. [Hydrogenation of oils.] S. RUBEN (E.P. 251,134, 16.6.25).—See U.S.P. 1,554,296; B., 1925, 999.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Influence of the hardening period upon the protective influence of paints. J. N. FRIEND and B. L. GRIFFIN (J. Oil and Colour Chem. Assoc., 1926, 9, 106–108).—During experimental work on a standard paint for use as a criterion for rust-preventive coatings, a series of mild steel plates were coated with a paint consisting of 80 pts. of Turkey-red oxide of iron and 100 pts. of raw linseed oil (no thinners or driers), and exposed to corrosion by immersion in rain water. The loss in weight of the plate after removal of paint and rust was found to diminish markedly as the interval between painting and exposure was prolonged, the corrosion being three and a half times greater when the time of hardening was 11 days than when the film was allowed to harden for 300 days before exposure. For one coat of this particular paint, a hardening period of about 250–300 days seemed to be a limiting value beyond which no additional protection was afforded. S. S. WOOLF.

Water-line paints [ships' "boot-topping"]. M. RAGG (Farben-Ztg., 1926, 31, 1795–1798).—The results obtained by Figg in an exposure test of water-line paints (J. Oil and Colour Chem. Assoc., 1925, 8, 314–334) are described and criticism is offered mainly on the ground that the panels were unavoidably left to harden for 65 days before exposure. This is divorced from practical ship-painting conditions, as a vessel has to be repainted etc. in the interval between voyages—a matter of a few days generally. It is also affirmed that the weathering effects on stationary iron structures subjected to tides are much less drastic than those on the water-line zone of sea-going ships. Some of the experimental paints prepared by Figg were too rich in volatile thinner, and all were too slow drying.

S. S. WOOLF.

Lead resins. C. COFFIGNIER (Rev. gén. Coll., 1926, 4, 1–8, 65–74).—10, 15, 20, and 25% solutions in benzene, turpentine oil, and white spirit (light petroleum) of resins of lead containing 5 and 10 pts. litharge to 100 pts. of rosin respectively were examined over a period of two months for

viscosity changes, amount and nature of sediment etc. (in the case of the 5% resinates in white spirit observations up to 6 months are recorded). The relative stabilities of the solutions are discussed (cf. B., 1925, 815). S. S. WOOLF.

Copal melting and the copal-melting process in closed kettles. H. RAHDER (Farben-Ztg., 1926, 31, 1742—1743).—Gum-running is a process of the utmost importance in varnish manufacture, but although the presence of air is known to be detrimental, open pots are largely used to-day throughout the industry. The preparation of varnishes in a closed glass retort showed that the removal of air and introduction of inert gases such as carbon dioxide give rise to a lighter product than can be prepared in an open vessel, at the same time permitting the use of higher temperatures than are customary as well as reducing fire risks and assisting the even heating of the mass. The experiments have been extended to the technical scale, and by means of various inlet tubes, the inert gas may be introduced at different places in the kettle, to suit the particular process occurring. Since greater latitude in temperature becomes possible owing to the exclusion of air, control is achieved by fixing temperature and time, without resort to the withdrawal of samples at intervals to ascertain progress. The closed kettle can also be used for oil-thickening and the preparation of glyceryl resinates (ester gums), but heating under reduced pressure presents new, and at present unsurmounted, difficulties.

S. S. WOOLF.

Examination of sandarac. H. WOLFF (Farben-Ztg., 1926, 31, 1682—1683).—Eighteen samples of African (Mogador) sandarac and two samples from Asia Minor showed considerable divergence amongst themselves, when examined for acid value, saponif. value, and percentage soluble in light petroleum, the last (about 15% for the African and about 25% for the Asia Minor samples) being found to be higher than previously reported in the literature. In view of the high acid value of sandarac and the fact that the light petroleum extract gives an orange-red coloration with the Liebermann-Storch reagent, the presence of rosin as an adulterant should not be regarded as proven unless gelatinisation occurs when a benzol solution of the light-petroleum extract is treated with ammonia, in addition to the production of a violet-red coloration in the Liebermann-Storch test.

S. S. WOOLF.

Analysis of oil varnishes. A. GÉRARD-VAUDIN (Ann. Falsif., 1926, 19, 267—275).—A review of published methods.

China wood oil. BAUER.—See XII.

Action of sulphur on linseed oil. WHITBY and CHATAWAY.—See XII.

Effect of blowing on composition of oils. THOMSON.—See XII.

PATENTS.

Compositions containing synthetic resins. G. PETROFF (E.P. 231,431, 16.1.25. Conv., 25.3.24).—The liquid condensation products obtained by warming phenol with formaldehyde in the absence of a catalyst are mixed with hydrocellulose or oxycellulose, alone or together with acid catalysts and with materials such as disintegrated infusible phenol-aldehyde condensation products, barium or calcium sulphate, or kaolin, moulded into shape at the ordinary temperature, and then heated to expel water and effect the final condensation. For example, the oily condensation product from 100 pts. of phenol and 110 pts. of 40% formaldehyde, after removal of the aqueous layer, is mixed with 2 pts. of benzenesulphonic acid, 5 pts. of 40% formaldehyde, and 110 pts. of hydrocellulose. When the mixture has attained a suitable consistency and strength, it is moulded into shape, and dried and hardened by heating gradually to 110°.

L. A. COLES.

Varnish and paint remover. M. PHILLIPS and M. J. Goss; dedicated to the People of the United States (U.S.P. 1,580,914, 13.4.6. Appl., 24.10.25).—A mixture of *p*-cymene and 95% ethyl alcohol is claimed.

S. S. WOOLF.

Paint. G. SHIMADZU (U.S.P. 1,584,152, 11.5.26. Appl., 18.4.24).—See E.P. 215,222; B., 1924, 566.

Corrosion apparatus [for manufacture of white lead]. C. P. TOLMAN, Assr. to NATIONAL LEAD Co. (U.S.P. 1,584,906, 18.5.26. Appl., 26.1.22).—See E.P. 186,114; B., 1922, 905 A.

Cracking tars and oils (E.P. 248,830).—See II.

Lignone derivatives (E.P. 248,834).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Bulk test with *p*-nitrophenol [as mould-preventive for rubber]. H. P. STEVENS (Bull. Rubber Growers' Assoc., 1926, 8, 177).—Large samples of smoked sheet and pale crêpe rubber prepared from latex by coagulation with acetic acid containing *p*-nitrophenol in solution were mould-free, of fairly good appearance, and of normal vulcanisation behaviour.

D. F. TWISS.

Chemical analysis of rubber. C. J. ENKLAAR (Chem. Weekblad, 1926, 23, 209—210).—Different rubbers may be characterised by means of the fractions obtained by dry distillation. A sample of Java crêpe so treated yielded nearly 3% of isoprene; a sample of sheet yielded no isoprene, but products resembling dimethylbutadiene were obtained from it.

S. I. LEVY.

Colloid-chemical processes during hot vulcanisation. H. POHLE (Kolloid-Z., 1926, 39, 1—7).—The nature and degree of dispersion of sulphur in mixtures of rubber and magnesium carbonate, lime, litharge, golden antimony sulphide, and the structure of Congo rubber were examined with the aid of the microscope, and in some cases, the ultra-microscope,

some of the specimens being unvulcanised and others vulcanised. The analogy between the effect of vulcanisation and that of reinforcement by amorphous carbon is again indicated; the probability that vulcanisation involves the formation of an additional disperse phase is confirmed by the concomitant increase in the Tyndall effect. No theory of vulcanisation which disregards the heterogeneous disperse character of the reaction mixture can be satisfactory.

D. F. TWISS.

Effect on mechanical properties of the formation of colloidal precipitates during vulcanisation of rubber. G. MARTIN and W. S. DAVEY (J.S.C.I., 1926, 45, 174—176 T).—Mixings containing acetone-extracted rubber, zinc oxide, and sulphur do not yield more than a trace of zinc sulphide on vulcanisation. In the presence of fatty acids or artificial accelerators appreciable quantities of zinc sulphide are formed. The zinc sulphide appears to be very finely divided but, contrary to Bysow's theory of vulcanisation, it has no vulcanising effect. Moreover, it has no appreciable effect on the physical properties of the vulcanised product.

Vulcanisation of "diffusion-caoutchouc." H. FEUCHTER (Gummi-Ztg., 1926, 40, 1691—1692; cf. B., 1925, 557).—"Diffusion-caoutchouc," *i.e.*, the fraction extractable from deresinised smoked sheet rubber by light petroleum at the ordinary temperature, can be vulcanised in the normal manner when mixed with sulphur, zinc oxide, and piperidine piperidinecarbothionolate. The vulcanised product, however, ages badly.

D. F. TWISS.

Variations in the tensile strength of rubber-sulphur vulcanisates. B. J. EATON and R. O. BISHOP (Malay. Agric. J., 1926, 14, 53—64).—Contrary to the results of earlier workers, the authors find that the addition of mineral particles from the soil does not decrease the tensile strength nor cause any definite increase in the deviation from the mean breaking load; the addition of bark reduces the average tensile strength. The variations in the tensile strength of plantation rubber are not entirely fortuitous, but do not depend on the presence of "dirt."

