

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

FEBRUARY 4, 1927.

### I.—GENERAL; PLANT; MACHINERY.

**Removal of precipitates from settling vessels without running off the supernatant solution.**

STEEN (Chem.-Ztg., 1926, 50, 961—962).—Considerable economy is often obtained if sediments can be removed from large volumes of liquid without pumping the whole bulk of liquid. A cylindrical settling tank with conical bottom may effect this, especially if to the bottom sludge outlet a pressure vessel is connected to balance the liquor head. Several conical sumps so fitted up may be connected to one large tank. If a uniform mixture of coarse and fine sediment is desired the settling vessel may take the form of a ring narrow in section at the bottom. The turbid liquid is fed in centrally and overflows the outer edge of the ring-shaped vessel into a gutter, from which it is removed. The coarse precipitate is deposited at the bottom, and the finer as the liquid ascends the outer wall, but all grades become uniformly mixed in the channel at the bottom, and may be pumped as a thick slurry into a final settling vessel placed centrally within the ring. C. IRWIN.

**Automatic apparatus for the determination of water.** R. KATTWINKEL (Chem.-Ztg., 1926, 50, 927).—An apparatus for the determination of water in a substance by heating with benzene, toluene, or other organic liquid consists of a distillation flask fitted with a reflux condenser having a ground glass joint in the neck of the flask. The issuing vapours enter the condenser through a side tube at the lower end, and the condensed liquid runs into a measuring burette, which consists of an extension of the lower end of the inside tube of the condenser through the outer walls, and terminates in a graduated portion to which a stopcock is attached. The water collects in the burette, and the lighter organic liquid overflows back into the flask through the tube by which it entered the condenser.

A. R. POWELL.

**Apparatus for the determination of moisture by distillation with xylene.** J. PRITZKER and R. JUNGKUNZ (Chem.-Ztg., 1926, 50, 962—963).—The accurate determination of moisture by continuous distillation with xylene requires attention to the form of the cooler. That used by the authors is of the internally-suspended type with outer vapour jacket. 15 c.c. of xylene are used, the weight of substance under test being adjusted as suitable. A graduated tube is connected to the bottom of the cooler with a connexion to enable clear water-free xylene to be returned to the distillation flask. The tube containing the turbid mixture is then centrifuged. A uniform correction of +0.1 c.c. is applied per 5 g. of water. This method is suitable for the determination of moisture in butter, sugar, peat, soap, and the like, and also for such inorganic compounds

as sodium chloride. Ferrous and copper sulphates retain 1 mol. of water of crystallisation when distilled with xylene. C. IRWIN.

**Lubricants for ground glass joints.** BRADLEY and WILSON.—See VIII.

### PATENTS.

**Furnace.** I. HECHENBLEIKNER and T. C. OLIVER, Assrs. to CHEMICAL CONSTRUCTION Co. (U.S.P. 1,603,462, 19.10.26. Appl., 25.3.21).—The nozzle of a burner is directed into a combustion chamber provided with an annular baffle surrounding the burner and a predetermined zone of the burner flame. A ring of gas inlets is arranged around the baffle, and these are constructed and arranged for imparting a whirling motion of the gas about the zone of the flame beyond the baffle.

H. HOLMES.

**Heat exchangers.** GRISCOM-RUSSELL Co., Assees. of J. PRICE (E.P. 248,712, 4.1.26. Conv., 3.3.25).—In a tubular heat exchanger where one liquid flows through two (or more) bundles of tubes in series, all the tubes at one end are supported in one tube plate, but at the other end the tube plate is subdivided, and one section is connected to another by means having a certain amount of "give," comprising, substantially, four elbows and a straight pipe. B. M. VENABLES.

**Pulverising apparatus.** C. J. REED, Assr. to J. G. BERRYHILL (U.S.P. 1,603,639, 19.10.26. Appl., 1.12.21).—One end of a horizontal high-speed rotary drum is provided with a stationary closure, and rollers, interconnected by a frame, move freely within the drum to co-operate with the surface for grinding. The frame carries means for continuously removing material from the drum, and the upper end of the stationary closure carries means for removing the fine dust produced.

H. HOLMES.

**Grinding members for ball, drum, and tubular mills.** O. KORDT (E.P. 261,664, 4.8.26).—A grinding member, preferably of steel, is of approximately equal diameter vertically and transversely, and is formed with flattened and plane surfaces at varying angles. Suitable shapes are described. H. HOLMES.

**Grinding, pulverising, or disintegrating mills.** W. A. CLOUD (E.P. 262,033, 20.7.25).—A disintegrator of the type comprising several rings of intercalating pins, or of similar construction, is provided with a tangential outlet for the ground material, which can be moved round the casing to the most advantageous position. B. M. VENABLES.

**Retort.** I. B. NEWBERRY (E.P. 261,575, 19.1.26).—The retort has an outer vertical and cylindrical casing, within which is a series of superposed rotating shelves. The material passes through the retort by being scraped



progressively from one shelf to the next below. Around the bottom of the retort is a series of upwardly-directed steam jets, whilst a steam-jet ejector is fitted in the off-take pipe at the top of the retort. The gases and vapours are thus removed while a partial vacuum is maintained in the retort. The shelves are progressively heated from below. R. A. A. TAYLOR.

**Drum dryer.** D. J. VAN MARLE, ASSR. to BUFFALO FOUNDRY & MACHINE Co., INC. (U.S.P. 1,603,886, 19.10.26. Appl., 17.1.25).—Liquid to be dried is delivered from a supply tank below a rotary drum to a feeding pan provided with an inclined bottom extending tangentially to the drum surface above the tank, with its inner edge spaced from the surface. The liquid is thus fed to the drum in a direction opposite to the rotation, and the excess, not adhering to the surface, passes back to the tank over the spaced edge. H. HOLMES.

**Dryer.** T. H. RHOADS, ASSR. to PROCTOR & SCHWARTZ, INC. (U.S.P. 1,604,074, 19.10.26. Appl., 19.6.26).—The drier is divided into two sections side by side, each provided with a fan for circulating air. The articles to be dried are carried on trucks within a tunnel extending through both sections. The air circulates first through one section, then through the other, and back through the first section, and means are provided for diverting a proportion of the air through the tunnel in direct contact with the articles. H. HOLMES.

**Dryer.** W. M. SCHWARTZ, ASSR. to PROCTOR & SCHWARTZ, INC. (U.S.P. 1,604,079, 19.10.26. Appl., 5.12.23).—The casing is made in sections, each comprising inner and outer metallic plates separated by non-conducting material. The edges of the sections are offset to provide overlap joints between the sections. The edges of the plates are disconnected at the offsets, and non-conducting strips are placed between the sections to prevent transmission of heat from the inner to the outer plates. H. HOLMES.

**Centrifugal separators.** A. W. EMPSON (E.P. 260,720, 15.9.25).—A centrifugal separator is provided with a number of baffles forming ducts, parallel to the wall and to the axis of the bowl, through which the fluid under treatment has to flow. The fluid may be constrained to flow through several ducts in series or series-parallel. B. M. VENABLES.

**Mixing machinery or apparatus.** J. B. PARNALL and W. W. VEITCH (E.P. 260,745, 10.10.25).—An open pan is rotated, and into it, off centre, so as to touch or nearly touch the side at one point, depends a stirring device free to rotate under the influence of the material in the pan. The stirring device is counterbalanced so that it may be easily lifted out, and, if desired, a fixed scraper may be provided in the pan at a point opposite the rotatable stirrer. B. M. VENABLES.

**Mixing methods and apparatus [for liming sugar juices etc.].** GILCHRIST & Co., Assees. of R. W. SHAFOR (E.P. 247,541, 25.1.26. Conv., 13.2.25).—The ingredients of the mixture are circulated together in a closed cycle, and baffles are provided for dividing the stream and producing sudden changes in the direction of flow of the subdivisions. Successive groups of the baffles may be staggered for re-distributing the mixture between

adjacent subdivisions. Means are provided for regulating the temperature of the stream. H. HOLMES.

**Apparatus for making emulsions.** L. KIRSCHBRAUN (U.S.P. 1,603,546, 19.10.26. Appl., 9.4.24).—A horizontal open-ended conduit is mounted within a mixing chamber provided with inlets for the materials to be mixed, and agitating elements are provided in the conduit and in the chamber. H. HOLMES.

**Distilling apparatus for volatile liquids.** H. A. HILLS (U.S.P. 1,604,353, 26.10.26. Appl., 14.1.21).—A still for purifying cleansing liquids has a nozzle placed horizontally and adjacent to the bottom of the still, which injects steam into the liquid. The nozzle has a sleeve which forms an annular chamber, and this is supplied with fluid from the bottom of the still simultaneously with the exhaust from the nozzle, the injected steam and fluid being deflected upward. W. G. CAREY.

**Removing volatile fluids from solids containing same.** R. E. WILSON, ASSR. to BALTIMORE GAS ENGINEERING CORP. (U.S.P. 1,603,568, 19.10.26. Appl., 1.6.22).—The solid material and an absorbent are subjected to relative movement while maintaining them in close proximity. H. HOLMES.

**Apparatus for separating solids [coal] from liquids.** WOODALL-DUCKHAM (1920), LTD., and R. KRALL (E.P. 261,850—1, 28.8.25).—The apparatus described in E.P. 219,651 (B., 1925, 271), which is especially suitable for drying coal, is provided with (A) a stationary or revolving hood to collect the water sprayed out of the material, and (B), a special form of joint between the rotating perforated screen and the stationary lower part of the apparatus to prevent re-mingling of coal and water. B. M. VENABLES.

**Centrifugal separation of particles suspended in a fluid.** TIRAGE & VENTILATION MÉCANIQUES (E.P. 261,617, 10.5.26. Conv., 28.11.25).—The fluid passes through the separating chamber between axially-alined inlet and outlet passages. The inlet passage is provided with stationary vanes for imparting gyratory, centrifugal motion to the fluid, and the outlet passage with stationary vanes, oppositely disposed to those in the inlet passage, for restoring to the fluid the pressure lost within the chamber. The two sets of vanes may be mounted one at each end of a drum, the chamber being constituted by an annular space between the drum and an outer casing. This space may be provided with an annular partition dividing the chamber into inner and outer zones communicating freely at each end. H. HOLMES.

**Filtering device.** J. J. NAUGLE (E.P. 261,204, 14.12.25).—A number of filter leaves are supported on a framework or yoke within a pressure-tight casing. The yoke and leaves can be rotated by means of trunnions extending through the casing, and the interior of each leaf communicates with the outside through a trunnion, and the duct is provided with a sight glass. In operation the prefilter, comprising, say, a sugar solution with a filter aid such as "suchar" (activated carbon) in suspension, is circulated through the casing from top to bottom, and the filtrate is drawn off through the majority (but not all) of the leaves until the casing is practically full of suchar and residue. The circulating



feed is then shut off and the pulp exhausted as dry as possible by means of the hitherto unused leaves. During the filtering any leaf that passes muddy liquor may be shut off and, at the end of the cycle, changed for a sound one. After filtering, the residue is subjected to a hot reverse wash, the heat being maintained by live steam injected into the casing, and after washing the suchar and residue are sluiced out by jets of water (or other liquid) within the casing, but outside the leaves. The filters are rotated slowly during filtration, but rapidly during washing and discharging.

B. M. VENABLES.

**[Continuous] pressure filters.** J. A. McCASKELL (E.P. 261,218, 18.1.26).—A number of filter discs are slowly rotated in a closed tank partly filled with the prefilter under pressure. Each leaf comprises a number of segments, the interiors of which are put, at the proper time, by means of conduits in the shaft and bearings of the filter, in communication with either a filtrate drain or a supply of air under a higher pressure than the filtering pressure. The supply of the pressure air is pulsed by a valve and shakes off the cakes of residue when the segments are raised above the liquid. The dislodged residue is caught by deflectors and discharged from the casing by means such as a worm conveyor.

B. M. VENABLES.

**Apparatus for crystallisation.** E. C. R. MARKS. From GRASSELLI CHEMICAL CO. (E.P. 261,085, 11.8.25).—A body of saturated solution is maintained in circulation, passing in the upward direction through the crystalliser proper, which is a vessel larger at the top than at the bottom, the average increase of section being less than a 60° cone. A multitude of small crystals are maintained in suspension in the crystalliser, and are caused to grow by the addition of supersaturated solution through the side of the crystalliser. Large crystals drop to the bottom of the crystalliser, and are removed by a conduit separate from and below the inlet for circulating solution. To prevent clogging of the return pipes etc. the solution is slightly heated when passing through the upper part of the crystalliser, and it may be reconditioned, *e.g.*, by evaporation, before re-use.

B. M. VENABLES.

**Purifying steam, vapours, and gases centrifugally.** W. ALEXANDER (E.P. 261,642, 17.6.26).—The steam etc. enters the separating chamber through convergent, parallel flow openings in the end plate separated by oblique or curved vanes which impart a vortex motion to the steam. The outer walls of the openings are parts of a concentric circular extension of the chamber wall. The end of the chamber adjacent to the drain outlet is sloped inwards, and the diameter of the chamber may be constant or may increase towards the drain outlet end. The admission end plate may be flat or convex towards the chamber. The steam outlet may be at the same end as the steam inlet, and its entrance may be provided with a double lip to prevent entraining of liquid with the outgoing steam.

H. HOLMES.

**Controlling the temperature of heated air in drying and like operations [for crops etc.].** B. J. OWEN (E.P. 261,446, 23.4.26).—A louvred or other shutter or damper on the outlet duct of the air-heating apparatus is controlled by a bimetallic strip thermostat mounted in the duct. The strip comprises two blades of

metals, such as brass and mild steel, having widely different coefficients of expansion, and is curved to extend adjacent to the curved wall of the duct. The strip is fixed to the wall at one end, and carries at the other end a lug extending through a slit in the wall for connexion to the shutter through bell-cranks and connecting rods.

H. HOLMES.

**Producing cooling liquids.** STRATMANN & WERNER, and F. WERNER (E.P. 260,760, 24.10.25).—A cooling mixture of low freezing point, which does not set free hydrochloric acid or other residues which corrode metal containers, is made by adding, under pressure, a solution of crystallised calcium chloride, which has previously been slowly heated, to a solution of magnesium chloride kept in motion at a high velocity.

W. G. CAREY.

**Liquefaction apparatus.** J. L. SCHLITT and W. DENNIS, Assrs. to AIR REDUCTION Co., INC. (U.S.P. 1,604,240, 26.10.26. Appl., 5.2.26).—A mixture of gases to be separated is introduced into tubes extending through a vaporiser section of a liquefaction column. Means are provided for withdrawing a gaseous product from the tubes, and means within the tubes retard the backward return of liquid formed therein in contact with the gaseous mixture.