D. F. TWISS.

Physical properties of rubber. I. Influence of high temperatures on the tensile curve of vulcanised rubber. A. VAN ROSSEM and H. VAN DER MEYDEN (Kolloid-Z., 1926, 39, 69—80; Proc. K. Akad. Wetensch. Amsterdam, 1926, 29, 296—305).—See B., 1926, 502.

PATENTS.

Jelutong product. BEECH-NUT PACKING CO., Asses. of W. A. BEATTY (E.P. 245,405, 13.1.25. Conv., 30.12.24).—Jelutong or pontianac produced by evaporation of fresh or preserved latex contains 20—25% of benzene-insoluble solids. These involve a corresponding increase in the yield and effectively protect the product against atmospheric oxidation, whereas the product obtained by coagulation undergoes rapid oxidation when dry. The evaporated product also has a pleasant taste and odour, and can

be used, without any refining process, for the manufacture of chewing gum.

D. F. TWISS.

Manufacture of rubber compositions. W. B. WIEGAND (E.P. 250,279, 29.9.24).—A high percentage of finely-divided powder, *e.g.*, carbon black (50% and upwards on the rubber content), can be incorporated into natural or concentrated latex by previously adding to the latter an anti-coagulant such as ammonia and an organic substance or substances, *e.g.*, gelatin, capable of lowering the surface tension or increasing the stability of the latex; the powder is introduced as an aqueous emulsion or paste, which also may contain ammonia and a suitable organic material, *e.g.*, saponin. The final addition of a little oleic acid to the mixture increases the degree of dispersion and the stability. Other ingredients such as vulcanising agents and colours may be introduced, together with the emulsified powders. (Reference is directed in pursuance of Sect. 7 (4), of the Patents and Designs Acts, 1907 and 1919, to E.P. 214,210, 212,597, and 205,487.)

D. F. TWISS.

Production of combinations of rubber and paper. E. HOPKINSON and R. P. ROSE, Assrs. to GEN. RUBBER CO. (U.S.P. 1,567,646, 29.12.25. Appl., 22.10.25).—In a mixture of paper pulp and rubber latex the rubber is agglomerated, and then, after further mixing, coagulated.

D. F. TWISS.

Method of making moulded rubber articles, mould and lubricant therefor. B. DALES and W. S. CABLE, Assrs. to B. F. GOODRICH CO. (U.S.P. [A] 1,580,966 and [B] 1,580,967, 13.4.26. Appl., 17.11.24 and 20.11.24).—The mould surfaces which make contact with the rubber during vulcanisation are coated, at least in part, with a substantially water-insoluble composition comprising (A) a wax resistant to absorption by rubber at temperatures of vulcanisation, or (B) sugar-cane wax.

D. F. TWISS.

Drying coagulable liquids [rubber latex]. E. HOPKINSON and R. P. ROSE, Assrs. to GEN. RUBBER CO. (U.S.P. 1,582,604, 27.4.26. Appl., 18.7.23).—A layer of latex is evaporated on a surface from which the dry residue is continuously removed.

D. F. TWISS.

Production of a vulcanising accelerator for rubber. C. O. NORTH (E.P. 251,005, 20.1.25).—See U.S.P. 1,467,984; B., 1923, 1083 A.

XV.—LEATHER; GLUE.

Finer structure of the collagen fibre. A. KÜNTZEL (Collegium, 1926, 176—189; cf. B., 1925, 966).—The collagen fibre consists of fine fibrils of indefinite length, 1×10^{-4} cm. in diameter. The elastic fibres consist of a basic collagen material sheathed by a substance which gives the fibres their characteristic properties. Pancreatin destroys the encasing material but not the whole fibre. The former gives the characteristic colour reactions of elastin, and these show that the gradual decomposition of this substance begins in the middle of the fibrils. Apparently the core of the fibril is the least

resistant and the outer portion the most resistant to reaction. The fibrils are built up of concentric layers. It is probable that the collagen fibril is similarly constituted since the fine fibrils of the grain are more difficult to lime than the coarser fibrils. The finer fibrils are merely outer layers, hence their greater resistance. The fibrils consist of chains of micelles arranged in the direction of the fibre and united by cementing substance, according to the Nägeli theory which explains swelling and taking up of water. The micellar theory explains the double refraction observed with collagen fibrils and the reversal of the double refraction by tanning materials. Gelatin micelles are not the same as collagen micelles.

D. WOODROFFE.

Industrial determination of tannin. L. MEUNIER and A. JAMET (*Chim. et Ind.*, 1926, 15, 499—505).—A critical résumé of the methods used for determining tannin. The official "shake method" is essentially empirical and requires a rigorous standardisation of the conditions under which it is applied, particularly with regard to the clarification and concentration of the solution, the preparation of the hide powder, and the proportion of the latter used. Centrifuging the solution is preferable to filtration through a Berkefeld filter, which removes part of the tannin present in colloidal form. The official method has the advantage of reproducing more nearly the conditions under which the tannin solutions are actually used, but the hide powder adsorbs material other than tannins from the solution. The method of Wilson and Kern (*B.*, 1921, 819A) gives useful information on the amount of material reversibly and irreversibly adsorbed respectively.

A. B. MANNING.

Concentration factor in the fixation of chromium compounds by hide substance from chromium chloride solutions. K. H. GUSTAVSON and P. J. WIDEN (*Collegium*, 1926, 153—165).—Experiments with chromium chloride liquors of 12.8%, 33.0%, and 54.6% basicity and different p_H values and concentration show that the amount of chromium fixed increases and the p_H of the liquors decreases with increased concentration. The p_H of chromium chloride liquors increases after dilution and keeping for 48 hrs., which contrasts with the behaviour of chromium sulphate liquors on dilution. The greatest increase in p_H was observed on keeping very basic chromium chloride solutions containing 20—40 g./litre of chromium. The p_H increases in solutions weaker than 10 g./litre, decreases in stronger solutions, and then increases again at greater concentrations although the liquors have a greater actual acidity in the more concentrated solutions. The different behaviour of chromium sulphates and chlorides is intimately connected with the marked difference in the tendencies of sulphate and chloride groups to form complexes and with the changes which result in the inner sphere. The function of the hydroxyl groups in the complex cation influences the activity of the liquors after dilution. The p_H value of used liquors diminishes on dilution. The p_H curves show a

steep rise with diminishing basicity figure. The amounts of chromium and chlorine fixed by the hide substance increase with increase in concentration. In all cases the basicity of the chrome-collagen compound is much higher than that of the original liquor, a difference which is more marked with acid liquors. This is very different to the results obtained with sulphate liquors. The amount of chromium fixed by hide substance increases with increase in the basicity of the liquors. The hydrogen-ion concentration does not affect the tanning power very much.

D. WOODROFFE.

Changes in the "true tanning figure" during tannage. G. POWARNIN and J. ESROCHI (*Collegium* 1926, 165—176).—Pieces of dried calfskin were tanned with quebracho, mimosa, and willow bark respectively, first in weak liquors, gradually strengthening each day, and finally in a drum with stronger liquors. In one series the pieces of pelt were swollen with $\frac{1}{2}$ % sulphuric acid solution before tanning. The preliminary treatment with sulphuric acid solution diminished the tensile strength of the leather but increased the true tanning figure (cf. *B.*, 1923, 1141A) by 10—16%. The moisture content of the leather and its tensile strength were connected. The true tanning figure was found to increase during the first two weeks of pit tanning, then diminish slightly during the third week, still more during the fourth week, show a large increase after the drumming in the first liquor, but another decrease after drumming in a stronger liquor. The sum of the "true tanning figure" and the "weighting figure" showed a continuous increase during the tannage, 43.85% after 1 week in quebracho liquors to 112% after 4 weeks in liquors and drumming for 24 hrs. This sum was 156% for leather previously swollen with weak acid, compared with 125% for leather tanned unswollen. The tensile strength depends on the moisture content of the leather and also on the "true tanning figure."

D. WOODROFFE.

Properties of shoe leather. III. Ventilating properties. J. A. WILSON and R. O. GUETTLER (*J. Amer. Leather Chem. Assoc.*, 1926, 21, 241—250).—The porosity of leather and its permeability to water vapour, described as ventilating properties, have been measured for the 18 different types of shoe leather described previously (*B.*, 1926, 504) by determining the rate of passage of water through leather from an atmosphere of high to one of low relative humidity. A large amount of water passed through leather as compared with that passing through free space and it increased in the same way with rise of temperature. It was directly dependent on the difference in relative humidity between the two spaces and was constant at 66% of that passing through free space at all relative humidities. The porosity to air and permeability to water vapour both diminished as the amount of grease or oil in the leather was increased, but they bore no direct relation to each other. The application of casein to the grain of the leather diminished the porosity but had little effect on the permeability. The application of collodion diminished both. Chrome

sole and patent leathers had a very small permeability to water vapour and no porosity; suede had maximum ventilating properties. Vegetable-tanned sole leather had a fair degree of permeability but low porosity.
D. WOODROFFE.

Variation in tensile strength of calf leathers with humidity. J. A. WILSON and E. J. KERN (J. Amer. Leather Chem. Assoc., 1926, 21, 250—257). Tensile strength tests on chrome- and vegetable-tanned calfskin pieces at different relative humidities have shown that the strength of chrome leather increases considerably (50%) with increased relative humidity, whereas that of vegetable-tanned leather increases only slightly (13%). The greater strength at higher relative humidities is probably due to the lubricating value of the absorbed water. The greatest amount of water absorbed by the vegetable-tanned leather was 30.18%, against 70.37% for the chrome leather.
D. WOODROFFE.

Bone glues. J. C. KERNOT and (MISS) N. E. SPEER (Second Report, Adhesives Research Committee, 1926, 17—22).—Maceration of bones in dilute alkali before extraction improves the quality of the glue. A short period of treatment under high pressure is preferable to a long period under low pressure for the extraction of undecalcified degreased bones. Air pressure gives the best results; if steam is used, this should be as dry as possible, as wet steam will cause hydrolysis of the gelatin and yield a glue of poor quality.
B. W. CLARKE.

Production of glue and gelatin from fish. J. C. KERNOT and (MISS) N. E. SPEER (Second Report, Adhesives Research Committee, 1926, 23—33).—Fish skins, e.g., the waste from filleting factories, can be used for the manufacture of odourless glue of good quality if subjected to preliminary maceration with dilute alkali and dilute acid (cf. E.P. 235,635; B., 1925, 683). The glue produced compares fairly well with the best hide or bone glue, and has a greater resistance to moisture. The process must be operated in conjunction with the manufacture of fish meal to be commercially successful.
B. W. CLARKE.

Gelatin. S. B. SCHRYVER (Second Report, Adhesives Research Committee, 1926, 7—16).—Gelatin may be purified by electrolysis and subsequent crystallisation, but the purest form is obtained by flocculation of a dilute solution in an electric field. A method has been developed for the analysis of proteins by precipitating the amino-acids as carbamates, by which at least 18 amino-acids can be separated. The Hausmann number is not satisfactory, since it is affected by the pre-treatment of the gelatin, and the treatment of the precursor must be taken into account (cf. Knaggs, Manning, and Schryver, B., 1923, 1084 A; Knaggs, B., 1924, 955; Kingston and Schryver, B., 1924, 955; Knaggs and Schryver, B., 1924, 955, 956; Manning, B., 1924, 955; Horne, B., 1924, 956; Schryver, Buston, and Mukherjee, B., 1925, 521).
B. W. CLARKE.

Adhesives and adhesive action. J. W. McBAIN and D. G. HOPKINS (Second Report, Adhesives Research Committee, 1926, 34—89; cf. B., 1925, 292; 1926, 291).—An adhesive joint may be mechanical, due to the embedding of a tenacious film of adhesive in a porous surface, or specific, between two smooth non-porous surfaces. Metal joints capable of withstanding a pull of $\frac{3}{4}$ ton per sq. in. have been set up with gum arabic, and up to 3 tons with shellac-cresote cements. The highest results are obtained with the thinnest films provided complete contact is obtained with the surfaces. The most accurate results are obtained by measuring the strength of thin films of adhesive, which is often much greater than that of joints set up with the adhesive in the usual way. Gelatin adhesives are the best for wood; high-grade gelatins and glues give practically the same values (12,000 lb. per sq. in. tensile strength). The adhesive properties of gelatin are only slightly affected by prolonged heating at 60°, and of glue at 80°, but higher temperatures produce more marked deterioration. The addition of fillers produces little effect on the strength of the joint. The strongest sodium silicate joints are obtained with the neutral silicate $\text{Na}_2\text{O}, 3\text{SiO}_2$.
B. W. CLARKE.

Mechanical tests of adhesives for timber. ROYAL AIRCRAFT ESTABLISHMENT (Second Report, Adhesives Research Committee, 1926, 90—121).—The double cover plate type of joint gives less variable results for the strength of glued joints than either the simple lap or cantilever type. The energy absorption type is being developed, and shows promise of being a more accurate method. In this method the energy necessary to pull off a given area of cotton webbing which has been glued to a piece of walnut-wood is measured. The variations obtained by present methods render the interpretation of results very difficult.
B. W. CLARKE.

Examination of glued joints by X-rays. (Second Report, Adhesives Research Committee, 1926, 122—123).—X-Ray examination indicates the presence of irregularities in glue films provided a suitable filling agent, e.g., lead sulphate, is added to the glue. The method promises to be useful in the detection of defective glued joints in wood structures.
B. W. CLARKE.

PATENTS.

Products useful in tanning and tawing. M. KAHN, E. LE BRETON, and G. SCHAEFFER (E.P. 247,977, 20.11.24. Conv., 20.11.23).—Brewers' or distillers' yeast, *Rhizopus*, *Amylomyces*, or the like is autolysed, and the autolysate is separated. The cellulosic residues heated to 50° or 80°, and with or without addition of antiseptics, are mixed with fats, oils, soaps, or the like, which may or may not be heated to 40—80° before mixing. Fish may be mixed with yeast, the skeleton of the fish and the autolysate separated from the undigested portions, and an emulsion of fish oil in cellulosic residues so obtained. The products may be used in tanning and tawing.
D. WOODROFFE.

Tanning substances. FARW. VORM. MEISTER, LUCIUS, & BRÜNING, G. KRÄNZLEIN, A. VOSS, and H. GÄRTNER (E.P. 250,398, 24.4.25. Addn. to 211,145 and 240,003; B., 1925, 605, 933).—A phenolic resin is combined with an aromatic or aliphatic compound containing one or more sulphonic groups capable of being exchanged easily, or a phenolic compound is condensed with an aromatic or aliphatic compound containing one or more sulphonic groups capable of being exchanged easily. The product in the latter case may be heated or treated with an agent capable of transforming phenols into phenolic resins. The products are tanning agents.

D. WOODROFFE.

Treatment of hides, skins, etc. C. J. M. M. LE PETIT, Assr. to J. S. BURNS (U.S.P. 1,585,395, 18.5.26. Appl., 11.12.23).—See E.P. 216,546; B., 1924, 644.

Soaking liquor for dried hides and the like. D. J. LLOYD and R. H. PICKARD, Assrs. to BRIT. LEATHER MANUFACTURERS' RESEARCH ASSOC. (U.S.P. 1,584,462, 11.5.26. Appl., 20.5.24).—See E.P. 221,058; B., 1924, 919.

XVI.—AGRICULTURE.

Determination of the p_H of soils by means of Biilmann's quinhydrone electrode. D. J. HISSINK and J. VAN DER SPEK (Verhdl. zweiten Komm. int. bodenk. Ges., 1926, Sept., 12 pp.; Chem. Zentr., 1926, I., 3099).—In using this method, the final reading can be made after 6—10 min. The soil suspension should be as concentrated as possible, and it is better not to add potassium chloride.

C. T. GIMINGHAM.

Titration curves for humic soils. D. J. HISSINK (Verhdl. zweiten Komm. int. bodenk. Ges., 1926, Sept., 20 pp.; Chem. Zentr., 1926, I., 3099).—In the determination of the "lime-requirement" of soils by means of potentiometric titrations, equilibrium is reached in about 3 days. Titration with calcium hydroxide gives more satisfactory results than with sodium hydroxide. Titration should be to the neutral point, not to equivalence.

C. T. GIMINGHAM.

Can the "lime-requirement" of a soil be deduced from the reaction of a suspension of the soil in potassium chloride solution? M. TRÉNEL (Mitt. Int. Bodenk. Ges., 1925, I., 215—221; Chem. Zentr., 1926, I., 2831).—A continuation of earlier work (cf. B., 1925, 328). Since, in "exchange acid" soils, the p_H is a function of the "exchangeable" aluminium ions, the relation between "lime-requirement" and hydrogen-ion concentration can be expressed graphically. The curves for humic soils are clearly distinguished from those for mineral soils, and indicate a higher "lime-requirement" for the latter.

C. T. GIMINGHAM.

Recognition of capacity of soil for nitrogen fixation. S. WINOGRADSKY (Compt. rend., 1926,

182, 1061—1063).—The process of nitrogen fixation in the soil depends on the density of *Azotobacter*, this being in its turn dependent on the fertility of the soil towards the bacteria. In a preliminary test a silica-gel plate 9 cm. in diameter is inoculated with 50—100 grains of the soil, and maintained at 30° for 48 hrs. If the soil is active, 25—50% of the grains should develop colonies of *Azotobacter*; if slightly active, the colonies appear more slowly (4—5 days) and are fewer in number. Incorporation of 0.5 g. of mannitol with 50 g. of soil, followed by similar treatment, should result in spontaneous growth of *Azotobacter* if the soil is active. Similar treatment with 5% of starch should cause formation of colonies in considerable numbers. 1 g. of dry soil spread on a silica-gel plate of 20 cm. diameter, should yield 800—1000 colonies per 100 sq. cm. Determination of the nitrogen fixed by active soil in presence of mannitol should show 20 mg. fixed per 2 g. of mannitol in 120 hrs. With soils of low activity spontaneous growth of *Azotobacter* on addition of mannitol is not observed.