H. HOLMES.

**Apparatus for liquefaction and separation of gaseous mixtures.** C. C. VAN NUYS, Assr. to AIR REDUCTION Co., INC. (U.S.P. 1,604,248—9, 26.10.26. Appl., 6.12.24; cf. B., 1925, 575, 791).—[A] The gaseous mixture is delivered to a chamber beneath a liquid-containing compartment within the rectifying column, and tubes passing through the compartment connect the chamber to a header above the compartment. Means are provided for delivering liquid from the chamber to a rectifier above the compartment, and for effecting preliminary rectification of the liquid formed in the tubes by contact with the incoming mixture. [B] The mixture is subjected to selective liquefaction to form a liquid containing the constituents and a residual gas, which is withdrawn and expanded, and the expanded gas is used to cool the entering mixture. The liquid is delivered to the top of a rectifier, and means are provided for withdrawing from the top of the rectifier an effluent having substantially the composition of the entering gaseous mixture, and for withdrawing separately the vapour from the rectified liquid.

H. HOLMES.

**Antifreezing mixture.** G. A. SCHULTHEISS (U.S.P. 1,605,377, 2.11.26. Appl., 25.9.25).—This consists of an oil dissolved in an alcohol and a calcium chloride solution.

H. ROYAL-DAWSON.

**Separation of the constituents of ternary gaseous mixtures.** C. C. VAN NUYS, Assr. to AIR REDUCTION Co., INC. (U.S.P. 1,607,323, 16.11.26. Appl., 11.7.25).—See E.P. 255,104; B., 1926, 1000.

**Separation of gases liquefying at different temperatures.** G. CLAUDE, Assr. to LAZOTE, INC. (U.S.P. 1,605,646, 2.11.26. Appl., 30.1.20. Conv., 10.2.14).—See F.P. 475,346; B., 1916, 31.

**Crucible.** J. D. M. SMITH, Assr. to W. & J. GEORGE, LTD. (U.S.P. 1,609,096, 30.11.26. Appl., 8.6.26. Conv., 5.6.25).—See E.P. 252,901; B., 1926, 650.

**Oil-fired furnace.** C. HAWKINS (E.P. 261,126, 27.8.25).



**Dry packing insulation against heat exchange with protective mantle.** F. BOHLE and R. SCHRÖDER (E.P. 252,198, 17.5.26. Conv., 16.5.25).

**Means for regulating the admission of steam to a heat accumulator.** SIEMENS-SCHUCKERTWERKE GES.M.B.H. (E.P. 255,491, 17.7.26. Conv., 18.7.25).

**Regulation of liquid level in a pressure vessel.** G. CAMPBELL (E.P. 245,445, 28.12.25. Conv., 5.1.25).

**Containers for storage and transport of gases.** UNION GÉNÉRALE CO-OPERATIVE (E.P. 252,386, 20.5.26. Conv., 20.5.25).

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

**Industrial conversion of coal into organic products of technical use.** G. PATART (Chim. et Ind., 1926, 16, 713—729).—The direct treatment of coal, *e.g.*, distillation, hydrogenation, etc., offers only a partial solution to the problem of its conversion into pure organic substances of use industrially. Some technical points are discussed relating to the conversion of coal into such products, *e.g.*, alcohols, acids, ketones, etc., by the direct combination of carbon monoxide and hydrogen under pressure in the presence of a catalyst (*cf.* B., 1925, 83). As this reaction proceeds, the concentration of the inert gases in the residual gas mixture increases to a point at which further reaction becomes practically ineffective, when the apparatus is purged and refilled. The loss of the active gases carried away with the inert gases on purging makes it necessary to use a process for gasifying the coal giving a minimum concentration of inert constituents. It is preferable to carbonise bituminous coals at high or at low temperatures, or possibly under pressure, and subsequently to gasify the coke, rather than to gasify directly; the synthesis of alcohols etc. is thus most favourably and economically carried out in association with a gas works. Technical applications of the products of the process are briefly discussed.

A. B. MANNING.

**Oxidation of pyrites as a factor in spontaneous combustion of coal.** S. H. LI and S. W. PARR (Ind. Eng. Chem., 1926, 18, 1299—1304).—The rate of oxidation between 25° and 100° of pyrites in coal in general increases with the fineness of division and with the temperature. At the ordinary temperature oxidation is favoured by high moisture content of the coal and by the presence of a current of oxygen, possibly because the moisture as it is removed is replaced by oxygen in a highly reactive state. In presence of certain types of clay, which probably contain sulphur-oxidising bacteria, oxidation may be as rapid at 25° as it is at 100° when the clay is absent. This is of importance, because it seems that in favourable circumstances the rate of oxidation of the coal itself may become serious above about 80°. Marcasite oxidises at about the same rate as pyrites does, but the process is facilitated by the ease with which it disintegrates into fine particles. R. CUTHILL.

**Rôle of metallic colloids in suppression of detonation.** H. L. OLIN, C. D. READ, and A. W. GOOS (Ind. Eng. Chem., 1926, 18, 1316—1318).—The efficiencies of

various substances as anti-knock materials have been compared by adding them to the petrol used to drive an engine and observing the minimum load necessary to produce knocking. The efficacy of a solution of lead tetraethyl which has been heated under pressure so as to produce some colloidal lead is not impaired if the latter is removed with an activated carbon, and seems, by comparison with an experiment in which undecomposed lead tetraethyl was used, to be due solely to the undecomposed compound. A colloidal solution of nickel has no measurable anti-knock effect. It thus seems that free metallic particles suspended in the combustion zone of the cylinder do not necessarily suppress detonation (*cf.* Charch, Mack, and Boord, B., 1926, 570).  
R. CUTHILL.

**Apparatus for the determination of moisture [in peat] by distillation with xylene.** PRITZKER and JUNGKUNZ.—See I.

### PATENTS.

**Fuel briquettes.** G. PLOCHMANN (U.S.P. 1,600,065, 14.9.26. Appl., 7.7.25. Conv., 2.5.25).—Lignites which have retained their woody structure are subjected to preliminary drying, so regulated that the material does not become actively hygroscopic. The pores of the dried material are charged with bitumen by exposure to the distillation gases of some suitable carbonaceous material, and the product is briquetted. S. PEXTON.

**Production of briquetting material.** W. PRUIJS, Assee. of H. SCHRADER (E.P. 250,925, 6.4.26. Conv., 15.4.25).—Coal dust is fed on to a rotating plate, whence it is thrown off and falls in the form of a hollow cylindrical cascade. A spray is arranged in the axis of, and below, the plate and rotates counter to the plate. It projects the binding material against the inner wall of the cascade of coal.  
R. A. A. TAYLOR.

**Obtaining hydrogenation gas for hydrogenating coal and hydrocarbons from the waste gases of the hydrogenation.** F. BERGIUS (E.P. 244,730, 16.11.25. Conv., 18.12.24).—The purified gaseous products from the hydrogenation of coal are treated with steam at a high temperature (about 1100°), whereby the hydrocarbon gases are converted into carbon monoxide and hydrogen is generated. The mixture of these two gases is then treated with steam at a lower temperature (300—500°), when, in the presence of a suitable catalyst, the carbon monoxide reacts to give carbon dioxide and hydrogen. The carbon dioxide is removed by washing.  
R. A. A. TAYLOR.

**Fuels from coal and oil mixtures.** W. E. TRENT (E.P. 261,594, 15.2.26).—Finely pulverised coal is treated with oil and then heated, when the oils distilled from the coal are recovered for the treatment of a further quantity of coal.  
R. A. A. TAYLOR.

**Production of carbon.** F. FISCHER and H. TROPSCH (E.P. 252,152, 5.5.26. Conv., 15.5.25).—Carbon monoxide, or a gas containing it, having been freed from sulphur compounds, is passed at 500°, preferably under pressure, over a catalyst of finely divided iron free from sulphur. Carbon is deposited on the iron and carbon dioxide is formed. The process is continued until the separated carbon includes not more than 10% of iron.  
R. A. A. TAYLOR.



**Production of lamp black.** H. A. PAGENKOFF (U.S.P. 1,606,380, 9.11.26. Appl., 16.9.24).—Oil and old tyres are burned in a restricted supply of air. The carbon is recovered from the smoke, first by deposition on a cooled revolving surface, then by passage through cooled and restricted spaces, and finally by spraying with water.  
R. A. A. TAYLOR.

**[Peat] carbonising apparatus.** W. C. E. DOMINICK, Assr. to DEUTSCHE VERKOHLUNGS- & DESTILLATIONSGES. M.B.H. (U.S.P. 1,604,738, 26.10.26. Appl., 14.7.24. Conv., 29.12.23).—An apparatus for the distillation of peat comprises 12 retorts arranged in a ring. The process employed is similar to that described by W. Dominick (cf. B., 1925, 59).  
T. S. WHEELER.

**Apparatus for the destructive distillation of organic matter.** W. K. LJUNGDAHL, Assr. to A. W. COOPER (U.S.P. 1,603,343 and 1,603,345, 19.10.26. Appl., [A] 25.1.23; [B] 19.1.26).—[A] Wood is placed in a chamber divided into separate compartments which are heated by gases from a furnace. One of the compartments forms a part of the closed circulatory system for the heating gases. The furnace is intermittently cut off from the circulatory system, when air is admitted to the former, and the heated products from it are used to heat another compartment. A cooling compartment is provided for the charge. [B] Each of a series of furnaces is connected to a regenerator and to a common equalising chamber. Any regenerator can be disconnected from the equalising chamber, but furnace gases may be caused to heat the disconnected regenerator. The gases from the equalising chamber pass to a retort.  
R. A. A. TAYLOR.

**Apparatus for distillation.** W. K. LJUNGDAHL (U.S.P. 1,603,344, 19.10.26. Appl., 13.6.23).—The retort (for wood) is heated by steam radiators. A system of pipes is connected to the retort, and is provided with means for cooling and condensing the vapours flowing through it, after which it discharges into the atmosphere. The separated condensates are collected in a still which is heated by exhaust steam from the radiators.  
R. A. A. TAYLOR.

**Kiln for destructive distillation of organic matter.** W. K. LJUNGDAHL, Assr. to A. W. COOPER (U.S.P. 1,603,346, 19.10.26. Appl., 19.1.26).—A long retort has an inlet door at one end and an outlet door at the other. At the opposite ends also are ports, one for admitting and one for exhausting gas, but the port at the inlet end is at least one charge-carrier length from the door.  
R. A. A. TAYLOR.

**Tunnel kiln.** W. K. LJUNGDAHL, Assr. to A. W. COOPER (U.S.P. 1,603,347, 19.10.26. Appl., 19.1.26).—In a kiln which has a heating and a cooling compartment which are to be traversed successively by the charge, means are provided for circulating a cooling gas, for cooling the gas, and, if required, for introducing to the cooling compartment gas from the heating compartment.  
R. A. A. TAYLOR.

**Oil-shale retort.** C. E. BARNHART (U.S.P. 1,604,253, 26.10.26. Appl., 14.2.25).—The retort consists of a number of horizontal roasting hearths, above and below each of which is a heating flue. The shale is drawn over the topmost hearth by a rotating screw, and falls

on to the second hearth, and so on, thus passing across each hearth until discharged from the retort. Revolving gas-tight partitions at various points between certain hearths divide the retort into separate heat-treating sections.  
W. N. HOYTE.

**Treating bituminiferous materials.** J. J. JAKOWSKY (U.S.P. 1,602,819, 12.10.26. Appl., 16.5.22).—The apparatus comprises a tubular retort, in which is a stationary heat-transmitting element, spaced from the walls of the retort, and having a flat face in contact with the material, which is fed into the retort and swept across the face of the heat element. Bases and vapours of distillation are withdrawn by a conduit.  
H. MOORE.

**Electrical treatment [cracking] of gases and vapours [hydrocarbons].** H. R. ROWLAND, Assr. to C. AND C. DEVELOPING CO. (U.S.P. 1,601,771, 5.10.26. Appl., 15.11.23).—An apparatus for subjecting hydrocarbons to the action of the silent electric discharge comprises a cylindrical chamber, which contains a wire electrode, connected to insulators at the top and bottom. The vapours under treatment pass up through the cylinder, and are subjected to the action of a discharge passing from the wire to the walls of the vessel. To prevent deposit of conducting material, e.g., carbon, on the insulators between the wire and the chamber a portion of the material under treatment is sprayed in liquid form upon them.  
T. S. WHEELER.

**Removal of deposited material from cracking plant.** L. C. HUFF, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,603,541, 19.10.26. Appl., 26.1.23. Renewed 27.12.24).—An expansive fluid, which is a non-supporter of combustion, is injected into the body of the deposited carbonaceous material, thereby disrupting it, and facilitating its removal.  
W. N. HOYTE.

**Reflux column.** W. W. HOLLAND, Assr. to STANDARD OIL CO. (U.S.P. 1,603,772, 19.10.26. Appl., 12.5.24).—A reflux column for attachment to a (petroleum) still is fitted with a number of horizontal plates, spaced about 1 ft. apart, and each perforated with about 70  $\frac{1}{4}$  in. holes per square foot. From each hole depends a tube about 5 in. in length, and these tubes form the only method of passage from one space to the next.  
T. S. WHEELER.

**Distillation [of petroleum oil].** W. F. FARAGHER, W. A. GRUSE, and F. H. GARNER, Assrs. to GULF REFINING CO. (U.S.P. 1,601,729, 5.10.26. Appl., 25.5.21).—Petroleum oil or the like is fractionally distilled by heating it in a still below the cracking temperature. The oil is caused to circulate vertically in the body of the oil and laterally at the top and bottom, by means of currents of gas, to promote mixing and uniformity of temperature, the distilled vapours being collected.  
H. MOORE.

**Treating water-in-oil emulsions. Breaking petroleum emulsions.** M. DE GROOTE, Assr. to W. S. BARNICKEL & CO. (U.S.P. 1,606,698—9, 9.11.26. Appl., [A] 23.12.24; [B] 29.4.26).—(A) An emulsion of water in oil is treated with an insoluble substance, which is wetted by water, can be dispersed colloiddally in it, and is free from a fatty acid radical, e.g., magnesium resinate or calcium cresylate. The emulsion is then passed through a bed of fine sand, or other substance composed



of insoluble particles, separated by capillary spaces, and capable of being wetted by oil, when separation rapidly ensues. (B) The emulsion is passed through a bed of fine sand resting on an earthed plate, between which and a disc placed above the sand a potential of about 11,000 volts is maintained. T. S. WHEELER.