H. J. EVANS.

Carbon dioxide and plant production. E. H. REINAU (Z. angew. Chem., 1926, 39, 495—503; cf. B., 1925, 142, 1002).—A general account of the part played by carbon dioxide in the production of farm and garden crops. The effects of various conditions on the percentage of carbon dioxide in the air, and on the amount evolved from the soil, and the results of the interactions between plant and soil on the distribution of carbon are discussed. The use of artificial means for increasing the carbon dioxide content of the air in the neighbourhood of growing plants is advocated, and the "Oco" process for this purpose, involving burning a special form of compressed carbon, is briefly described.

C. T. GIMINGHAM.

See also A., May, 545, **Effect of metallic aluminium on nitrogen-fixing bacteria** (TRUFFAUT and BEZSSONOFF); **Bacterial oxidation of sulphur** (GUITTONNEAU). 547, **Rapid method for demonstrating effects of plants on a culture solution** (HIBBARD).

Spent molasses wash as fertiliser. CERASOLI.—See XVIII.

PATENTS.

Apparatus for treating acid phosphate [superphosphate]. E. H. ARMSTRONG (U.S.P. 1,576,022, 9.3.26. Appl., 2.12.22).—Phosphates and the like are passed through a tower, or casing, fitted with a series of conical guides spaced a suitable distance apart and with a rotating support carrying spreaders having convex upper surfaces. Means are provided for regulating the passage of hot air through the casing against the materials passing through.

F. SALT.

Production of superphosphates. B. G. KLUGH, Assr. to FEDERAL PHOSPHORUS Co. (U.S.P. 1,583,054, 4.5.26. Appl., 7.12.20).—The process consists in passing gases containing phosphoric oxide through a mass of finely-divided materials which will react

with the phosphoric oxide and retain it, and in passing heated air through the mass to accelerate the reaction.

C. T. GIMMINGHAM.

Process for treating seed grain. FARBW. VORM. MEISTER, LUCIUS, & BRÜNING, Assees. of A. SCHMIDT, A. STEINDORFF, A. FLUSS, and O. SCHAFFRATH (U.S.P. 1,565,237, 8.12.25. Appl., 7.4.23).—Equal parts of a 0.2% solution of the dihydrochloride of diaminodihydroxyarsenobenzene in water and of a 2% solution of copper sulphate are mixed, and the grain to be treated is immersed in this liquid for 1 hr.

C. T. GIMMINGHAM.

Insecticide for use on tobacco plants. H. HOWARD, Assr. to GRASSELLI CHEMICAL CO. (U.S.P. 1,580,200, 13.4.26. Appl., 15.5.22).—A finely divided brown-coloured material, capable of suspension in water, comprising about 2 pts. of an arsenate of manganese (containing not less than 48% of arsenic pentoxide), and about 1 pt. of burnt umber, is claimed as an insecticide.

C. T. GIMMINGHAM.

Procedure for destruction of the vine louse [*Phylloxera*]. J. H. HORST (G.P. 421,241, 23.12.24).—The procedure consists in a winter treatment of the vine stocks with a mixture of nitrobenzene and aniline (or benzonitrile), followed by a summer treatment with pure carbon disulphide. The pest is completely controlled and the vines are not injured. Very little carbon disulphide is required, since most of the insects are killed by the winter treatment.

C. T. GIMMINGHAM.

Means for eradication of animal and plant pests. E. STRICKLER (G.P. 421,833, 10.4.23).—Amyl acetate, mixed with oils, fats, waxes, resins, soaps, paraffin, vaseline, alcohol, etc., or absorbed in porous substances, such as pumice, charcoal, or infusorial earth, is claimed as a means for destroying all animal pests with which it is brought into contact. Weeds, such as plantains, are killed in 2–3 weeks by sprinkling with a dilute solution of amyl acetate.

C. T. GIMMINGHAM.

Procedure for combating plant diseases. A. WACKER, GES. F. ELEKTROCHEM. IND. M.B.H., Assees. of F. KAUFER (G.P. 422,912, 5.5.22).—Cuprous halides (especially cuprous chloride) are claimed as a means for combating plant diseases. They are more easily obtainable than cupric salts, contain a higher proportion of copper and, being very slightly soluble, do not exert a corrosive action on foliage. Addition of alkalis to the spray fluid is usually unnecessary, and a higher concentration of copper can be used than with cupric salts.

C. T. GIMMINGHAM.

Process of making a fertiliser. E. L. PEASE (U.S.P. 1,584,130, 11.5.26. Appl., 1.4.22).—See E.P. 192,778; B., 1923, 321 A.

Producing a fertiliser of urea. J. H. LIDHOLM (U.S.P. 1,584,875, 13.5.26. Appl., 9.1.24).—See F.P. 586,025; B., 1926, 208.

Insecticide (E.P. 234,456).—See XIX.

XVII.—SUGARS; STARCHES; GUMS.

Prevention of the deterioration of raw sugars by inoculation with *Torula*. W. L. OWEN (Facts about Sugar; Int. Sugar J., 1926, 28, 198–203).—Deterioration of raw sugar during storage or shipment by the action of moulds is shown to be prevented by inoculation with *Torula*, the action of which is to restrain the attacking organism by the carbon dioxide developed within the molasses film surrounding the crystals. Sugars thus treated were found to be safer with a non-sucrose to moisture ratio of 2:1 than are inoculated sugars with a ratio of 3:1 (as prescribed by the "factor of safety"), and they are much less hygroscopic and also lighter in colour. The concentration of carbon dioxide required appears to be approximately 0.036%, which is reached generally during the first or second week after inoculation. (Cf. U.S.P., 1,579,387; following.)

J. P. OGILVIE.

Use of chlorine in the refining of sugar. S. OCHI (Trans. Amer. Electrochem. Soc., 1926, 49, 147–156. Advance copy).—Mill juice at p_H 3.5–4.0 is treated with chlorine to give a chlorine concentration not exceeding 0.02*N*, the temperature not exceeding 55°, and the time being less than 30 min., after which the liquid may be filtered for the separation of the suspended matter and colloids thus coagulated, treated with a sufficient amount of activated carbon (as "Norit" or "Darco") to adsorb the excess of chlorine and the hydrochloric and hypochlorous acids, and neutralised. Raw sugar liquor (at about 40° Brix) may similarly be treated, using 0.1–0.4% of chlorine on the weight of solids in the syrup, the temperature being about 50°, but the time should not exceed 30 min., while the amount of carbon added in this case is not less than 2%. Chlorination of sugar juices and liquors is capable of removing 80–90% of the colour under the conditions specified, the bleaching effect being completed by the carbon.

J. P. OGILVIE.

Precipitation of acids during defecation and carbonatation. F. PACHLOPNIK (Z. Zuckerind. Czechoslov., 1926, 50, 269–276, 281–288).—Lactic, saccharic, and hydrochloric acids were not precipitated to any extent during the defecation and carbonatation of beet juice, but phosphoric and oxalic acids were thrown down in both operations, almost quantitatively. Malic acid was eliminated in defecation only slightly, and citric acid to the extent of about 50%, though carbonatation removed the greater part of both. Hardly any sulphuric, aspartic, and glutamic acids were precipitated during defecation, but after carbonatation 74, 60, and 40% respectively passed into the scums. Salts of these acids behaved in general similarly when carbonatation was stopped at an alkalinity of 0.1%, but on continuing approximately to neutrality the quantity of acids precipitated was as a rule diminished.

J. P. OGILVIE.

Decolorisation of intermediate and thick juices by "Carboraffin" and "Norit." A. LINSBAUER and J. FISER (Z. Zuckerind. Czechoslov.,

1926, 50, 225—232, 233—239, 241—247, 249—254).—Extended large-scale tests in a beet factory making white sugar showed that activated carbons applied to the decolorisation of the evaporator syrup (intermediate and thick juice) by pumping through layers of the preparation in special filters resulted in such a very small increase of purity and decrease of colour as to make the procedure uneconomical. Filtration through carbon, however, is of value in producing a bright syrup for crystallising in the vacuum pans, and for this reason the use of very cheap preparations without considering their decolorising power is advised. In the refinery carbons used as pre-filtering media may be of value in reducing the cost of operating the char station.

J. P. OGILVIE.

Rapid method for determining the loss of polarisation in beet analysis. V. STANEK and J. VONDRAK (Z. Zuckerind. Czechoslov., 1926, 50, 257—265).—75 c.c. of juice which had been clarified and "de-leaded" were heated with 5 c.c. of 20% sodium hydroxide during 15 min., cooled, neutralised with acetic acid, made up to 100 c.c., and polarised, a control observation being made with the same amount of juice in the presence of the same amount of sodium acetate. It was found in this way that the dextrorotatory substances likely thus to be destroyed may vary between 0.09 and 0.20% of the root. Their amount in beets depends upon the conditions of cultivation and growth.

J. P. OGILVIE.

Determination of water in sugar factory products by distillation with hydrocarbons. R. VYSKOCIL (Z. Zuckerind. Czechoslov., 1926, 50, 289—295).—An apparatus is described for the rapid determination of water in beet slices, carbonation scums, syrups, massecuites, sugars, molasses, coal, bone-char, etc., by distillation with hydrocarbons. Xylene and toluene gave results with these products which uniformly were practically identical with those found by the desiccation method.

J. P. OGILVIE.

Temperature corrections to readings of Baumé hydrometers. Bureau of Standards Baumé scale for sugar solutions (standard at 20°) (U.S. Bureau of Standards, Circ. 295, 16.1.26, pp. 3).—A table is given by the use of which Baumé hydrometer readings between 0° and 40° B., made at temperatures between 0° and 80°, may be corrected to the standard temperature 20°. It is based upon Plato's values for the thermal expansion of sugar solutions (Wiss. Abh. Kaiserl. Normal Eichungs-Kommission, 1900, 2, 153). The Bureau of Standards official scale, the old or Holland, and the new or Gerlach scales are compared from 40° to 44° B. The first scale gives readings 0.1—0.2° higher than the second and 0.6° lower than the third.

G. T. PEARD.

Preparation of lævulose. R. F. JACKSON, C. G. SILSBEE, and M. J. PROFFITT (Sci. Papers U.S. Bur. Standards, 1926, 20, [519], 587—617; cf. B., 1925, 255).—Juices from the artichoke were found

to contain from 8.1 to 23.8% of lævulose in the form of polysaccharides convertible by acidifying and heating to 70—80° for 30—40 min., while those from the dahlia have 9.3 to 14% of inulin, convertible to a syrup of 86—90% purity. Artichoke juices of high purity after conversion were directly crystallised, but from impure liquors the lævulose was separated as its calcium compound, which was filtered off, washed, decomposed with carbon dioxide, and the resulting solution crystallised after evaporation in a vacuum. In the process of crystallisation from aqueous solutions described, syrups with 90—92% of solids were heated to 55°, "seeded," maintained in slow movement for a sufficient time, and the mass finally centrifuged. From syrups of high purity the crystallisation proceeded so satisfactorily that the temperature could be lowered to about 25° in 24 to 36 hrs., but syrups of lower purity required 4—5 days. Crystals obtained by the authors' procedure had the form of slightly tilted cubes resembling *en masse* ordinary sugar, but adversely affected in an atmosphere of high humidity.

J. P. OGILVIE.

Oxidation products of lactose and maltose on treatment with Fehling's solution. F. HERZFELD (Z. Ver. deuts. Zucker-Ind., 1926, 177—192).—Lactose reduced Kraisy's solution (cf. B., 1921, 315A) much less than Fehling's solution. When after separation of the cuprous oxide, the blue filtrates were acidified and boiled, and the alkalinity was restored, a greater reduction was obtained with the former than with the latter reagent. Kraisy's solution is therefore recommended in preference to the other for the examination of products containing lactose in presence of sucrose. Likewise in determining maltose the weaker copper reagent should be used, dextrin and isomaltose apparently being not affected by it.

J. P. OGILVIE.

Analysis of lime. BAILAR.—See VII.

PATENTS.

Automatically regulating the juice level in evaporating or boiling apparatus of sugar-treating plants. NAAML. VENNOOTS. DE NEDERLANDSCH INDISCHE IND. (E.P. 235,206, 4.6.25. Conv., 7.6.24).—Juice supply or discharge is divided into a substantially constant flow for a quantity below that required for normal working, and a variable flow for a quantity determined by a controlling means, and automatically adjusted by the juice level in the apparatus. An independently arranged tank having a float is placed in communication with both the steam space and juice space of the apparatus of which the juice level is to be regulated, a by-pass connected to the juice supply or discharge conduit being regulated by a controlling device actuated by the float. The controlling device may be so constructed that between the controlling parts thereof in the closed position, there is still a small open passage.

J. P. OGILVIE.

Refining of sugar juice by chlorination. S. OCHI and F. KOTERA (E.P. 249,191, 15.12.24).—

Sugar juice is chlorinated at a temperature not exceeding 55°, the excess of chlorine being removed by means of activated charcoal, and the treated juice neutralised. Chlorination may be effected by blowing chlorine into the juice, or by the electrolysis of an added chloride, (C.f. Ochi, preceding.)

J. P. OGILVIE.

Treatment of molasses and sugary juices, with a view to recovery of the sugar. C. DEGUIDE (E.P. 249,759, 15.10.25).—Monobarium silicate, or the compound, $2\text{SiO}_2 \cdot 3\text{BaO}$, intermediate between this and the dibarium silicate, is added to molasses and juices for the precipitation or destruction of the impurities (in particular gums and reducing sugars), after which the sucrose is precipitated in known manner.

J. P. OGILVIE.

Increasing sugar recovery [in the clarification of cane juices]. R. VACHIER (U.S.P. 1,578,220, 23.3.26. Appl., 12.5.25).—In a process of separating sugar from partially exhausted mud obtained as the residue in the clarification of cane juice, the mud is subjected to fresh water and a quantity of lime sufficient to produce coagulation and to extract the sugar content. Clear sweet-water is thus obtained, suitable as maceration water for use in the milling process.

J. P. OGILVIE.

Clarifier for saccharine solutions [for use in the defecation of cane juices]. R. VACHIER (U.S.P. 1,578,221, 23.3.26. Appl., 15.10.25).—An outer shell has a central cylindrical liquid chamber with a downwardly inclined conical bottom open at its centre, a mud chamber below in the form of an inverted, hollow, truncated cone and an upper scum chamber, also in the form of a hollow truncated cone. Means are provided for supplying the liquid to be clarified to the scum chamber, and for heating it therein. A series of inverted hollow truncated baffle cones project downwards into the shell, each being provided with a cylindrical band at its lower open end, and there is a vertical adjustable overflow cone mounted in the scum chamber with means for carrying off the scum from it. Means are also provided for carrying off the clear liquid from the upper portions of the V-shaped annular spaces between the baffle cones and the outer shell. A rotary hollow perforated shaft projects up through the axis of the mud chamber, scrapers being carried by it for scraping the bottoms of the liquid and mud chambers. Water is supplied under pressure to the shaft, which is rotated, and the mud and liquid contents are removed from the bottom of the mud chamber.

J. P. OGILVIE.

Process of manufacturing beet sugar. E. E. NICHOLSON and R. B. BEAL (U.S.P. 1,578,463, 30.3.26. Appl., 6.10.24).—Raw juice is treated with an excess of calcium oxide, and then with a quantity of water-soluble aluminate to precipitate part of the excess of lime and other hydroxides present. The flocculent granular precipitate is filtered off, after which the filtrate is treated with carbon dioxide, and again filtered.

J. P. OGILVIE.

Preservation of [white] sugar. W. L. OWEN; dedicated to the People of the United States (U.S.P.

1,579,387, 6.4.26. Appl., 14.9.25).—White sugar is preserved by inoculating it with *Torulæ* (cf. p. 559).

J. P. OGILVIE.

Recovering calcium hydrate [hydroxide, from calcium saccharate]. G. M. DRUMMOND, Assr. to HOLLY SUGAR CORP. (U.S.P. 1,580,024, 6.4.26. Appl., 23.9.24).—Calcium hydroxide precipitated from "cold saccharate" mixtures is recovered by introducing the mixture into positive pressure or vacuum "thickeners," from which the clear liquid flows continuously, while the precipitate is removed from the bottom, and filtered off, the filtering medium in the thickener being completely submerged throughout.

J. P. OGILVIE.

Manufacture of sugar. H. SCHREIBER (U.S.P. 1,581,663, 20.4.26. Appl., 23.5.25).—Acidified beet diffusion juice is treated with pepsin, and after reaction has taken place excess of milk of lime is added and carbon dioxide is bubbled through until the alkalinity is reduced to such a point as to obtain a "break" (flocculation). The juice is then filtered and treated subsequently in the usual manner.

G. T. PEARD.

Manufacture of sugar. P. J. MCGUIRE, Assr. to DORR Co. (U.S.P. 1,583,299, 4.5.26. Appl., 25.3.20).—Sugar juice at 85–93° is limed and then, still at the same temperature, subjected to the action of carbon dioxide at a temperature between 85° and 102°.

G. T. PEARD.

Decomposition of barium saccharate. W. SCHEERMESSE (G.P. 425,170, 27.1.25).—Towards the end of the process of decomposing barium saccharate by means of carbon dioxide, a direct or alternating electric current is passed through the solution, the addition of carbon dioxide, preferably under increased pressure, being continued during the passage of the current.

L. A. COLES.

Manufacture of dry halogen-calcium-starch preparations. HENKEL & Co. G.M.B.H. (E.P. 244,708, 20.8.25. Conv., 16.12.24).—The addition of small quantities of borax (up to 2%) to mixtures of starch and calcium chloride (cf. G.P. 308,616; B., 1919, 56A) confers on them the property of forming smooth pastes when mixed with water. These pastes may be used for adhesive purposes or for dressing, finishing, or stiffening fabrics. Excess of borax beyond certain limits gives undesirable properties to the mixtures. Borax in the finest possible state of division is stirred into a mixture of water, calcium chloride, and starch flour shortly before it sets, or the borax may be crushed with the solid calcium chloride and starch.

Ⓐ. COULTHARD.