**Process of treating hydrocarbons.** H. I. LEA (U.S.P. 1,606,246, 9.11.26. Appl., 26.5.22).—Hydrocarbons and chlorine are passed at an elevated temperature over alumina, whereby aluminium chloride is formed. The excess hydrocarbons are then acted upon by the aluminium chloride. R. A. A. TAYLOR.

**Treatment of carbonaceous material.** H. NIELSEN, Assr. to B. LAING (U.S.P. 1,605,761, 2.11.26. Appl., 7.3.25).—See E.P. 198,385; B., 1923, 760.

**Drying of finely-divided carbonised fuel.** W. BROADBRIDGE, E. EDSEER, and W. W. STENNING, Assrs. to MINERALS SEPARATION NORTH AMERICAN CORP. (U.S.P. 1,606,867, 16.11.26. Appl., 20.11.22. Conv., 29.11.21).—See E.P. 193,947; B., 1923, 438 A.

**Process for purifying oils and the like.** H. M. RIDGE and W. R. HODGKINSON (U.S.P. 1,608,339, 23.11.26. Appl., 3.3.23. Conv., 4.3.22).—See E.P. 203,354; B., 1923, 1118.

**Vibratory screens or sieves [for coal etc.].** T. WOOD (E.P. 261,561, 18.12.25).

**[Apparatus for] manufacture of gas retorts or other pipes from clay or the like.** R. GAY and GIBBONS (DUDLEY), LTD. (E.P. 261,512, 24.9.25).

**Apparatus for use in the discharging of retorts.** H. J. TOOGOOD, and R. DEMPSTER & SONS, LTD. (E.P. 261,059, 9.7.25).

**Gaseous fuel burners.** C. F. HAMMOND and W. SHACKLETON (E.P. 261,808, 2.6.25).

**Apparatus for separating solids from liquids** (E.P. 261,850—1).—See I.

**Retort [for treating shale]** (E.P. 261,575).—See I.

**Producing ammonium sulphate [from coal gas]** (E.P. 262,320).—See VII.

**Purifying and concentrating sulphur dioxide** (U.S.P. 1,606,299).—See VII.

**Solvents** (E.P. 259,293).—See XX.

### III.—TAR AND TAR PRODUCTS.

**Tar from a commercial low-temperature retort.** H. A. CURTIS and H. A. BEEKHUIS (Chem. Met. Eng., 1926, 33, 666—669).—Some properties of a tar produced by the carbonisation of a West Virginia coal in a "Carbo-coal" retort at 600—650° are described. The m.p. and the free carbon in the pitch are linear functions of the volume per cent. of the tar distilled. The tar was repeatedly fractionated, cuts being taken at 10—15° intervals up to 270°, and the tar acids were separated from each fraction. The sp. gr. of the tar acid fractions pass through a minimum at b.p. 224—238°. Other samples of the tar acids were methylated and the methyl ethers fractionated; the sp. gr. again pass through a minimum, the curve of average equivalent weights showing a corresponding break. The condensation

products of the tar acids and formaldehyde were inferior to those obtained by using pure phenol. The condensation products with sulphur chloride may serve as acid-resisting varnishes. The neutral oils were fractionated; if care is taken to remove completely the sulphuric acid used in extracting the bases, the lower-boiling fractions of the neutral oil retain their light yellow colour for several months, whilst the fractions above 220° darken slowly. All the neutral oil fractions contain sulphur (0.4—0.6%), which is not readily removed. The neutral oils are highly unsaturated, absorbing, for example, up to 50% of their weight of chlorine; they are, however, not so readily attacked by potassium permanganate. Liquid-phase cracking of the neutral oils is very slow below 400°, whilst above 450° coke formation becomes rapid. The effect of cracking any fraction is to widen the boiling range, but the same relative difference in the boiling range between fractions is maintained even after extensive cracking.

A. B. MANNING.

**Behaviour of lignite producer tar and lignite low-temperature tar when heated under pressure.** A. ERDÉLY (Brennstoff-Chem., 1926, 7, 359—361).—Producer tar oils and low-temperature tar oils, obtained from Tata lignite, give a lower yield of cracked oils than American gas oil, the optimum temperature in each case being 450°. When heated with hydrogen under pressure the lignite tar oils give 23—30% of cracked oils, as against 33% for the gas oil. Tar oils that have been refined with sulphuric acid and sodium hydroxide give the same yield as the gas oil, but the refining losses are heavy. The benzene obtained from the gas oil does not darken when kept, whereas that from the tar oils does. The presence of hydrogen under pressure increases the benzene yield and lessens the losses due to the formation of coke and gas. W. T. K. BRAUNHOLTZ.

#### PATENTS.

**Process for obtaining pressed asphalt compositions and similar products** (E.P. 248,752).—See IX.

**Drying of finely-divided carbonised fuel** (U.S.P. 1,606,867).—See II.

### IV.—DYESTUFFS AND INTERMEDIATES.

#### PATENTS.

**Manufacture of vat dyestuffs of the anthraquinone series.** A. G. BLOXAM. From CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 261,510, 23.9.25).—Arylketodihydro-*p*-thiazines, dihalogenated in the methylene group (cf. G.P. 379,318, A., 1924, i, 1118), are condensed with aryl-*o*-diamines or *N*-alkyl or -aryl derivatives of these. The products are vat dyes, if at least one reaction component is an anthraquinone derivative. Use of primary *o*-diamines yields dyes which are not fast to alkali, but which can be converted into alkali-fast dyes by alkylation. *E.g.*, 7.5 pts. of anthraquinonedichloroketodihydro-1 : 2-thiazine, 4.8 pts. of 1 : 2-diaminoanthraquinone, 80 pts. (by vol.) of nitrobenzene, and 3.5 pts. of anhydrous sodium acetate are heated for 2—3 hrs. at 140—150°. The yellowish-brown product obtained by distilling off the nitrobenzene in steam, after purification, dyes cotton yellow from a blue vat. It is ethylated by heating 4 pts. of it with 30 pts. of



ethyl toluenesulphonate at 200° for 7 hrs. The melt is diluted with alcohol and the yellow dye filtered off, washed, and purified by hypochlorite solution. The structure of the condensation products has not been determined.

A. DAVIDSON.

**Azo dyestuffs.** W. CARPMAEL. From I.-G. FARBENIND. A.-G. (E.P. 261,568, 1.1.26).—Four azo dyes of the class described in E.P. 245,865 (B., 1926, 233), but not specifically mentioned therein, are prepared by coupling diazotised 4-amino-4'-hydroxydiphenylsulphone-2:3'-dicarboxylic acid with 2-naphthol-8-sulphonic acid, and by coupling diazotised 2-amino-4-sulpho-4'-hydroxydiphenylsulphone-3'-carboxylic acid with methyl ketol, with 2-amino-8-naphthol-6-sulphonic acid, and with *o*-amino-*p*-tolyl ether, the product in the last case being diazotised and coupled with salicylic acid.

A. DAVIDSON.

**Dry diazo composition.** F. KELLER and K. SCHNITZPAHN, Assrs. to GRASSELLI DYESTUFF CORP. U.S.P. 1,607,462, 16.11.26. Appl., 20.1.26. Conv., 30.1.25; cf. E.P. 21,227/94; B., 1895, 962).—Organic diazo compounds, when mixed with about an equal weight of crystalline alum or aluminium sulphate, about two-thirds dehydrated, can be stored for long periods without deterioration.

T. S. WHEELER.

**Preparation useful for the production of dyestuffs.** A. ZITSCHER, Assr. to I.-G. FARBENIND. A.-G. (U.S.P. 1,608,284, 23.11.26. Appl., 2.6.24. Conv., 11.6.23).—See E.P. 217,594; B., 1925, 541.

**Process for making azo-dyestuff from barbituric acid.** G. DE MONTMOLLIN, F. STRAUB, and J. SPIELER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,606,209, 9.11.26. Appl., 1.12.25. Conv., 24.12.24).—See E.P. 257,797; B., 1926, 910.

**Sulphur dye and process of making same.** W. MERTE, Assr. to KALLE & Co. A.-G. (U.S.P. 1,606,254, 9.11.26. Appl., 17.10.24. Conv., 12.8.22).—See F.P. 588,874; B., 1926, 149.

**Manufacture of a new thioindoxyl derivative.** SOC. CHEM. IND. IN BASLE (E.P. 246,156, 14.1.26. Conv., 14.1.25).—See Swiss P. 111,997; B., 1926, 943.

**Manufacture of ice colours** (E.P. 261,542).—See VI.

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

**Decomposition of raw vegetable fibrous material by chlorine.** H. WENZL (Papierfabr., 1926, 24, 809—817).—Comparisons are made between the actions of chlorine gas, chlorine water, and hypochlorite solution upon both pine-wood meal and straw under standard conditions. With wood, the highest percentage of chlorine used and the lowest ratio of combined chlorine to hydrochloric acid produced are shown when the chlorination is carried out in carbon tetrachloride, hydrolysis under these conditions being negligible. The values are a minimum and maximum, respectively, when chlorine water is used, hydrolysis of the chlorine then being complete. With straw, chlorine gas and hypochlorite are similar in action, whilst chlorine water differs in producing a smaller increase of copper number and decrease of baryta number. Further comparison is made by the effect of these three reagents upon the pentosan

and lignin content and upon the yield of pure cellulose. Differences in the nature of the reactions with chlorine water and gas are ascribed to the fact that in the former case a substitution action predominates, whilst in the latter oxidation is the more important. Acid produced in the chlorine water process exerts a strongly hydrolysing action on the cellulose, resulting in a smaller yield of pure cellulose. Decomposition of wood by a single chlorination is successful only if a vigorous preliminary treatment with alkali is given. On a price basis, the chlorine gas method is the most economical.

B. P. RIDGE.

**Viscosity of cuprammonium solutions of cotton cellulose.** F. C. HAHN and H. BRADSHAW (Ind. Eng. Chem., 1926, 18, 1259—1260).—Viscosity measurements by the falling ball method show that linters yield more viscous solutions than does long-staple cotton. If  $\eta$  is the viscosity of a solution containing  $m$  g. of solute/100 c.c., and  $\eta'$  is the viscosity of the solvent, then  $\log \eta/\eta'$  is a linear function of  $m$ , except at the higher concentrations (cf. Farrow and Neale, B., 1924, 506).

R. CUTHILL.

**Viscose. III. Conditions of xanthation.** M. NUMA (Cellulose Ind. Tokyo, 1926, 2, 321—335).—The quantity of carbon disulphide required to produce complete xanthation is 40—75%; the highest yield of xanthate and the most stable conditions correspond with about 50%. When the temperature of ripening is comparatively high it has little influence on the quantity of combined alkali, but the combined sulphur decreases rapidly. Hence it is contended that the combined alkali and combined sulphur do not necessarily vary proportionally. The most favourable temperature of xanthation is 15°. During the ripening the changes in the colloidal properties of the viscose gel particles and in the structure of the viscose sol take place at different velocities. The optimum duration of ripening is between 1 and 4 days, corresponding with the optimum properties of the filament or film. When this duration is surpassed the combined alkali and sulphur decrease, and the viscose tends to become unstable. The optimum degree of ripening may be recognised by the appearance of the precipitate formed on decomposing viscose of a certain concentration with 20% sulphuric acid. At the optimum point the regenerated cellulose is in the form of long fibrous particles, and should not be short or powdery. The dispersity of the viscose gel increases with the advance of ripening up to a maximum on the third day, and then decreases. J. F. BRIGGS.

**[Detection of abnormal treatment of] artificial silk (cellulose hydrate).** O. FAUST and K. LITTMANN (Cellulosechem., 1926, 7, 166—168).—Determinations such as that of the copper number, breaking strain, etc. do not give reliable indications of the treatment to which a sample of cellulose has been subjected. The following method, due to Weltzien (B., 1926, 737), is shown to yield useful information concerning the previous history of the sample. The dry material is treated with water, then with 4% sodium hydroxide, then with water again, and is then dried, the percentage change in the fibre-length at each stage being determined, and the procedure is then repeated on the same sample. A normal fibre increases to 104% of its original length on treatment



with water, shrinks to 103% with 4% sodium hydroxide solution, increases again to 104% with water, and on drying shrinks to slightly less than its original length. On repetition of the treatment closely similar results are obtained. With a fibre which had previously undergone abnormal mechanical strain by being loaded with a weight of 50 g., the fibre length after water treatment was 100.5%, after alkali treatment 98.5%, after second water treatment 99.2%, on drying 95.5% of the original. When the process was repeated this material gave results similar to those of a normal fibre. The effect of abnormal mechanical strain, which may result in a change in the dyeing properties and lustre of the fabric, can thus be corrected by alkaline immersion. A fibre which had undergone abnormal chemical treatment, viz., immersion in 0.48% sulphuric acid solution and dehydration at 60°, gave the following results. After water treatment the fibre length was 101.5% of the original, after alkali treatment 100.5%, after second water treatment 102%, on drying 94%. A repetition of the procedure gave 106.5%, 106%, 107%, and 100%. The effect of acid cannot, therefore, be counteracted by alkaline immersion, as the treatment results in a fibre which shows abnormally high changes in length with water and sodium hydroxide. This may be due to the formation with acid of alkali-soluble products which are removed during the first treatment, and the fibre is thus rendered more penetrable. Treatment of the fibre with dilute hydrochloric acid has a similar effect, but acetic acid causes little change. Excessive bleaching results in abnormally high changes in length when the fibre is treated with water and alkali in the manner described above (106%, 105%, 107%, and 97%), and still more abnormal results when the process is repeated (108.5%, 108%, 109%, and 98.5%). It is therefore claimed that the method not only detects fibres which have undergone severe treatment, but also gives an indication of the conditions to which the sample has been subjected.

W. J. POWELL.

**Cellulose benzoate.** K. ATSUKI and K. SHIMOYAMA (Cellulose Ind. Tokyo, 1926, 2, 336—345).—Cellulose dibenzoate is prepared by treating regenerated or normal cellulose with 35% sodium hydroxide solution, ageing for 24 hrs., and shaking with benzoyl chloride dissolved in benzene. The proportions used are  $C_6H_{10}O_5 : 4NaOH : 10C_7H_5OCl$ . The reaction is finished off by heating at 50—60° for 1—2 hrs. The dibenzoate from regenerated cellulose gives clear solutions in chloroform and acetone of very low viscosity, that from normal cellulose is not completely soluble, and the viscosity is very high. The films have a tensile strength of 5 kg./sq. mm. and elongation of 5%; they are very brittle.