Manufacture of conversion products of starch. J. M. WIDMER, Assr. to PENICK & FORD, LTD. (U.S.P. 1,578,568, 30.3.26. Appl., 23.5.23).—Starch is converted into glucose in the presence of a hydrolysing acid, which is subsequently neutralised in the presence of phosphoric acid. The precipitated impurities are removed by filtration and the liquor is

treated with tannic acid, the coagulated proteins and insoluble mineral salts are filtered off, and the liquor is treated with carbon to remove excess of tannic acid and traces of colour. B. W. CLARKE.

Purification of starch. M. J. PRUCHA and J. M. WIDMER, Assrs. to PENICK & FORD, LTD. (U.S.P. 1,582,537, 27.4.26. Appl., 10.12.20).—Starch which has been separated from its vegetable source and from the bulk of the proteins, is freed from residual proteins by treating with sulphur dioxide to prevent bacterial life and then with pepsin to render the proteins soluble; the latter are then removed in solution. B. M. VENABLES.

Antifreezing solution (U.S.P. 1,581,179).—See I.

XVIII.—FERMENTATION INDUSTRIES.

Occurrence of methyl alcohol in grapes and the products of their fermentation. J. VILAR (Anal. Asoc. Quim. Argentina, 1926, 13, 543—550).—Methyl alcohol is only formed from the skins and the solid portion of the pulp of grapes. Its production is independent of the access of air during fermentation, is favoured by an alkaline reaction, and hindered by sterilisation or an acid reaction. Methyl alcohol is formed even from skins which have been washed with water and are free from sugar. No practicable method was found for preventing the formation of methyl alcohol where fermentation takes place in the presence of skins.

G. W. ROBINSON.

Clarification of saccharine solutions [musts and wines] with mercuric salts. L. SEMICHON and FLANZY (Ann. Falsif., 1926, 19, 208—226).—Various reagents (salts of lead and mercury and animal charcoal) have been tested for their suitability for the clarification of wines and musts, preparatory to determination of sugars by reduction or rotation. Acid mercuric sulphate is the most suitable, the excess of mercury being precipitated by hydrogen sulphide. The solution is completely decolorised and remains permanently clear; owing to its acidity the reagent may cause partial hydrolysis of hydrolysable sugars such as sucrose and hence may not be used where these are present. Except in the above respect it has no effect upon the sugars present, whereas lead salts and animal charcoal may have a considerable effect. The mercury reagent removes the greater part of the optically active gums and pectins present, which the other two reagents fail to do.

G. T. PEARD.

Behaviour of gelatin towards colouring matter in wines. A. KICKTON and F. MAYER (Z. Unters. Lebensm., 1926, 51, 132—137).—Gelatin-coated filter paper (cf. Van Eck, Pharm. Weekblad, 1925, 62, 365) is not a satisfactory material for detecting the presence of artificial colouring matter in wines. For this purpose the appearance of the precipitate produced by lead acetate may be of value as a preliminary test, but the most trustworthy indications are given by the wool fibre and amyl alcohol tests.

B. W. CLARKE.

Determination of aldehyde in alcoholic liquors by Schiff's reagent. K. C. BAILEY (Proc. Roy. Irish Acad., 1925, 37, B., 58—70).—The results are accurate only when the alcohol-water ratio of the sample is the same as that of the standards. The colour developed is approximately proportional to the aldehyde concentration only in liquors containing 30% of alcohol. Alcohol free from aldehyde is difficult to prepare and is unnecessary since a progressive development of colour in the test solution, upon keeping, has been noticed. Hence the final reading should be taken soon (20—25 min.) after addition of the reagent. The error due to the aldehyde in the alcohol can be calculated and allowed for. The reagent should be prepared from rosaniline base or hydrochloride and should not be yellow. A modified technique for the determination is suggested.

G. T. PEARD.

Utilisation of molasses spent wash as a nitrogen-potash fertiliser. E. CERASOLI (Giorn. Chim. Ind. Appl., 1926, 8, 61—62).—In Italian molasses distilleries the spent wash is concentrated and then calcined to recover the potassium salts, which are mostly exported. In order to obviate the complete loss of the nitrogen compounds involved in this procedure, absorption of the spent wash, after concentration to 32—36° Brix ($d_{15} 1.139$ — 1.159) by kieselguhr or porous silica derived from the treatment of leucitic rocks is recommended. The friable, granular powder thus obtained is named kalinzote and serves as a nitrogen-potash fertiliser.

T. H. POPE.

See also A., May, 542, **Inactivation of invertase by heat** (VON EULER and JOSEPHSON); **Inhibition phenomena during enzymic hydrolysis of sucrose** (VON EULER and JOSEPHSON). 543, **Hydrogen-ion concentration and alcoholic fermentation** (HÄGGLUND, SÖDERBLUM, and TROBERG; HÄGGLUND and AUGUSTSSON); **Alcoholic fermentation by dried yeast** (ABDERHALDEN); **Formation of acetylmethylcarbinol during fermentation of sucrose by yeast** (ELION). 544, **Direct fermentation of maltose by yeast rich in maltase** (WILLSTÄTTER and BAMANN); **Synthesis and fermentation of glycogen by maltase-free yeast** (GOTTSCHALK); **Influence of previous treatment of yeast on affinity constants of invertase** (VON EULER and JOSEPHSON); **Fermentation of galactose by yeast** (VON EULER and NILSSON); **Zymase production and co-enzyme activity** (LEBEDEV); **Reductase of yeast** (VON EULER and NILSSON); **Carbologase** (BEHRENS and IVANOV). 545, **Protective action of milk yeasts on lactic acid bacteria** (SLOBODSKA-ZAYKOVSKA); **Acetone-ethyl alcohol fermentation** (BAKONYI); **Oxidation of glycerol to dihydroxyacetone by bacteria** (VIRTANEN and BÄRLAND).

PATENTS.

Recovery of ammonia from vinasses. G. P. GUIGNARD (U.S.P. 1,576,427, 9.3.26. Appl., 25.10.21).—In a process for recovering the ammonia from vinasses containing water, the product is heated under

vacuum at a temperature constantly maintained between 450° and 550°. J. P. OGILVIE.

Process of making compressed yeast. R. KUSSEROW (U.S.P. 1,580,500, 13.4.26. Appl., 8.5.24).—A nutrient solution for the manufacture of yeast is prepared by diluting molasses containing lactic acid bacteria with water to about *d* 1-160, and then adding at the ordinary temperature sufficient acid to acidify but not to sterilise the solution, a thiosulphate, and yeast nutrients. G. T. PEARD.

Manufacturing alcohol by fermentation. H. W. DAHLBERG, Assr. to GREAT WESTERN SUGAR CO. (U.S.P. 1,581,001, 13.4.26. Appl., 21.3.25).—A beet molasses mash, containing organic nitrogen compounds but substantially no commercially recoverable sugar, is fermented with yeast and the alcohol recovered. G. T. PEARD.

Production of butyl alcohol and acetone. D. A. LEGG, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,582,408, 27.4.26. Appl., 30.3.25).—A mash containing protein-free carbohydrates and soluble nitrogenous matter derived from maize is inoculated with butyl alcohol-acetone bacteria and the products are recovered from the fermented liquor. G. T. PEARD.

Liquid fuel (E.P. 249,577).—See II.

Products useful in tanning (E.P. 247,977).—See XV.

XIX.—FOODS.

Conditioning of wheat. J. S. REMINGTON (Ind. Chem., 1926, 2, 203—207).—Perfect conditioning of wheat, involving both physical and chemical changes, is best carried out by washing the wheat at 25—40° after centrifuging, and passing through a steam-heated jacketed worm at a temperature determined by the grade of wheat or mixture used, and subsequent mellowing for about 3 hrs. in a hot condition. After cooling and storing for a few hours the wheat is ready for milling. Unless the process is carefully controlled, the wheat is very liable to be damaged; it is important to check the moisture content at all stages of the process. Conditioning must be a slow process to develop fully the latent "strength" of the wheat and produce flour of the best quality on milling. The hydrogen-ion concentration developed in the flour during conditioning will affect the baking process. Experiments with a small-scale conditioning apparatus ("hydroliser") will indicate the optimum conditions for temperature, moisture, time, etc. for any given sample of wheat. B. W. CLARKE.

Determination of the degree of milling of flour in bread. H. KALNING (Z. Unters. Lebensm., 1926, 51, 145—147).—The ash content of bread is not a direct guide to the ash content of the flour used, which is an indication of the efficiency of milling. This is owing to the addition of salt during baking; the chlorine is mostly driven off, but the sodium is fixed in the ash as sodium carbonate, and the ash

in the bread is therefore affected by the amount of salt added. A more trustworthy guide is obtained by a determination of the phosphoric acid in the ash (reckoned as P₂O₅); the ash of flour always contains 48—49% P₂O₅, and can thus be calculated from the amount and P₂O₅ content of the bread ash. This method cannot be adopted if calcium phosphate has been used as an improver during baking, as shown by a high calcium content in the ash. The ash of the bread is therefore not a very trustworthy indication of the efficiency of the milling process. B. W. CLARKE.

Determination of fat in milk by the Thomé method. J. B. PLATON (Z. Unters. Lebensm., 1926, 51, 120—125).—The Thomé method (Svensk Kem. Tidsk., 1917, 29, 148) gives slightly higher values than the Röse-Gottlieb method for the determination of fat in whole milk, skimmed milk, and buttermilk, the figure being 0.008 higher, independent of the fat content of the milk. The method differs from the Röse-Gottlieb method in that ethyl ether is used instead of light petroleum for the second extraction, and that the ether can be recovered and used again after distillation. The addition of potassium dichromate to the milk as a preservative does not affect the determination of the fat until the milk is about 3 months old. The Thomé method has the disadvantage that it is much more sensitive to the concentration of alcohol used, and is therefore more subject to error on this account. B. W. CLARKE.

Determination of fat in dried milk. M. DELLEPIANE (Anal. Assoc. [Quim. Argentina, 1925, 13, 533—542).—The author discusses the determination of fat in dried milk by the Werner-Schmidt and Röse-Gottlieb methods. Results obtained by both methods are compared for a number of samples. Somewhat lower figures are obtained by the second method owing to slight saponification of the fat by the ammonia solution which is added at a certain stage in the procedure. G. W. ROBINSON.

New value in the examination of fat mixtures and butter. A. VON RAALTE (Chem. Weekblad, 1926, 23, 222—224).—The "xylene-number" expresses the proportion of volatile fatty acids which remain dissolved in water when the product of steam distillation of the free fatty acids is extracted with xylene. The number is high for butter, since the lower fatty acids are little soluble in xylene, but lower for other fats, owing to the great solubility of caproic and higher acids in this medium. 5 g. of the fat are saponified, the free acids distilled in steam, 110 c.c. of distillate collected and filtered. 100 c.c. of the filtrate are shaken with 10 c.c. of water and 22 c.c. of xylene, the aqueous layer is filtered, and 100 c.c. are titrated with 0.05*N*-sodium hydroxide. The volume required in c.c. $\times 1.21 \div 2$ is the xylene number. The percentage of butter in a fat is calculated from the formula (xylene number $\div 0.63) \div 0.23$. Results of analyses of various mixtures are given. S. I. LEVY.

Utilisation of chlorinated water in canning and preserving processes. H. SERGER (Z. Unters. Lebensm., 1926, 51, 125—132).—Water purified by means of chlorine is quite suitable for use in preserving vegetables etc.; it has no effect on the flavour or appearance of preserved asparagus, carrots, or beans when used for cooking, washing, and filling purposes. No free or loosely combined chlorine can be detected in the water or the vegetables when as much as 1 g. of chlorine per cubic metre of water is used for the purification.

B. W. CLARKE.

Acids in fruits used for jam making. C. F. MUTTELET (Ann. Falsif., 1926, 19, 235—238).—Citric and malic acids may best be determined by precipitation of their barium salts from alcoholic solution (cf. B., 1922, 726 A). The barium citrate is precipitated by the addition of $\frac{1}{2}$ vol. of 95% alcohol, the malate by the addition of 2 vols. of alcohol. Malic acid is found in apples, but not in the fruits usually employed for jelly making (gooseberries, raspberries, strawberries, and currants); hence the detection of malic acid in such "pure fruit" juice denotes adulteration. In the same way adulteration of fruits containing much citric and little malic acid may be detected by a change in the ratio of the two acids. Thus the ratio is altered from 15:1 to 15:10 when cranberry juice is mixed with an equal weight of apple juice. The author recapitulates his earlier work on this subject (cf. B., 1923, 197 A).

G. T. PEARD.

Fermentation of cacao. A. W. KNAPP (J.S.C.I., 1926, 45, 140—142 T).—Experiments on 2000 lb. of selected Trinidad cacao (mainly Forastero) show that unripe cacao does not ferment satisfactorily; it failed to reach the high temperatures normally attained, the yield was low, and the quality inferior. Over-ripe cacao ferments similarly to ripe, but the shells are fragile, and if the over-ripeness approaches germination, the flavour is spoiled. The addition of 4% of a 2½% solution of acetic acid hastened the colour changes associated with fermentation, but did not improve the final product. The addition of 0.003% of diastase (diastatic power 19:1) only slightly increased the breaking down of the pulp, and did not improve the final product. The addition of 0.3% of 3% hydrogen peroxide to the cacao did not materially increase the speed of the oxydase reaction.

Gossypol and *d*-gossypol content of some North Carolina cottonseed meals. F. W. SHERWOOD (J. Agric. Res., 1926, 32, 793—800).—40 N. Carolina cottonseed meals contained the following amounts of gossypol and *d*-gossypol, respectively: 14 open-kettle meals, 0.021—0.150%, 0.544—0.963%; 22 continuous cooker meals, 0.007—0.228%, 0.633—1.076%; 4 "cold-press" meals, 0.02—0.102%, 0.335—0.505%. During the manufacture of the meal, 75% or more of the gossypol present in the kernels (average 0.6%) is converted into the less toxic *d*-gossypol, which unpublished data suggest is a hydrolysis product of the unstable gossypol. The toxicity of diet containing cottonseed meal is

discussed. Assuming that *d*-gossypol is non-toxic, of which there is indirect evidence (cf. Schwartz and Alsberg, B., 1924, 885), only 12.5% of the above meals contained sufficient gossypol to cause injury in feeding experiments to the white rat, using a well-balanced diet containing not more than 50% of cottonseed meal.

E. H. SHARPLES.

See also A., May, 546, **Antirachitic value of irradiated cholesterol and phytosterol** (HESS, WEINSTOCK, and SHERMAN); **Diet and reproduction** (GRUJNS).

Detecting coconut oil in chocolate. RUFFY.—See XII.

PATENTS.

Insecticide or fumigant and fumigating method. R. C. ROARK (E.P. 234,456, 30.3.25. Conv., 26.5.24).—A mixture of a fatty acid ester, of boiling point not higher than 150°, with carbon tetrachloride is claimed as an insecticide or fumigant, for use especially against weevils in grain. Purified ethyl acetate is particularly suitable; a mixture of 2 pts. by volume of the acetate with 3 pts. of carbon tetrachloride is non-inflammable and inoffensive. Grain may be treated in a relatively closed space, such as a box car or bin, using about 40 lb. of the fumigant per 1000 cub. ft. of space. C. T. GIMMINGHAM.

Producing a jelly base. A. GUSMER (U.S.P. 1,580,665, 13.4.26. Appl., 10.1.21).—In the preparation of a jelly base from a pectose extract obtained from vegetable pulp, a fungus possessing both diastatic and zymotic properties is added in slightly acid medium, whereby the starch and albumin constituents of the extract are modified and their precipitation is prevented.

G. T. PEARD.

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

Preparation of lecithin from eggs. C. BARRO (Giorn. Farm. Chim., 1926, 75, 59—61; Chem. Zentr., 1926, I., 3188).—A mixture of 180 g. of yolk of egg with washed and calcined pumice stone is dried *in vacuo* at 50°, extracted with acetone until no more colouring matter is removed, and then treated three times with about 300 c.c. of cold 95% alcohol. On evaporation of the alcohol, lecithin is obtained as a neutral, waxy solid, from which traces of fat are removed by extraction with acetone. On drying the residue *in vacuo* over sulphuric acid, 16.5 g. of lecithin are obtained; the product, however, still contains some cephalin and lutein. The factor, 11.40, corresponding to distearinlecithin, given by Moreau for calculating the weight of lecithin from its P₂O₅ content, is too high for use with egg lecithin, which consists of palmitinoleinlecithin having as factor 10.95; using this value, the product obtained as above contains 94.68% of lecithin.

L. A. COLES.

Determination of the strength of glacial acetic acid. C. O. HARVEY (Analyst, 1926, 51, 238—239).—After determining the approximate freezing point

of the acid in a freezing mixture, a second quantity of acid is cooled about 5° below this point and supercooling allowed to take place to about 1°. The thermometer is then rapidly dipped into the first tube of frozen acid, transferred back with a few adhering crystals to the supercooled acid, and the maximum recorded temperature taken as the freezing point. For concentrations of acid between 97 and 100% by weight the relationship between concentration and freezing point is practically rectilinear and the percentage of anhydrous acetic acid may found be from the equation $x=0.64t+89.5$, where t is the freezing point.
D. G. HEWER.

Alum as catalyst for production of ether [from alcohol]. S. K. K. JATKAR and H. E. WATSON (J.S.C.I., 1926, 45, 168—170 T).—By means of an improved alum catalyst yields of ether about ten times as great as those previously recorded for an equal weight of catalyst have been obtained. The best operating conditions are outlined. The ether is very pure and the quantity of ethylene formed is negligible. A description is given of a unit sufficiently large to supply the requirements of a laboratory.

So-called "Western" (South Dakota) oil of American wormseed. Distillation report for 1924. Origin, nature, and physiological rôle of the essential oil. A. HOGSTAD, JUN. (Amer. J. Pharm., 1926, 98, 188—204).—The distillation results substantiate those of the 1923 harvest (cf. B., 1925, 25), the highest yield of oil being obtained just previous to or at the time of pollination, with a gradual decrease as the plant reaches maturity. The ascaridole content of oils from the fresh plant rises, from flowering to maturity, from 2 to 62% and is increased by air-drying. Most of the oil and ascaridole is obtained in the first 5 or 10 min. of the steam distillation. Of 54 oils examined, only four satisfied the optical activity requirements of the U.S.P. and all failed to comply in relation to solubility in alcohol. A detailed microchemical study of all parts of the plant in all stages of its development is described and photomicrographs are given illustrating stages in the growth of the glandular hairs in which the oil is contained. The oil is apparently secreted by a "resinogenous" layer and serves as a protection to the plant by lowering of transpiration. The value of microchemical studies for the determination of the optimum conditions for distillation is emphasised.
E. H. SHARPLES.