J. F. BRIGGS.

**Cellulose lacquer solvents etc.** VAN HOEK.—See XIII.

#### PATENTS.

**Washing, cleaning, or otherwise treating wools, silk, soiled fabrics, or other fibrous textile materials.** E. C. DUHAMEL and COMP. GEN. DES INDUSTRIES TEXTILES (E.P. 259,969, 20.5.25. Conv., 20.5.24).—The material to be washed or cleaned is first treated with hot, relatively concentrated, suint liquor for a considerable time before entering the first of a series

of washing bowls containing concentrated suint liquor. Apparatus for this purpose comprises an arrangement of a vertical shaft, down which the material passes while being impregnated with liquor, conveying lattices, beater rolls for opening and distributing the material uniformly in thin layers, squeeze rollers to express mud and grease, and a series of washing bowls in which the suint liquor is maintained in a successively cleaner condition.

B. P. RIDGE.

**Cleaning wool, fabrics, etc.** B. PAYNE (U.S.P. 1,608,418, 23.11.26. Appl., 21.6.24).—Bentonite is agitated with water, the mixture kept for maximum hydration, and the supernatant colloidal solution used for washing textile materials.

A. J. HALL.

**Improving cellulosic materials, in particular vegetable textile fabrics.** L. DE WOLF (E.P. 252,360, 12.5.26. Conv., 25.5.25).—The rate of defibring, scouring, and bleaching of cellulosic materials by the action of sodium hydroxide or alkaline phenolate solutions is greatly enhanced by use of a mixture of the two, the former being in excess. With certain fibres a bath containing at least 5% of sodium hydroxide and 1% of phenol results in scouring, lanification, mercerisation, or even gelatinisation and bleaching in one operation. The efficiency of oxidising agents such as permanganates or alkali hypochlorites, when added to such a bath, is increased by the action of the phenol in solution.

B. P. RIDGE.

**Making artificial silk and staple fibre.** M. HÖLKEN (U.S.P. 1,607,400, 16.11.26. Appl., 26.8.21. Conv., 2.12.18).—A precipitating bath is prepared by mechanically softening a hard water, to deprive it of its temporary hardness, and adding a substance which destroys the permanent hardness, but does not affect the product precipitated in the bath.

H. ROYAL-DAWSON.

**Treating artificial silk fibres.** OBERREINISCHE HANDELSGES.M.B.H. (E.P. 260,935, 19.1.26. Conv., 6.11.25).—The spinning of artificial silk is facilitated by improving the adhering and intertwining properties of the fibres. For this purpose a roughening treatment is given prior to spinning or carding, in which substances such as talc, cement, oxides, or salts are fixed upon the fibres by precipitation, drying, or by adhesives. Curling or crisping, and pounding, pressing, fulling, or centrifuging the fibres in addition to the above treatment, further improve their spinning qualities.

B. P. RIDGE.

**Manufacture of a rubberised fibre composition.** W. G. O'BRIEN, ASSR. to THE GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,606,595, 9.11.26. Appl., 31.7.22. Renewed 12.1.26).—The fibres are first separated, rubber particles caused to adhere to them, the product is treated with a liquid comprising a mixture of rubber precipitant and rubber solvent, and finally cured. The solvent is removed from a rubber solution by successive treatments with liquid mixtures containing increasing proportions of alcohol from 23 to 60%.

B. P. RIDGE.

**Manufacture of cellulose-containing solutions.** A. CLASSEN (E.P. 261,494, 3.9.25. Addn. to 236,281; B., 1926, 707).—The preparation of cellulose solutions in concentrated hydrochloric acid is improved by the use of catalysts in the form of metal oxides upon which the action of the acid is negligible, or nearly so. Increased



efficiency is obtained by employing two or more catalysts (such as metals and metal oxides in close contact) and by increasing the active surface. Thus the catalyst may be in the form of fine wire, or as metal wool, the wires being either superficially oxidised or provided with active surface layers, or it may be deposited on metallic or non-metallic carriers which are themselves either active or inert.

B. P. RIDGE.

**Stabilised cellulose ester.** H. S. MORK, Assr. to LUSTRON Co. INC. (U.S.P. 1,607,474, 16.11.26. Appl., 12.3.21).—Cellulose acetate, and the artificial silk prepared from it, are rendered more stable to the action of heat, carbonisation being inhibited, by incorporation, before or during the manufacturing process, of 0.5–2% of the potassium or sodium salt of oleic, salicylic, or other organic acid.

T. S. WHEELER.

**Paper making.** J. J. GANNON, L. W. MAHLE, and F. B. WELLS, Assrs. to THE OHIO BOXBOARD Co. (U.S.P. 1,606,162, 9.11.26. Appl., 6.4.25).—A layer of filler material is superimposed upon another of pergameneous material, the surplus water being afterwards expressed from the combined layers in a direction away from the pergameneous material and through the filler.

B. P. RIDGE.

**Making waterproof products.** L. KIRSCHBRAUN (U.S.P. 1,606,427, 9.11.26. Appl., 16.1.25).—Waste fibrous products, containing a bituminous waterproofing binder with a dispersing agent, are beaten up with an aqueous vehicle at a temperature near to the melting point of the binder, thus causing separation of the fibres. The bituminous material is precipitated on the fibres to render them non-sticky, and the stock is felted on a paper machine.

B. P. RIDGE.

**Process of paper making.** C. ELLIS, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,607,517, 16.11.26. Appl., 8.4.26).—A hot aqueous wax dispersion is added to a hydrocellulose pulp.

H. ROYAL-DAWSON.

**Treating paper pulp.** C. ELLIS, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,607,518, 16.11.26. Appl., 12.4.26).—The hydrogen-ion concentration of the pulp is adjusted so as to increase the absorption of a wax emulsion.

H. ROYAL-DAWSON.

**Paper product.** C. ELLIS, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,607,519, 16.11.26. Appl., 21.4.26).—The product consists of a rosin-sized paper of hydrocellulose containing wax in excess of rosin.

H. ROYAL-DAWSON.

**Production of transparent and waterproof paper.** A. E. MAZE, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,607,552, 16.11.26. Appl., 30.12.25).—An emulsion of paraffin wax is incorporated in the fibre resulting when moist paper pulp is subjected to a beating operation.

H. ROYAL-DAWSON.

**Manufacture of products containing cellulose.** C. DREYFUS (E.P. 262,157, 6.6.25).—See U.S.P. 1,546,211; B., 1925, 707.

**Hank yarn dryers.** H. HAAS (E.P. 261,686, 25.9.26. Conv., 9.3.26).

**Method of cocoon-storage by cooling and drying.** K. KOBORI, and KANEGAFUCHI BOSEKI KABUSHIKI KWAISHA (E.P. 261,093, 13.8.25).

**[Apparatus for] manufacture of lustrous fabrics.** F. WOLF, Assee. of H. KUHN (E.P. 259,940, 28.9.26. Conv., 13.10.25).

**Spindles of spinning machines for artificial silk and the like.** HARBENS (VISCOSE SILK MANUFACTURERS) LTD., W. E. SHARPLES, and GENERAL ELECTRIC Co., LTD. (E.P. 261,499, 16.9.25).

**[Cellulose] lacquer for shoe heels etc.** (U.S.P. 1,607,516).—See XIII.

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

### PATENTS.

**Discharge effects on [textile] materials containing cellulose acetate.** BRIT. DYESTUFFS CORP., LTD., and L. SMITH (E.P. 262,254, 25.11.25).—Clear white and coloured discharge effects on cellulose acetate silk dyed with those dyes commonly used for this type of silk are obtained in the usual manner by means of reducing agents containing sodium formaldehyde-sulphoxylate, provided that thiocyanates such as calcium, barium, and ammonium thiocyanate are present. A satisfactory discharge paste contains 15 g. of formosul (sodium formaldehyde-sulphoxylate), 70 g. of 3% gum tragacanth solution, and 10 g. of a solution of calcium thiocyanate (d 1.44).

A. J. HALL.

**Treatment of ["immunised"] cotton fibres, preparatory to dyeing.** P. KARRER (E.P. 249,842, 11.3.26).—Cotton partially esterified with the sulphonic acids of benzene, toluene, or naphthalene (cf. E.P. 195,619; B., 1924, 128), and heated with ammonia, aliphatic or aromatic primary, secondary, or tertiary amines, or with hydrazine or hydrazine derivatives, in suitable solvents, acquires an affinity for acid dyes such as tartrazine, Orange II, and sulphorhodamine; e.g., 10 pts. of cotton partially esterified by means of *p*-toluenesulphochloride are heated for 6 hrs. with 50 pts. of a 25% aqueous solution of ammonia in an autoclave at 100°.

A. J. HALL.

**Dyeing process [for wool].** BADISCHE ANILIN- u. SODA-FABR. (E.P. 242,233, 8.8.25. Conv., 30.10.24).—Sulphonic acids of propylated or butylated aromatic hydrocarbons, particularly naphthalene derivatives, are added to dye liquors for the purpose of assisting wetting-out of the textile (wool) material and level dyeing.

A. J. HALL.

**Decorative printing of textile fabrics.** CALICO PRINTERS' ASSOC., LTD., and A. SWALLOW (E.P. 261,448, 17.7.25).—Brocade effects are obtained by printing fabrics consisting of mixed fibres, e.g., cotton and wool, or cotton and a suitable artificial silk, with a multi-coloured floral pattern in fit with a blotch pattern, the spaces between the two patterns being of sufficient width to prevent creeping of the printing pastes used. The blotch pattern is produced by printing the fabric with a caustic alkali capable of destroying one of the fibres (wool or artificial silk) forming the fabric.

A. J. HALL.

**Manufacture of ice colours.** A. G. BLOXAM. From CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 261,542) 19.11.25).—In dyeing yarns and fabrics by means of naphthol AS dyes (cf. E.P. 230,022; B., 1925, 878,



the formation of loose colour (not fast to rubbing) on the surface of the fibres is avoided by displacing the unabsorbed "prepare" solution by treating the fabric before development with a much weaker "prepare" solution containing sodium chloride. For example, yarn prepared with a solution containing 7 g. of naphthol AS is treated with a similar solution containing only 0.5 g. of naphthol AS, but also 30 g. of sodium chloride per litre. In many cases this method enables the usual formaldehyde in the original "prepare" solution to be omitted.

A. J. HALL.

**Obtaining lustre and matt effects on fabrics composed wholly or in part of viscose in the sulphur state.** BURGESS, LEDWARD & Co., LTD., F. SCHOLEFIELD, and N. DENVER (B.P. 261,099, 14.8.25).—Lustre and matt effects are obtained by printing fabric containing non-desulphurised viscose silk with a paste containing sulphite of sodium, potassium, ammonium, or calcium, together with gum or glycerin or other suitable assistant, then steaming, rinsing, soaping, and drying; the printed parts are thereby desulphurised and rendered more lustrous. Sodium sulphide (cf. Scholefield, J. Soc. Dyers and Col., 1923, 39, 342) is not suitable for use as a desulphurising agent because of its deleterious action on the usual copper printing rollers. Suitable direct, basic, and vat dyes may be added to the desulphurising paste. A satisfactory coloured printing paste contains 1.5 lb. of benzopurpurine, 10 lb. of glycerin, 1.5 lb. of sodium phosphate, and 10 lb. of sodium sulphite.

A. J. HALL.

**Apparatus for mercerising fabrics.** L. MELLERSH-JACKSON. From MASCHINENFABR. BENNINGER A.-G. (E.P. 262,343, 24.6.26).

**Improving cellulosic materials, in particular vegetable textile fabrics** (E.P. 252,360).—See V.

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

**Absorption of water vapour by sulphuric acid solutions.** C. H. GREENEWALT (Ind. Eng. Chem., 1926, 18, 1291—1295).—It is found that in the absorption of water vapour by sulphuric acid in a wet-walled tower the resistance of the stagnant film of liquid at the gas-liquid interface is negligible (cf. Lewis and Whitman, B., 1925, 55). By altering the degree of turbulence by using various shapes of entrance nozzles for the gas or introducing packing into the tower, and by varying the velocity of the carrier gas, it has been shown that for counter-current flow both these variables are proportional to the resistance to flow through the tower, in so far as they affect the overall gas-film diffusion coefficient.

R. CUTHILL.

**Mass law in the manufacture of nitric acid.** C. W. CUNO (Ind. Eng. Chem., 1926, 18, 1298—1299).—If the partial decomposition of nitric acid when prepared from sodium nitrate is due in part to the reaction  $4\text{HNO}_3 = 2\text{H}_2\text{O} + \text{O}_2 + 2\text{N}_2\text{O}_4$  brought about by the dehydrating action of the sulphuric acid, introduction of an excess of oxygen or air into the retort might lessen the decomposition, and, in any case, the lower oxides of nitrogen would probably be in the most reactive state in the retort. Experiments show that the formation of

brown fumes in the distillation can be very nearly prevented in this way, the amount of oxygen required increasing with the temperature of distillation. Cf. U.S.P. 1,552,117 (B., 1925, 847).

R. CUTHILL.

**Lime-kiln operation in the ammonia-soda process.** J. KIRCHNER (Chem.-Ztg., 1926, 50, 957—958).—In the ammonia-soda cycle the production of a satisfactory kiln-gas is more important than the quality of the lime. 10% above the theoretical quantity of carbon dioxide is needed owing to incomplete absorption. Loss also occurs in the recovery of carbon dioxide from the sodium bicarbonate, so that for the production of 100 kg. of soda ash 90 kg. of carbon dioxide must be supplied, 60% of this from the lime-kilns. From a calculation of gas volumes it is shown that the raising of the kiln-gas from 30% to 40%  $\text{CO}_2$  saves 4 tons of coal per day, in a plant producing 100 tons of soda ash daily, in respect of economy in power in the gas compressors. A saving in ammonia also results. With a coke consumption of 9.5% in the kiln, a gas can in theory be produced containing 41.5% of  $\text{CO}_2$ . Increase in the coke ratio naturally produces a more dilute gas, but it is better to keep the gas concentrated, even if the limestone is incompletely burnt. The coke pieces should be of the size of the fist, and the limestone that of a child's head, as "smalls" produce irregularities in the gas flow, which should also be controlled by a fan. The kiln should be kept under pressure. The exit gases should not leave at a temperature above  $150^\circ$ , and the burnt lime should come out only hand-warm. Continuous charging is desirable. C. IRWIN.