See also A., May, 531, Microchemical reactions of tutocaine (ROSENTHALER). 532, Alkyl esters of aromatic sulphonic acids for alkylation of organic compounds (RODIONOW). 533, Reaction of alkaloids with furfuraldehyde-sulphuric acid (EKKERT). 534, Constitution of cyanomercurisalicyclic acids and of hydrargyrum salicylicum (RUPP and GERSCH). 535, Alkalimetric determination of formaldehyde by means of sodium sulphite (TÄUFEL and WAGNER); Oxidimetric determination of tartaric acid in presence of nitrate (WIKUL). 540, Trypanocidal action of

cinchona alkaloids *in vitro* (TSAKALOTOS); Hypnotics of barbituric acid series (NIELSEN, HIGGINS, and SPEUTH). 541, Pharmacology of benzyl alcohols (CHRISTOMANOS). 547, Ethereal oils of tobacco; Resins and aromatic substances of tobacco; Carbohydrate complex of tobacco (SCHMUCK). 548, Alkaloids of *Ceanothus americanus* (CLARK).

PATENTS.

Manufacture of alkali salts of aromatic sulphochloramides. FARBENFABR. VORM. F. BAYER & Co. (E.P. 241,579, 17.10.25. Conv., 18.10.24).—The compounds are obtained by treating concentrated aqueous solutions of aromatic sulphonamides or their salts with bleaching powder, and simultaneously or subsequently adding an alkali salt, e.g., sodium sulphate or carbonate, containing an acid radical capable of precipitating the calcium. The solution is filtered hot, e.g., at 60—70°, to remove the precipitated calcium salt, and the desired sodium salt crystallises out on cooling.
L. A. COLES.

Manufacture of hydrogenated cinchona alkaloids. HOWARDS & SONS, LTD., and J. W. BLAGDEN (E.P. 250,380, 24.3.25).—Cinchona alkaloids such as quinine or cinchonidine or their substitution derivatives can be hydrogenated to hexahydroderivatives by means of hydrogen in the presence of nickel or other catalyst of this group at temperatures above 30°, and preferably at 60—80° (cf. E.P. 21,883 and 21,948 of 1914; B., 1915, 1225). The parent material may be the non-hydrogenated or the dihydro-derivative of the alkaloid in the form of base or salt and may be used dissolved in water, alcohol, or other solvent. For example, a solution of 5 pts. of quinine in 100 pts. of absolute alcohol is agitated at 20° with 3—4 pts. of a nickel catalyst in an atmosphere of hydrogen under a pressure of 20—40 lb. per sq. in. The absorption of gas ceases when the quantity of hydrogen corresponding to the production of dihydroquinine has been taken up. On raising the temperature above 40°, absorption (slower) again sets in and at about 80° double the amount of hydrogen absorbed at the first stage will have been taken up. The hexahydroquinine is isolated by evaporating the filtered solution.

A. COULTHARD.

Manufacture of methyl alcohol and formaldehyde from methane. BAKELITE GES.M.B.H., and R. HESSEN (G.P. 421,215, 9.12.22).—Methane and excess oxygen, air, or ozonised air, are heated for a short time with non-metallic inorganic porous contact material at a red heat under pressure. Immediately after the reaction, the pressure is reduced and the products (methyl alcohol and formaldehyde) are cooled and condensed by means of a stream of air or gas flowing in the opposite direction. The reaction chamber is closed with a porous or perforated plate, the openings in which are small compared with the inner diameter of the chamber. As catalysts, pumice, brick, or earthenware fragments, highly porous slag, asbestos, quartz sand, or slag wool may be used. It is advantageous to add to the

catalyst (either in layers or mixed with it) at ordinary or raised temperature, substances which take up water and give it up again at high temperatures, such as the oxides and hydroxides of the alkalis or alkaline-earths, magnesium or calcium chlorides, or anhydrous copper sulphate. A. COULTHARD.

Preparation of aminotetrazole. R. STOLLÉ and E. SCHICK (G.P. 426,343, 12.7.24).—Dicyandiamide in aqueous solution reacts quantitatively with 2 mols. of azoimide to give 2 mols. of *C*-aminotetrazole. A. COULTHARD.

Reduction of organic compounds (G.P. 423,029).—See IV.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Relation between time and intensity in photographic exposure. III. L. A. JONES, E. HUSE, and V. C. HALL (J. Opt. Soc. Amer., 1926, 12, 321—348; cf. B., 1925, 972).—The failure of the reciprocity law in the case of Wratten and Wainwright panchromatic and Eastman double coated orthochromatic plates is somewhat greater than that reported previously for fast non-colour-sensitive emulsions. The panchromatic plates show a rise of 0.16 in gamma for a range of increase of intensity of one to eight million. With the orthochromatic plates there is no systematic change of gamma with intensity. Wratten slow ordinary and Eastman slow lantern plates show very great failure of the law, and also a very marked change of gamma with intensity, especially in the lower intensities. Increase of time of development of panchromatic plates results in an increase in amount of the failure, and raises the "optimal intensity." Kron's catenary formula fits the experimental results excellently for the higher speed materials and at high intensities for the materials of low speed. In general relatively fast materials, in which the average grain size is high, have a relatively low value of "optimal intensity," while slow emulsions, which have in general grains of smaller and more uniform size, have much higher "optimal intensities." Gamma is much more dependent on intensity in the case of low-speed fine-grained materials. Fast materials show only a slight variation of density for constant exposure over an enormous range of intensity, while slow emulsions give a great density variation for constant exposure. The decrease of gamma with low intensities in the case of slow emulsions is explained as due to an effective broadening of the sensitivity or size distribution, since when intensity is decreased the "optimal intensity" of the bigger, more sensitive grains is approached, while that of the less sensitive, smaller classes is departed from; the more sensitive grains thus become relatively more effective, and the less sensitive grains relatively less effective in contributing to density. W. CLARK.

Neocyanine: a new sensitiser for the infra-red. M. L. DUNDON, A. L. SCHOEN, and R. M. BRIGGS (J. Opt. Soc. Amer., 1926, 12, 397—400).—Neocyanine, a new sensitiser obtained first during

the manufacture of Kryptocyanine, and less soluble than the latter, sensitises more powerfully than any other known dye at wave-lengths greater than 800 μ . The sensitised band stretches from 700 μ to 900 μ with a maximum at about 830 μ . For the mercury line at 1014 μ , Neocyanine-sensitised plates require one-fifth the exposure of plates sensitised with Dicyanine, and the line at 1128.8 μ is easily photographed with long exposures. The dye gives excellent results by bathing or in the emulsion, and plates can be hypersensitised with ammonia. W. CLARK.

Colour sensitivity of photographic materials. C. E. K. MEES (J. Franklin Inst., 1926, 201, 525—551).—An account is given of work on the normal colour sensitivity of emulsions, and the variation of gamma with wave-length; sensitising by dyes and other means is dealt with somewhat fully, and certain practical applications of sensitised materials are discussed. W. CLARK.

PATENTS.

Photographic developer. P. SCHESTAKOFF and B. MEREJKOVSKY (F.P. 600,532, 21.4.25).—Salts of acids containing nitrogen, hydroxylamine, salts of sulphonic acids of hydroxylamine, and similar compounds are added to the developer to act against the effects of under- and over-exposure, fog, and similar faults. W. CLARK.

Development in bright illumination and daylight. K. BINDER (F.P. 600,801, 15.7.25).—Complex iron phenolic compounds, especially alkali salts of the tripyrocatechol-ferric complex, are added to the developer. The plate is immersed in the developer in red light, after which any convenient illumination can be used. W. CLARK.

XXII.—EXPLOSIVES; MATCHES.

PATENT.

Preventing or quenching explosions or fires in mines and other places. WASHINGTON CHEMICAL CO., LTD., and G. S. NEWALL (E.P. 250,739, 26.3.25).—The explosion of a blasting charge causes the dispersion of dry pulverulent basic magnesium carbonate enclosed in containers or cartridges inserted in the shot-hole in close association with the blasting charge, producing a zone of carbon dioxide, water vapour, and non-combustible powder sufficiently large to prevent or quench explosion or fire due to ignition of coal dust. J. S. G. THOMAS.

XXIII.—SANITATION; WATER PURIFICATION.

See A., May, 515, Preparation of 2:4-dihydroxydiphenylmethane and of 2:4-dihydroxydiphenylethane (KLARMANN). 516, Alkyl and aryl derivatives of phloroglucinol (KLARMANN and FIGDOR). 541, Effect of hydrogen-ion concentration on antiseptic action of certain phenols and aromatic acids (KURODA).

PATENTS

Treating waste liquors containing cyanogen compounds (E.P. 250,824).—See VII.

Fumigant (E.P. 234,456).—See XIX.