**Ammonia catalysts.** A. T. LARSON and A. P. BROOKS (Ind. Eng. Chem., 1926, 18, 1305—1307).—The catalytic activities of iron, tungsten, molybdenum, nickel, and cobalt in the synthesis of ammonia from its elements at  $450^\circ$ ,  $500^\circ$ , and  $550^\circ$  and under a pressure of 30 atm. have been examined. Of these metals the last two are inactive, and iron is the most active, although its activity falls off very rapidly, especially at the higher temperatures. Mixtures of molybdenum with iron, cobalt, and nickel are slightly less effective. The addition of silica, thoria, alumina, zirconia, or ceria increases the activity of iron. Mixtures of oxides are most effective in this way when the constituent oxides are not of the same type; thus an iron catalyst containing 1% of potassium oxide and 3% of alumina is specially recommended.

R. CUTHILL.

**Pure iron and promoted iron catalysts for ammonia synthesis.** J. A. ALMQUIST and E. D. CRITTENDEN (Ind. Eng. Chem., 1926, 18, 1307—1309).—Iron is most active in catalysing the synthesis of ammonia if it has been prepared by reduction of ferrosferic oxide. If the original oxide contained an excess of ferrous oxide, there is a continuous decrease in activity with increase in the amount of this component. Potassium oxide appears to depress the activity of iron, unless some other refractory oxide such as silica is also present. Further evidence of the marked "promoting" effect of mixtures of potassium oxide with difficultly reducible amphoteric oxides such as silica and alumina has been obtained (cf. Larson and Brooks, preceding abstract; also B., 1922, 325 A).

R. CUTHILL.



**Simple apparatus for the removal of ammonium salts by volatilisation.** K. FISCHER (Z. angew. Chem., 1926, 39, 1574—1576).—An apparatus for volatilising ammonium salts from residues, such as are obtained in the analysis of the alkali metals, consists of a metal funnel from which the stem has been removed and which is lined inside with a clay tube in the shape of a truncated cone. The inside of the clay tube is covered with iron wire gauze, which also closes the hole at the lower end, at which point it is covered with a perforated metal plate. The basin containing the salts is placed inside the top of the gauze funnel, and is heated by means of a Bunsen burner, the flame of which is at least 12 cm. long when the gas is full on. The top of the burner is 1 cm. below the perforated plate, so that by the use of a small flame the solution may be evaporated to dryness without spirting, and subsequently the ammonium salts are volatilised by the use of the full flame of the burner.

A. R. POWELL.

**Distillation of calcium acetate.** E. G. R. ARDAGH, A. D. BARBOUR, G. E. MCCLELLAN, and E. W. MCBRIDE (Fac. Appl. Sci., Toronto, 1926, Bull. 6, 85—104).—Calcium acetate, made by adding a slight deficiency of calcium carbonate to acetic acid and evaporating to dryness, does not become completely anhydrous even after prolonged heating at temperatures below 160°. After 12 hrs. at 160° the salt still contains 2% of free acetic acid; at higher temperatures decomposition takes place and traces of acetone begin to be evolved, but a temperature above 400° is necessary for the rapid production of acetone. In order to obtain a high yield of this compound the vapours in the retort must be removed as rapidly as they are formed, preferably by means of a current of nitrogen or carbon dioxide; air should not be used as it reduces the yield considerably. A nearly theoretical yield is obtained at 450—490° in a good current of gas, and the presence of iron or copper has no deleterious effect; at 430° the maximum yield is 93%, and at 420° only 85%.

A. R. POWELL.

**Behaviour of difficultly soluble metal oxides in solutions of their salts. Magnesium oxide.** I. W. FEITKNECHT (Helv. Chim. Acta, 1926, 9, 1018—1049).—The behaviour of magnesium oxide in solutions of magnesium chloride, bromide, sulphate, and nitrate has been studied in detail, with special reference to the effects of concentration, temperature, method of preparation of the oxide, and other influences on the velocity of the formation of the basic salt, and the composition and crystalline form of the product, the examination being carried out by viscosimetric and volume-change measurements, and microscopical examination of the precipitates formed. Four types of magnesium oxide were employed, these being prepared, respectively, from magnesium carbonate at 600° (a) and 1100° (b), and from magnesium oxalate at the same temperatures (c and d). The main results obtained are as follows: the solubility of magnesium oxide in magnesium chloride solutions increases with rise of temperature and with increase in the concentration of the salt solution, and in supersaturated solutions the velocity of formation of the basic chloride is more rapid the higher the temperature and the greater the degree of supersaturation, and for each temperature

there exists a definite concentration below which, instead of the basic chloride, magnesium hydroxide separates, and this concentration increases with rise in temperature. In solutions of magnesium chloride containing an excess of magnesium oxide in suspension the behaviour of the mixture depends very largely upon the concentration of the chloride solution and the sample of oxide used. In general, in solutions of sufficient concentrations for any change to occur, the basic salt separates first in an amorphous form, which then slowly changes into a crystalline form, the velocity and duration of the reaction depending on the factors mentioned. In dilute solutions, i.e., in concentrations below which any change in the form of the basic salt occurs, the reaction starts sooner, and there is originally a large swelling, accompanied by an increase in the viscosity of the medium to a maximum value, which then disappears, the viscosity falling again. The amount of this swelling is greater with sample (a), which is very disperse, than with the more compact sample (c). Heating the oxide to 1100° causes a large diminution in the surface area, and the reaction velocity is thereby decreased, the adsorption complex being slowly formed and going slowly into solution, so that the basic salt separates slowly in large crystalline form. In more concentrated solutions the viscosity increases more slowly to a greater maximum value, and in higher concentrations (7—8N) of the chloride the whole reaction mixture becomes stiff. The value of the concentration below which no change in form occurs is usually considerably higher than the concentration at which the basic chloride separates from supersaturated solutions, the two being more nearly identical with samples (b) and (d), and it increases rapidly with rising temperature. The ratio of oxide to chloride directly affects mainly the rate at which the reaction sets in. Magnesium hydroxide reacts much more slowly than the oxide, the velocity still being immeasurably small in relatively concentrated solutions. The basic salt formed was separated and analysed in each case; its crystalline form and chemical composition were found to depend upon the sample of oxide used, the concentration of the solution, and the temperature. The proportion of magnesium oxide was found, in general, to be greater the more dilute the solution, the value of the ratio  $\text{MgCl}_2/\text{MgO}$  in the product varying approximately from 1/2.5 to 1/5. The water content of the product, dried over phosphorus pentoxide, corresponds to 10 mols. for each mol. of magnesium chloride. The product obtained from 3.5N- and 4N-solutions agreed with the composition  $\text{MgCl}_2 \cdot 5\text{MgO} \cdot 10\text{H}_2\text{O}$ ; that from 3N-solutions with  $\text{MgCl}_2 \cdot 4\text{MgO} \cdot 10\text{H}_2\text{O}$ ; that from 5N-solutions, and when magnesium hydroxide was used in place of the oxide, with  $\text{MgCl}_2 \cdot 3\text{MgO} \cdot 10\text{H}_2\text{O}$ ; and that from 6N- and 8N-solutions with  $2\text{MgCl}_2 \cdot 5\text{MgO} \cdot 16\text{H}_2\text{O}$ , but long contact with the mother-liquors alters both the crystalline form and the composition of the product. Similar results were obtained in the other magnesium salt solutions.

J. W. BAKER.

**Fixation of nitrogen as aluminium nitride.** H. J. KRASE, J. G. THOMPSON, and J. Y. YEE (Ind. Eng. Chem., 1926, 18, 1287—1290).—Preliminary experiments in which mixtures of bauxite, coke, and magnetite



were heated in an electric furnace showed that, although reduction was incomplete, alloys containing 40% or more of aluminium were formed. If a ferroaluminium containing 50% Al is heated at 1400° in nitrogen under atmospheric pressure, about 60% of the aluminium is converted into nitride. The presence of small amounts of magnesium promotes nitrification, whilst calcium, silicon, and titanium have the opposite effect. If small quantities of cryolite, or of the chlorides or fluorides of aluminium, magnesium, sodium, or calcium are present, nitrification may be rapid and practically complete even at 1200°. With ferrotitanium or aluminium carbide at 1400° the absorption of nitrogen is only about 20% of the theoretical, whilst for ferrosilicon the figure is only 2%.

R. CUTHILL.

**Variations in density of the atmosphere.** A STOCK and G. RITTER (*Z. angew. Chem.*, 1926, 39, 1463—1464).—This preliminary communication shows that the common belief in the constancy of the density and composition of air free from moisture and carbon dioxide is in error. With the aid of a special air-density balance (A., 1926, 669) it is demonstrated that the variations in density may be as much as 0.13% even in rural surroundings. This corresponds to a variation of 0.75% of oxygen, a matter which it is proposed to examine further.

E. HOLMES.

## PATENTS.

**Manufacture of [concentrated] oleum.** J. H. SHAPLEIGH, Assr. to HERCULES POWDER CO. (U.S.P. 1,605,004, 2.11.26. Appl., 3.2.25).—Gases from a converter containing about 7% SO<sub>2</sub> are cooled, and passed into 20% oleum, to which 98% sulphuric acid is added to balance the sulphur trioxide absorbed. The gases are then led into a second absorber, where they are treated with water to remove the residual sulphur trioxide with formation of 98% sulphuric acid, which is employed in the first absorber. The heat evolved in the formation of the 98% acid is used to distil pure sulphur trioxide from a portion of the 20% oleum, which is then returned to the first absorber, and the evolved gas is passed into a further quantity of 20% oleum to form concentrated oleum of any desired strength.

T. S. WHEELER.

**Manufacture of sulphuric acid.** E. R. WOLCOTT, Assr. to THE TEXAS CO. (U.S.P. 1,605,088. 2.11.26. Appl., 4.12.24).—Sulphur dioxide and oxygen are passed over heated diaspore to produce sulphur trioxide.

H. ROYAL-DAWSON.

**Absorbing sulphur trioxide from gases containing same.** J. H. SHAPLEIGH, Assr. to HERCULES POWDER CO. (U.S.P. 1,608,006, 23.11.26. Appl., 22.4.25).—Sulphuric acid is sprayed on the contact gases, counter-current to the gas flow and at a number of points along the path of the gases passing through a horizontal cylinder. By this means a progressive reduction in the sulphur trioxide vapour pressure is produced at certain points of absorption, and acid of progressively decreasing strength is continuously produced. The separate products are collected within the cylinder.

H. ROYAL-DAWSON.

**Manufacture of silicic acid gel.** W. CARPMAEL. From I.-G. FARBENIND. A.-G. (E.P. 262,306, 19.2.26).—Silicic acid jelly is washed, or dehydrated by pressure,

prior to treatment with sodium carbonate solution, or with ammonia, water glass, and the like, the product being subsequently dried.

H. ROYAL-DAWSON.

**Production of moulded silicic acid gel.** M. PRÄTORIUS and K. WOLF (E.P. 249,555, 19.3.26. Conv., 21.3.25).—A silicic acid jelly containing 75—95% of water is mixed with 1—2% of a colloidal silicic acid solution of 1—10% strength, the homogeneous mass being then moulded and dried at 110—180°. A modification of the process is obtained by using the silicic acid jelly itself and gradually heating it under pressure at 100—300°, the mass being then moulded and dried as before.

H. ROYAL-DAWSON.

**Manufacture of hydrocyanic acid.** J. Y. JOHNSON. From I.-G. FARBENIND. A.-G. (E.P. 261,559, 14.12.25).—Formamide, alone or mixed with ammonium formate, is rapidly heated between 400° and 900° in a quartz tube in the absence of other gases. The reaction may be carried out in the presence of various dehydrating catalysts, e.g., alumina, bauxite, titanium oxide, silica gel, etc., supported with pumice, asbestos, or active charcoal, the resulting hydrocyanic acid being rapidly cooled.

H. ROYAL-DAWSON.

**Production of hydrocyanic acid.** B. S. LACY, Assr. to THE ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,608,700, 30.11.26. Appl., 20.2.26).—In the thermal decomposition of formamide in the presence of a catalyst capable of causing the formation of hydrocyanic acid and water, the formamide vapour is mixed with a large volume of an inert heating gas at above 500° before it comes in contact with the catalyst.

H. ROYAL-DAWSON.

**Process for oxidising a ferrous salt.** R. F. GRANT and E. WETHERBEE, Assrs. to H. M. HANNA (U.S.P. 1,606,470, 9.11.26. Appl., 9.3.25).—A subdivided catalyst (manganese dioxide) is introduced into a ferrous sulphate solution, which is circulated in contact with it in the presence of air.

H. ROYAL-DAWSON.

**Producing pure aluminium hydroxide, particularly from aluminium-containing raw materials rich in silica.** E. L. RINMAN (E.P. 243,356, 18.11.25; Conv., 22.11.24).—The sulphate solution, after first treating the raw material with sulphuric acid, is mixed with a hot solution of sodium hydrosulphide containing sodium sulphide to precipitate the aluminium hydroxide and liberate hydrogen sulphide. The aluminium hydroxide obtained is dissolved in sodium sulphide solution, precipitated with hydrogen sulphide, filtered, and washed.

H. ROYAL-DAWSON.

**Method of producing pure aluminium hydroxide.** G. H. HULTMAN (U.S.P. 1,607,279, 16.11.26. Appl., 8.5.25. Conv., 25.1.24).—Aluminium mineral is heated with sulphuric acid producing a solution of the sulphate in which the iron present is reduced to the ferrous state; on addition of calcium carbonate to the solution, the ferrous salt, undissolved aluminium hydroxide, and calcium sulphate are separated and washed with water to dissolve out the calcium sulphate.

H. ROYAL-DAWSON.

**Manufacture of chromates.** W. CARPMAEL. From I.-G. FARBENIND. A.-G. (E.P. 261,647, 25.6.26).—A mixture containing ground chrome iron ore, sodium car-



bonate, ferric oxide, and a small quantity of magnesia, is introduced in the wet or dry state into a mechanical furnace and heated above 1000°, after which the melt is quenched in hot water to dissolve out the aluminium salts. The residue is dried, ground, and again brought to the desired composition with ferric oxide or magnesia and re-used with fresh material. The alumina is precipitated from the chromate liquor by waste gases or acids.

H. ROYAL-DAWSON.

**Separating aluminium nitrate from mixed solutions of potassium, sodium, iron, calcium, and magnesium nitrates.** S.I.P. Soc. ITALIANA POTASSA (E.P. 246,827, 18.1.26. Conv., 28.1.25).—Nitric acid is added to the unevaporated mixed solution of these salts in such a manner as to produce the crystallisation of aluminium nitrate, which is complete according to the degree of acidity, while the remaining nitrates remain in solution.

H. ROYAL-DAWSON.

**Process for producing phosphorus pentoxide.** C. E. PARSONS and S. PEACOCK, ASSRS. to METAL RESEARCH CORP. (U.S.P. 1,606,319, 9.11.26. Appl., 29.3.24).—The blast furnace is charged with material containing calcium phosphate, silica, coke, and an alkali chloride.

H. ROYAL-DAWSON.

**Process and apparatus for making sodium chloride.** H. T. REID (U.S.P. 1,605,375, 2.11.26. Appl., 19.2.24).—The top surface of the salt solution is heated uniformly with hot air without causing ebullition or disturbance of the solution, which is maintained at a fixed level.

H. ROYAL-DAWSON.

**Production of potassium salts [from sea water].** E. NICCOLI (E.P. 261,991, 13.5.26; cf. F.P. 594,904 and E.P. 247,405; B., 1926, 237, 360).—The process described in F.P. 594,904 is modified so that instead of dissolving the mixture of sodium, potassium, and magnesium salts in water to form a saturated solution, the salts are treated with a quantity of water insufficient to give complete solution, whereby the sodium salts and chlorides are dissolved, leaving the double salt of potassium and magnesium sulphate as a residue.

W. G. CAREY.

**Obtaining sulphur from alkaline-earth sulphates.** SALZWERK HEILBRONN A.-G., T. LICHTENBERGER, and K. FLOR (E.P. 251,942, 29.1.26).—Carbon in the form of coke is added to alkaline-earth sulphate dissolved in molten sodium chloride, and, after reduction, the alkali chloride is separated from the alkaline-earth sulphide by sedimentation, the latter being converted, by treatment with vapour or acid, into the oxide or salt and hydrogen sulphide, from which sulphur is obtained in the usual manner.

H. ROYAL-DAWSON.

**Purifying and concentrating sulphur dioxide.** G. C. HOWARD (U.S.P. 1,606,299, 9.11.26. Appl., 3.4.23).—In the absence of water and phenols, a liquid cyclic hydrocarbon forms compounds with sulphur dioxide, which can thus be removed from a dry gas containing it. The hydrocarbon and the sulphur dioxide are separated and recovered by heating.

R. A. A. TAYLOR.

**Manufacture of carbon disulphide.** A. J. STEPHENS. From ZAHN & Co. BAU CHEM. FABR. G.M.B.H. (E.P. 261,990, 12.5.26).—Raw sulphur is freed from impurities

in a melting apparatus comprising two parts, so that impurities can be removed by a siphon device at one side, while the other part is in use. The fluid sulphur is led to a retort constructed so that the lower part is broadened and ribbed to increase the heating surface; the retort contains wood charcoal, and the mixture is heated from the outside. The crude carbon disulphide is distilled, and on cooling much of the hydrogen sulphide present as an impurity escapes and is led to a specially constructed Claus furnace for conversion into sulphur for use again in the process. The last traces of hydrogen sulphide are removed by spraying the carbon disulphide into a separator containing pure, nearly boiling carbon disulphide, the escaping hydrogen sulphide being freed from carbon disulphide vapour in the cooler and conveyed to the Claus furnace, while the carbon disulphide is drawn off from the separator by a siphon, and is cooled and stored. The residue in the distilling vessel, consisting of carbon disulphide rich in dissolved sulphur, is drawn off periodically into a funnel-shaped container with ribbed walls, strongly heated on all sides, in which the carbon disulphide evaporates and is sent to the cooling system, after which the sulphur melts and is run back through a heated siphon to the retort. W. G. CAREY.

**Separation of hydrocyanic acid from gaseous mixtures containing it.** O. LIEBKNECHT, ASSR. to DEUTSCHE GOLD- & SILBER-SCHNEIDANSTALT VORM. ROESSLER (U.S.P. 1,605,897, 2.11.26. Appl., 9.8.24. Conv., 1.8.23).—See G.P. 403,378; B., 1925, 205.

**Electrolyser for the production of hydrogen and oxygen** (E.P. 242,635).—See XI.

**Preparation of titanium compounds [for pigments]** (E.P. 261,051).—See XIII.

**Producing a titanium pigment [titanium oxide]** (U.S.P. 1,605,851).—See XIII.

## VIII.—GLASS; CERAMICS.

**Lubricants for ground-glass joints.** M. J. BRADLEY and H. E. WILSON (Ind. Eng. Chem., 1926, 18, 1279—1280).—A number of lubricating mixtures are described, including one which is very resistant to the action of strong reagents. Heavy liquid paraffin oil is superior to vaseline as a base.

R. CUTHILL.

**Effect of porosity upon thermal conductivity, diffusibility, and heat capacity at high temperatures.** Y. TADOKORO (Sci. Rep. Tôhoku Imp. Univ., 1926, 15, 567—596).—Refractory materials were prepared from a mixture of 2 pts. (by vol.) of alundum powder (80 mesh/in.) and 1 pt. of washed "Gairome" fireclay, and the porosity was varied by adding increasing amounts of sawdust. The properties of these materials were then examined, and also of specimens made from diatomaceous earth. The porosity and gas permeability vary similarly. The permanent changes in weight and length caused by heating at temperatures up to 1300° were measured; if diatomaceous earth is heated to 1100° before use, it can subsequently be heated at high temperatures without fear of cracking from volume change. Both thermal expansion and crushing strength decrease with increasing porosity, the curve connecting these constants resembling a rectangular hyperbola.



If  $K$  is the thermal conductivity,  $\rho$  the sp. gr., and  $C$  the sp. heat, the diffusibility  $h^2$  is given by  $h^2 = K/\rho C$ . The effects of porosity on diffusibility, thermal conductivity, and heat capacity have been determined; in all cases a minimum value is given by a porosity of 37–38%. The spalling tendency is expressed by the relation  $(\alpha \times \text{crushing strength})/h$ , where  $\alpha$  is the coefficient of expansion; here, again, a porosity of 37–38% is a critical point which is of importance in the manufacture of fire-bricks. Diatomaceous earth is a very good insulating material, but its crushing strength is low.

W. HUME-ROTHERY.

**Modulus of rupture of dried clays as a measure of plasticity.** H. KOHL (Ber. Deuts. Keram. Ges., 1926, 7, 19–31).—Previous investigations have indicated a certain relation between the mechanical strength and the bonding strength of clays. The modulus of rupture test, in preference to other mechanical tests, was chosen to give an indication of plasticity because of its simplicity in operation, and because the results give a more uniform and reliable indication of the chief factor in plasticity, viz., the strength of the bond in clays. The tests were carried out on air-dried specimens of clays and body mixtures. Blue clays gave the highest results—47 kg./sq. cm.—and fine kaolins the lowest—2.8 kg./sq. cm. For practical purposes, the minimum strengths, in kg./sq. cm., are: for earthenware, 7–8; for porcelain, 4–5; and for saggars, 5–7. The addition of alkali hydroxides to casting slips almost doubled the dry strength as compared with specimens moulded from the same body. Cast samples were also more dense than moulded. The results of the modulus of rupture tests were characteristic of the different mixtures tested, and agreed reasonably well with the working properties as observed in actual practice.

F. SALT.

**Effect of industrial usage on thermal conductivity of semi-silica material used in a coke-oven wall.** A. T. GREEN (Trans. Ceram. Soc., 1925–6, 25, 258–261).—See B., 1926, 947.

#### PATENTS.

**Manufacture of glass and articles therefrom.** E. A. HAILWOOD (E.P. 261,491, 1.9.25).—Molten glass of high fluidity, e.g., a glass prepared from 150 pts. of sand, 84 pts. of sodium carbonate, 42 pts. of borax, 6 pts. of magnesia, 6 pts. of zinc oxide, and 12 pts. of calcium carbonate spar, is cast in sand, plaster, or metal moulds, and annealed either in the moulds or after removal therefrom.

B. W. CLARKE.

**Method and apparatus for purifying quartz.** BRITISH THOMSON-HOUSTON CO., LTD., Assees. of E. THOMSON (E.P. 252,747, 1.6.26. Conv., 1.6.25).—Silica is fused at 1650–1700° in an electrically-heated crucible rotating at 1500 r.p.m., so that the lighter portion of the melt containing entrapped gases is segregated from the remainder, which upon solidifying forms a homogeneous mass of clear quartz.

B. W. CLARKE.

**Pottery and other kilns.** MORGAN CRUCIBLE CO., and C. W. SPEIRS (E.P. 261,866, 9.9.25 and 18.12.25).—In a tunnel kiln the firing zone is heated by one or more electrical resistors (E.P. 225,608), forming either the roof alone, the roof and sides, or placed centrally in the tunnel.

Flues may be provided for burning oil, gas, or waste gases, so as to minimise radiation of electrically-developed heat. Other modifications are (1) an endless circular tunnel, (2) a sloping tunnel with a raised firing zone, (3) suitable heating of other zones of the kiln by resistors, (4) regulation to give an oxidising or reducing atmosphere as required.

A. COUSEN.

**Manufacture of grinding or abrading stones.** E. MENZIONE and L. MAYER (E.P. 261,942, 19.1.26).—The stones are made from a paste consisting of burnt magnesite, graphite, minium, steel filings, and corundum or carborundum, together with hydrochloric acid. The paste, reinforced with wire netting, is moulded and dried by pressure. It is finally treated with a solution of magnesium fluosilicate at about 80° and then dried.

A. COUSEN.

**Neutral refractory cement.** W. F. ROCHOW, Assr. to HARBISON-WALKER REFRACTORIES CO. (U.S.P. 1,606,481, 9.11.26. Appl., 20.1.23).—A refractory composition consists of an intimate mixture of powdered aluminium oxide and powdered sodium silicate.

B. W. CLARKE.

**Dental cements.** S. S. WHITE DENTAL MANUFACTURING CO., Assees. of N. E. EBERLY (E.P. 248,698, 11.8.25. Conv., 3.3.25).—A dental cement consists of from 70 to 98% of a basic glass, prepared by fusing together silica and alumina in the presence of a suitable flux, e.g., calcium fluoride, which is powdered and mixed with a powder containing basic oxides, e.g., zinc oxide and silica, bismuth oxide, etc. Aqueous phosphoric acid containing phosphates of zinc, aluminium, etc. in solution is added to give the required consistency.

B. W. CLARKE.

**Manufacture of sheet glass.** E. C. R. MARKS. From ERIE GLASS CO. (E.P. 261,150, 28.9.25).

**Apparatus for forming sheet glass.** L. MELLERSH-JACKSON. From HARTFORD-EMPIRE CO. (E.P. 261,627, 31.5.26).

**Means for making gas-tight joints between metal and glass or quartz** (E.P. 243,000).—See X.

**Sealing metal to glass** (E.P. 251,951).—See X.

**Sealing molybdenum or similar refractory metals and fused quartz** (U.S.P. 1,608,612).—See X.

## IX.—BUILDING MATERIALS.

**Lime-kiln operation in the ammonia-soda process.** KIRCHNER.—See VII.

#### PATENTS.

**Manufacture of cement.** T. RIGBY (E.P. 261,814, 24 and 25.5.26, and 12.8.25).—In the manufacture of cement by the wet method in a rotary kiln, a higher kiln output is attained by spraying the slurry into the kiln, thus reducing its water content to about 10% before it is deposited on the wall, and preventing the formation of "slurry rings" or large masses. One or more sets of long sprays are employed, originating outside the kiln, and disposed at such an angle to the axis that even if projected several feet into the kiln the spread is not greater than the internal diameter of the kiln, so that the still moist slurry is gently deposited upon the wall. Slurry lifting or agitating gear is provided



internally only beyond the region where the atomised material is mainly deposited, and a scraping device is added which, during rotation, frees the kiln mouth from deposit.

W. G. CAREY.

**Calcining and clinkering cement-forming materials.** R. D. PIKE (U.S.P. 1,605,279, 2.11.26. Appl., 13.6.23. Cf. U.S.P. 1,468,168; B., 1923, 1073).—The material to be treated is passed through a calcining chamber and a clinkering chamber, and the hot material is then agitated in a cooling chamber, through which is passed substantially the whole of the air needed to support combustion in the other two chambers, in order to absorb the sensible heat from the hot clinker.

B. W. CLARKE.

**Treating cement mix and other materials.** J. W. HORNSEY, ASSR. to GRANULAR IRON CO. (U.S.P. 1,606,125, 9.11.26. Appl., 9.7.19. Renewed 26.1.25).—Cement mix is calcined in a rotary kiln supplied with fuel in fixed proportions, and arranged so that the egress of gases from the furnace end is prevented, and the total air supply at the furnace end relative to the draught outlets at the stack end is restricted until the oxygen supply is practically limited to the theoretical requirement of the fuel used.

B. W. CLARKE.

**Utilising heat from cement clinker.** W. S. SPEED, ASSR. to LOUISVILLE CEMENT CO. (U.S.P. 1,606,850, 16.11.26. Appl., 23.3.20).—Clinker from rotary or other types of kilns is fed into a closed chamber, and air is passed backwards and forwards through the clinker the air then being used as a heating medium.

W. G. CAREY.

**Ageing or seasoning cement.** J. W. FULLER (E.P. 250,599, 8.4.26. Conv., 9.4.25).—Freshly-ground cement is discharged into a cement pump consisting of a cylindrical barrel in which a differential screw rotates at high speed, and moist gases, having a high carbon dioxide content from the products of combustion in the cement kiln, are injected through orifices at high pressure into the cement. The moist gas, in intimate mixture with the cement, converts any free lime into carbonate and hydroxide, and also causes the cement to flow more easily. Cement so treated can be used almost immediately after grinding.

W. G. CAREY.

**Process for ageing or seasoning cement.** J. W. FULLER (U.S.P. 1,608,499, 30.11.26. Appl., 9.4.25).—See E.P. 250,599; preceding.

**Preservation of stone.** A. P. LAURIE (U.S.P. 1,607,762, 23.11.26. Appl., 31.10.25. Conv., 29.8.23).—See E.P. 221,342; B., 1924, 911.

**Concrete mixing apparatus.** RANSOMES & RAPIER, LTD., and P. D. IONIDES (E.P. 261,910, 23.11.25).

**Grinding members for ball, drum, and tubular mills [for Portland cement]** (E.P. 261,664).—See I.

**Process and apparatus for electrodeposition [for "copperclad shingles"]** (E.P. 238,230).—See XI.

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

**Dendritic crystallisation and grain formation in steels.** V. N. KRIVOBOK (Trans. Amer. Soc. Steel

Treat., 1926, 10, 758—781).—The characteristics of dendrites are considered. Photomicrographs are given of the structure of dendritic skeletons from low- and high-carbon steels which show that on cooling a single dendrite is changed into a number of grains. The causes of granulation may include changes in structure accompanying change in atomic arrangement at the A3 point, conditions of strain existing in the ingot after solidification, and the presence of mechanically-held inclusions. The grain size in castings does not depend on the size of the original dendrites. Large dendrites accompany slow cooling, which favours the formation of inclusions in patterns and pronounced segregation. The formation of banded structure in hot-rolled blooms is due to the same cause.

T. H. BURNHAM.

**Chromium-cobalt steels.** F. M. OSTRAGA (Compt. rend., 1926, 183, 882—885).—The effect of a second heating, after a primary heating to temperatures between 900° and 1100°, has been studied for two chromium-cobalt steels (composition given), both dilatometrically and micrographically. It is concluded that they are at least as sensitive as, if not more so than, high-speed chromium-nickel steels to the temperature of heating and the rate of cooling. Indications are obtained that an austenite-troostite transformation takes place in the steels after heating them to a point just above their transition temperature, and slowly cooling.

S. J. GREGG.

**Forging by the upset process.** J. C. KIELMAN (Trans. Amer. Soc. Steel Treat., 1926, 10, 599—614).—Ball-bearing rings are forged by upsetting bars of steel containing 0.95—1.10% C and 1.20—1.50% Cr, using tungsten steel dies. The pierced-out centre is left on the end of the bar and used in the next forging. The forging temperature is approximately 1100°. The flow of metal in the forgings is shown by an etching process. The wear of the dies does not seem to depend on their ball hardness, and the temperature had a greater effect than the pressure. The oil-fired furnaces for heating the bars are described.

T. H. BURNHAM.

**Behaviour of industrial copper during cold working.** E. SEIDL and E. SCHIEBOLD (Z. Metallk., 1926, 18, 241—246, 315—321, 343—346).—When ordinary commercial copper is cast into ingots in open moulds crystallisation takes place in such a way that the cross-section of the ingot may be divided into several well-marked zones according to the crystal structure; the outer surface layer immediately against the walls of the mould consists of a heterogeneous mixture of very small crystals with a large proportion of the impurities, especially cuprous oxide, next there is a broad layer of large fibrous crystals arranged perpendicular to the walls of the mould, and, finally, the interior is built up of three distinct zones, the lower consisting of small "seed" crystals, the middle of larger, more regular crystals, and the upper of somewhat smaller, unevenly oriented crystals forming generally a somewhat porous mass owing to inclusions of gas. Pores may also be discerned along the boundaries of the fibrous crystals, between the fibrous layer and the central zones, and, to a much smaller extent, in the mass of large crystals in the centre of the ingot. Besides exhibiting differences of macro-



structure the zones also show characteristic X-ray diffraction patterns, and the hardness and mechanical properties vary considerably, the fibrous zones being generally the toughest and the surface skin the hardest. During cold rolling the surface layers usually develop fissures which on severe rolling tend to penetrate the middle layers; after removal of the surface skin, however, the remainder of the ingot may be rolled without danger of fracture, although sometimes the central zone will develop cracks after a 60% reduction in thickness. The presence of cuprous oxide or gas inclusions between the fibrous crystals will also sometimes cause splitting when they are rolled along the longest diagonal. Prolonged annealing of the cast bars at 750° results in a more homogeneous structure, but causes the separation of relatively coarse particles of cuprous oxide. The only satisfactory procedure for eliminating troubles in rolling consists of ensuring more homogeneous castings by avoiding oxidation of the metal as much as possible during casting and by pouring quickly in a steady stream at the correct casting temperature into thick-walled moulds. A uniformly fine structure may then be obtained by forging the ingot to reduce its cross-section by 5–10% and subsequently annealing at 750° for 1 hr. previous to rolling.

A. R. POWELL.

**Equilibrium between zinc vapour, carbon monoxide, and carbon dioxide.** E. THEIS (Z. angew. Chem., 1926, 39, 1568–1574).—The equilibrium of the reaction  $\text{ZnO} + \text{CO} = \text{Zn} + \text{CO}_2$  is given by the equation  $\log(p_{\text{Zn}} \cdot p_{\text{CO}_2}/p_{\text{CO}}) = -10,100/T + 9.62$  where  $p_{\text{Zn}}$ ,  $p_{\text{CO}}$ , and  $p_{\text{CO}_2}$  are the partial pressures of zinc vapour, carbon monoxide, and carbon dioxide, respectively, at a temperature of  $T^\circ$  Abs. The total vapour pressure and the partial pressures of zinc vapour, carbon monoxide, and carbon dioxide over a heated mixture of graphite and zinc oxide at 660–880° have been calculated from this equation; the results agree fairly well with those of Maier and Ralston (A., 1926, 358) and indicate that in the presence of liquid zinc the total vapour pressure reaches 1 atm. at above 870°.

A. R. POWELL.

**Structure and properties of red brass.** R. KÜHNEL (Z. Metallk., 1926, 18, 273–278, 306–311).—Red brass alloys containing 80–85% Cu, 4–9% Sn, and 9–4% Zn, with or without the addition of 2–3% Pb, are used as bearing metals on the German State Railways, the usual alloy containing 85% Cu, 9% Sn, and 6% Zn. Occasionally brittleness has developed, and this is shown to be due either to the presence of sulphur, as little as 0.02% of which causes the segregation of a sulphide constituent in the grain boundaries, or to inverse segregation. The latter is especially marked when about 2.5% Pb is added to the alloy and when dry sand moulds are used; in these cases the tin and lead contents of the outer shell of the casting may each be up to 0.5% above that of the core. The phenomena causing inverse segregation are discussed with reference to the work of other investigators, and experiments are described in which the physical and mechanical properties of many other alloys of the same type have been determined in an endeavour to find a cheaper alloy having similar properties to the above-mentioned alloy, but which has not such a tendency to inverse segregation. The most satisfactory alloy contained 82.8% Cu,

5.7% Sn, 9% Zn, and 2.3% Pb; it showed less wear with light loads than the 9% Sn alloy, but slightly more wear with heavy loads, otherwise its mechanical properties closely resembled those of the 9% Sn alloy.

A. R. POWELL.

**Structure of red brass [copper-tin-zinc alloys].** M. HANSEN (Z. Metallk., 1926, 18, 347–349).—From the results obtained by the author in earlier work, and from those obtained by numerous other investigators, the ternary diagram of the system copper-tin-zinc has been constructed for alloys containing more than 50% Cu, and, on this as a base with vertical temperature ordinates, a space model has been constructed to show the equilibrium diagram of these alloys for temperatures up to 1100°. With reference to this model the behaviour of certain alloys of the system during cooling is discussed.

A. R. POWELL.

**Sand-cast aluminium-magnesium silicide alloys.** S. DANIELS (Ind. Eng. Chem., 1926, 18, 1280–1285; cf. B., 1926, 494).—The properties of alloys of aluminium with up to 13.5% of magnesium silicide have been examined. Alloys containing 1.25–1.75% of the silicide manifest, when quenched and artificially aged, both strength and ductility in a high degree. Annealing causes reduction in both strength and hardness compared with the sand-cast material, but increase in ductility.

R. CUTHILL.

**Determination of metallic lead in metallurgical products and pigments.** D. H. MCINTOSH (Ind. Eng. Chem., 1926, 18, 1320–1321).—The material to be analysed is boiled with a 15% solution of sodium hydroxide, which dissolves most of the lead salts, and the solution is then filtered. All the metallic lead remains in the residue, and is dissolved out with a 10% solution of silver nitrate, from which it is reprecipitated with sulphuric acid. Finally the lead sulphate is dissolved in a hot saturated solution of ammonium acetate, and titrated with standard ammonium molybdate solution, using tannic acid as indicator.

R. CUTHILL.

**Structure of electro-deposited metals.** F. FOERSTER and J. FISCHER (Z. Elektrochem., 1926, 32, 525–534).—The dependence of the structure of electro-deposited tin on various factors, particularly on the nature and structure of the cathode material, has been examined. An electrolyte containing 0.25 mol. of stannous sulphate, 0.15 mol. of sulphuric acid, and 0.16 mol. of cresolsulphonic acid per litre (cf. Stack, B., 1924, 521), yields smooth, regular, thick deposits of tin at 0.25–0.5 amp./dm.<sup>2</sup> Considerably higher concentrations of cresolsulphonic acid cause very soft, dull grey deposits unless the sulphuric acid concentration is correspondingly raised. Cresolsulphonic acid prepared from pure *m*-cresol acts much better than that from commercial cresol. Sulphonic acids derived from phenol,  $\beta$ -naphthol, naphthalene, or  $\alpha$ -naphthylamine may also be used. Using a cathode of bright tin-plate in the electrolyte specified, the deposit has an appearance like that of tin-plate which has been etched with acid (“frost-flower” structure), the deposited tin atoms having arranged themselves so as to continue the lattices of the crystals forming the cathode surface (cf. Blum and Rawdon, B., 1923, 405; Graham, *ibid.*, 984). But at a cathode



of tin-plate roughened with emery or of tin foil, or at cathodes of other metals, tin is deposited under similar conditions in a fine-grained form, small crystals developing from many new nuclei. Whereas with deposits of the former kind, variations of current density, temperature, or electrolyte composition within wide limits produce little change in structure, with those of the latter type notable modifications may be produced similar to those observed in the deposition of copper or iron. On continued electrolysis, however, deposits of the "frost-flower" type eventually become overgrown with a fine-grained deposit; the higher the current density and the lower the tin content of the solution, the sooner this change sets in. The "frost-flower" structure of deposits on tin-plate can also be obtained from other tin baths capable of yielding smooth deposits, *e.g.*, from sulphostannate solutions or from a strongly acidified stannous sulphate solution containing 1% of gelatin. Addition of *m*-cresol itself to a strongly-acidified stannous sulphate bath restrains the formation of outgrowths, whilst the addition of 5–10 c.c. of *m*-cresol and 0.5 g. of gelatin to a litre of such a bath gives at 11° and 0.5 amp./dm.<sup>2</sup> an extremely fine-grained, silvery deposit suitable for plating purposes. Deposition of cadmium from faintly acid cadmium sulphate solutions on coarsely crystalline cadmium surfaces also results in the continued growth of the crystals forming the cathode surface. Cresolsulphonic acid has no effect in this case, but it suppresses the formation of outgrowths in the deposition of cadmium from ammoniacal cadmium solutions. The addition of cresolsulphonic acid to acid stannous sulphate solutions does not notably change the stannous-ion concentration, nor does it appear to alter the nature of the cathodic deposition process, for neither the equilibrium potential of tin in the solution nor the cathodic polarisation is much changed. Its possible behaviour as a colloid might account for the suppression of outgrowths, but not for its favourable influence on the formation of "frost-flower" structure, which only forms when colloids are reduced to a minimum. The anion of cresolsulphonic acid probably acts in a similar manner to that of the anions  $\text{ClO}_4^-$ ,  $\text{BF}_4^-$ , and  $\text{SiF}_6^{2-}$  in the deposition of lead, or of  $\text{SO}_4^{2-}$  in the deposition of silver, actions which are not explained by the theory of Blum and Rawdon (B., 1923, 1076). The observation that the polarisations at given current density in the deposition of the "frost-flower" and the fine-grained forms of tin are identical within the experimental error (1 millivolt) is, however, not necessarily in disagreement with that theory.

H. J. T. ELLINGHAM.

**Influence of prolonged heating on resilience of some exhaust-valve metals.** J. F. SAFFY (Compt. rend., 1926, 183, 861–863).—The effect of prolonged heating at temperatures below the transition point has been studied for four steels (composition given), the samples being heated for periods up to 12 weeks in a furnace kept at 600°; two of the steels were also treated at 850°. The resilience of the samples was then determined, and by far the most satisfactory was an austenitic nickel-chrome steel, the final resilience of which was greater than the initial value.

S. J. GREGG.

**Electric annealing of magnetic materials for telephone apparatus.** W. A. TIMM (Trans. Amer.

Soc. Steel Treat., 1926, 10, 782–799).—Coal- and oil-fired annealing furnaces have been replaced by a recuperative electric furnace treating six tons of telephone parts daily. The furnace is of the car type, eight annealing pots weighing 3000 lb. when charged being loaded on each car. No packing material is used around the parts in the pots. The furnace has two tracks, the cars passing in opposite directions through the three furnace compartments. Heating elements are placed only in the central compartments, but there are elements immediately below the top plate of each car. A cold charge entering an end compartment is preheated by a hot charge in process of cooling down. A car stays approximately 6 hrs. in each compartment. The maximum power input is 158 kw. Temperature control is automatic. The maximum temperature difference between individual pots is never more than 16°. Tests show that the magnetic quality and uniformity of the material are improved.

T. H. BURNHAM.

#### PATENTS.

**Production of wrought iron.** E. F. BLESSING (E.P. 261,449, 17.7.25).—Pig iron is refined in a furnace provided with hollow trunnions which rest on supporting rollers and connect with a chimney-shaft. The contour of the refractory lining of the furnace is designed so as to form a series of hearths for the several stages of the process. At the commencement, when the metal is in a liquid state, the hearth has a large radius of curvature, so that a large area of surface of the metal is exposed to the oxidising gases in the furnace. As the furnace is tilted the curvature of the bed increases, and finally, the iron, beginning to "ball up," is crowded into a comparatively deep and narrow section producing a rounded form or ball.

C. A. KING.

**Production of iron castings with a low carbon content.** K. EMMEL, and THYSSEN & Co., A.-G. (E.P. 244,405, 11.6.25. Conv., 9.12.24).—For the production of castings having a low carbon content, the charge for the cupola consists of 50% or more of wrought iron or steel and the remainder pig iron, together with the usual additions. The coke required amounts to 9–13% of the charge, and the blast pressure to 400–800 mm. water gauge. The scrap metal is used preferably in the form of briquettes.

C. A. KING.

**Manufacture of iron and steel alloys.** W. B. HAMILTON and T. A. EVANS (E.P. 262,206, 24.9.25. Cf. J., 1922, 220 A; J., 1923, 1028 A; B., 1924, 338, 522).—High-percentage iron and steel alloys may be produced from scrap containing the alloying metal by melting together a mixture of rustless iron scrap, with or without mild steel scrap, and slag-forming materials consisting of a mixture of limestone, scale (iron oxide), fluorspar, and chromite, the quantities being sufficient to make a bath of the required mass and a slag having sufficient mass and heat to start and support the exothermic action when the reduction mixture is added. Sufficient reduction mixture (ferrosilicon and chromite) is now added to increase the chromium content to that required, and must contain a proportion of thermo-reducing agent 10–15% in excess of that theoretically required in order to reduce back again into the bath all the chromium which may have become oxidised into the slag during



melting. The carbon content of the bath during melting may be reduced by the addition of a suitable oxide such as mill-scale. The high-percentage steel alloy so produced is free from contamination from the carbon electrodes and from gases from the electric arc.

M. E. NOTTAGE.

**Process for treating blast-furnace slag and the like.** A. and J. CRAWFORD (E.P. 261,976, 26.3.26).—To reduce the tendency of blast-furnace slag to disintegrate on exposure to the atmosphere, acid steel slag (1 to 1½ cwt.), preferably finely ground, is added to blast-furnace slag (1 ton) after leaving the furnace and while still in a molten condition. C. A. KING.

**Treatment of magnesium or its alloys for casting and other operations.** G. MICHEL (E.P. 261,528, 17.10.25).—A mixture of magnesium or its alloys with magnesium fluoride is heated and, while still in a semi-fluid condition, 0.05–0.3% of calcium is added in the form of small particles which will pass through a 5-mesh and be retained by a 50-mesh sieve. The addition of calcium lowers the m.p. of the metal or alloy, and causes vigorous agitation of the mass with subsequent separation of the magnesium fluoride, which rises to the surface carrying with it any chlorides and oxychlorides. The surface of the bath is then sprinkled with ammonium chloride, which reduces all the oxide impurities. Magnesium alloys are thus rendered perfectly stable, and are therefore adapted to all thermo-mechanical operations such as forging and rolling, and according to their composition possess hardness, elasticity, and ductility, and are capable of autogenous welding.

M. E. NOTTAGE.

**Solder for aluminium or its alloys and process for using same.** A. J. LINE (E.P. 262,192, 8.9.25).—The solder consists of a mixture of 3 pts. by wt. of pure silver and 12 pts. by wt. of aluminium heated together until the aluminium melts and the silver is absorbed by it; or filings of the metals may be mixed together and the solder used in this form. A nickel soldering bit is used without flux.

M. E. NOTTAGE.

**Purification of gallium, indium, and similar metals, and their alloys.** BRITISH THOMSON-HOUSTON CO., LTD., Assees. of S. BOYER (E.P. 256,950, 30.7.26. Conv., 12.8.25).—A film of a halogen compound is first formed on the surface of the electrolytically-deposited metal either by treating it with dilute hydrochloric acid (1:1), fusing it under the acid, and then resolidifying it, or by heating the metal in a container in contact with a dry halogen gas, the air having been first withdrawn. The metal is then placed in a quartz container, and the halogen compound volatilised by heating it at about 500° in a vacuum. Occluded gas is removed by raising the temperature to about 1000°. The metal is cooled, reheated several times, and then transferred to a second receptacle connected with the container, in which it is sealed off from the latter and finally removed by distillation or otherwise.

M. E. NOTTAGE.

**Manufacture of heavy-metal carbides and similar difficultly-melting compounds.** GEWERKSCHAFT WALLRAM (E.P. 242,951, 11.8.25. Conv., 14.11.24. Addn. to 239,499; B., 1925, 926).—Heavy-metal carbides, e.g., tungsten monocarbide, are melted in a

graphite container in an electric carbon tube furnace. The container forms part of a wedge-shaped block which extends outside the furnace for use as a handle, and in which a channel is cut to form a duct for the molten charge to run into a mould outside the furnace. Melting and carbon absorption are controlled by observation of a check block placed above the charge, and after a certain empirically determined time the charge is tapped in a condition of maximum fluidity.

C. A. KING.

**Metallurgical process.** C. L. BURDICK, Assr. to GUGGENHEIM BROS. (U.S.P. 1,606,343, 9.11.26. Appl., 7.6.24).—Volatile sulphur compounds of metals may be formed from metal-bearing material by subjecting the latter, at an elevated temperature, and in the presence of a sulphur component, to the action of a reducing gas in intimate contact with it. The volatile product is withdrawn and condensed.

M. E. NOTTAGE.

**Protective coatings for metals.** N. E. NORTH (E.P. 261,502, 17.9.25).—Iron or steel articles may be "sherardised" by heating them in contact with, or immersed in, a finely-powdered mixture of zinc with 1–20% of cadmium, with or without the addition of sand and ground carbon. The heating is continued at a temperature well below that of the m.p. of zinc, the furnace being rotated or periodically tilted during the heating.

M. E. NOTTAGE.

**Protection of ferrous metals against corrosion.** G. B. ELLIS. From T. E. MURRAY (E.P. 261,809, 24.6.25).—Ferrous metal is coated, preferably electrolytically, with chromium and nickel, and the coated metal heated so as to cause the chromium to alloy with the iron and to form an adherent oxide coating. Successive coatings of chromium, iron, chromium, and nickel may be subsequently applied.

J. S. G. THOMAS.

**Means for making gas-tight joints between metal and glass or quartz.** INTERNAT. GENERAL ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTROITÄTSGES. (E.P. 243,000, 12.11.25; Conv., 12.11.24. Addn. to E.P. 239,871).—The unglazed, slightly corrugated end of the glass or quartz is sprayed with metal, e.g., copper or brass, and the layer is tinned. The metal cap is then attached, preferably with the interposition of a ring washer, by soldering a metal casing over the cap and tinned layer.

A. COUSEN.

**Sealing metal to glass.** N. V. PHILIPS' GLOEILAMP-EN-FABR. (E.P. 251,951, 1.4.26. Conv., 11.5.25).—An alloy suitable for making an air-tight seal between metal discs, plates, wires, etc., and glass, consists of approximately 25% Cr, up to 5% Al, remainder Fe.

A. COUSEN.

**Sealing molybdenum or similar refractory metals and fused quartz.** H. MILLER, Assr. to HANOVIA CHEM. AND MANUF. CO. (U.S.P. 1,608,612, 30.11.26. Appl., 8.1.26).—Molybdenum and allied metals may be united with fused quartz by fitting the metal on to the quartz, treating it with certain salts of the alkali metals, heating the metal at its junction with the quartz until the latter becomes plastic, and then stressing or pressing the quartz until it flows and blends with the metal.

M. E. NOTTAGE.

**Alloys suitable for loading telephone and telegraph conductors.** W. S. SMITH and H. J. GARNETT



(E.P. 261,060, 10.7.25).—Alloys having a high sp. resistance and high initial magnetic permeability are composed of 62–69% Ni, 11–19% Fe, 6–10% Cu, 5–9% Cr, with (or without) not more than 0.5% Mn as a deoxidiser. An alloy containing 65% Ni, 18% Fe, 10% Cu, 7% Cr, and 0.5% Mn has  $\mu = 1200$ ,  $\rho = 99$  microhms/c.c. F. G. CROSSE.

**Alloy [for electrical contacts].** H. M. FREDRIKSEN (U.S.P. 1,605,432, 2.11.26. Appl., 2.8.23).—An alloy containing 87% Ag, 12% Cu, and 1% C. C. A. KING.

**Producing "non-sag" tungsten [filament] wire.** P. McALLISTER, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,605,192, 2.11.26. Appl., 23.12.22).—Tungsten filaments which do not sag in use are obtained by flashing tungsten wire in the vapour of a reducing agent, such as phosphorus or arsenic, either before or after insertion in the electric lamp.

T. S. WHEELER.

**Alloy steel.** H. B. KINNEAR, Assr. to MARION STEAM SHOVEL CO. (U.S.P. 1,607,086, 16.11.26. Appl., 30.12.25).—See E.P. 260,835; B., 1927, 46.

**Method for decarbonising highly carbonaceous ferrochromium.** L. FRIDERICH and W. RODENHAUSER, Assrs. to SIEMENS & HALSKE A.-G. (U.S.P. 1,608,270, 23.11.26. Appl., 5.9.25. Conv., 26.6.24).—See F.P. 600,079; B., 1926, 412.

**Process and apparatus for casting liquid molten metal.** A. KADOW, Assr. to VACUUM CASTING CO. (U.S.P. 1,606,236, 9.11.26. Appl., 15.3.26).—See E.P. 260,743; B., 1927, 47.

**Treatment of oxidised ores or other oxidised compounds of copper and zinc.** G. W. EDWARDS and H. T. DURANT (U.S.P. 1,608,844, 30.11.26. Appl., 10.11.23. Conv., 20.11.22).—See E.P. 215,802; B., 1924, 601.

**Magnetisable material [alloys].** E. SCHÜRER, Assr. to FELTEN UND GUILLEAUME CARLSWERK A.-G. (U.S.P. 1,608,434, 23.11.26. Appl., 13.8.25. Conv., 30.8.24).—See E.P. 239,231; B., 1925, 925.

**Puddling furnaces.** E. F. BLESSING (E.P. 261,812, 17.7.25).

**Metallurgical apparatus [moulds etc.].** Soc. ANON. MÉTALLURGIQUE D'AUBRIVES ET VILLERUPT (E.P. 261,269, 29.4.26. Conv., 3.4.26).

## XI.—ELECTROTECHNICS.

**High-frequency [induction] furnaces.** J. BRONN (Z. Metallk., 1926, 18, 333–338).—A discussion of the principles and efficiency of high-frequency inductive heating, with especial reference to the Ribaud furnace.

A. R. POWELL.

**Investigations relating to the pyrolusite [Leclanché] cell.** K. ARNDT, H. WALTER, and E. ZENDER (Z. angew. Chem., 1926, 39, 1426–1429).—The electrochemistry of the "dry" Leclanché cell is studied. The chemical changes occurring during discharge of the cell are shown to be represented by  $\text{Zn} + 2\text{NH}_4\text{Cl} + 2\text{MnO}_2 = \text{Zn}(\text{NH}_3)_2\text{Cl}_2 + \text{H}_2\text{O} + \text{Mn}_2\text{O}_3$ , and  $\text{Zn} + \text{H}_2\text{O} + 2\text{MnO}_2 = \text{Zn}(\text{OH})_2 + \text{Mn}_2\text{O}_3$ . The carbon-manganese dioxide electrode must be considered as a true oxygen

electrode, atmospheric oxygen taking part in the chemical changes involved during discharge of the cell.

J. S. G. THOMAS.

**Electric annealing of magnetic materials for telephone apparatus.** TIMM.—See X.

## PATENTS.

**Electrolyser for the production of hydrogen and oxygen.** "MONTECATINI" SOC. GEN. PER L'IND. MINERARIA ED AGRICOLA, Assees. of G. FAUSER (E.P. 242,635, 30.10.25. Conv., 7.11.24. Cf. U.S.P. 1,495,681; B., 1924, 633).—The electrodes employed are each enclosed in an asbestos canvas bag, and comprise two parallel plates spaced apart for circulation of an electrolyte and uniformly divided by vertical slots forming channels extending from their top to bottom edges. T-shaped spacing members are provided between adjacent vertical edges of the plates.

J. S. G. THOMAS.

**Process and apparatus for electrodeposition.** INTERNATIONAL COPPERCLAD CO., Assees. of T. ROBINSON (E.P. 238,230, 4.8.25. Conv., 5.8.24).—Apparatus for electrodeposition applicable to the manufacture of roofing elements comprises an anode and cathode suspended side by side and carried together through an electrolyte. Thus films of lead and copper may be successively deposited on a cathode, the films stripped from the cathode, and non-metallic bases fixed to them.

J. S. G. THOMAS.

**Electrolytic apparatus.** A. E. KNOWLES (E.P. 261,080, 11.8.25).—To maintain the temperature of the electrolyte, tubes or coils, through which hot or cold fluid may be circulated, are arranged adjacent to the ends of the electrodes and at right angles to their planes.

J. S. G. THOMAS.

**Electrolytic apparatus.** A. E. KNOWLES (E.P. 261,164, 19.10.25).—Water or other fluid, fed into one or more chambers in which the gas or gases evolved from the electrolytic cell bubble through the fluid, passes into a chamber containing a float operating a valve controlling the supply of fluid to the former chamber or chambers, and then flows to the cell in order to maintain the level of electrolyte therein.

J. S. G. THOMAS.

**Manufacture of metal sheets by electrodeposition.** A. Z. PEDERSEN (E.P. 261,592, 9.2.26).—A number of separable sheets of an anodically-corrosive metal, more especially nickel or cobalt, are deposited upon a cathode in an electrolytic bath, the direction of the current being reversed between the deposition of each sheet for a period of time which is short compared with that necessary for the deposition of a sheet.

J. S. G. THOMAS.

**Manufacture of an electrolyte.** A. MILLER (U.S.P. 1,605,756, 2.11.26. Appl., 19.7.24).—An electrolyte is composed of litharge, lead sulphate, lead peroxide, and sulphuric acid, which has been subjected to the action of an electric current, and from which surplus acid has been removed.

J. S. G. THOMAS.

**Diaphragms for primary or secondary cells, electrolytic cells, filters, and the like.** H. BECKMANN (E.P. 238,870, 6.8.25. Conv., 22.8.24).—A spongy rubber diaphragm having a large number of extremely minute pores is produced by coagulating latex or other suitable rubber dispersion by solutions of magnesium or



barium salts or other coagulating agents. If desired the pores may be impregnated with a solution of wood-extract, phenol, etc. J. S. G. THOMAS.

**Depolarising composition [for dry cells].** G. W. HEISE, Assr. to NATIONAL CARBON CO., INC. (U.S.P. 1,605,582, 2.11.26. Appl., 29.10.23).—A depolarising composition, for use in dry cells, is obtained by extracting diatomaceous earth with dilute sulphuric acid, grinding the purified earth with carbon, and incorporating the mixture with manganese dioxide. T. S. WHEELER.

**Electro-deposition of metals.** C. P. MADSEN, Assr. to MADSENELL CORP. (U.S.P. 1,608,706, 30.11.26. Appl., 26.4.19. Renewed 20.12.24).—See E.P. 142,432; B., 1921, 87.

**Electrical deposition of organic materials [rubber].** S. E. SHEPPARD and L. W. EBERLIN, Assrs. to KODAK, LTD. (E.P. 253,085, 30.4.26. Conv., 8.6.25).—See U.S.P. 1,580,795; B., 1926, 639.

**Thermo-electric element.** O. HERMANN, Assr. to THERMO ELECTRIC BATTERY CO. (Reissue 16,453 of U.S.P. 1,572,117, 9.2.26. Appl., 4.5.25).—See B., 1926, 371.

**Electrical treatment of gases and vapours** (U.S.P. 1,601,771).—See II.

**Refining petroleum [by electrical precipitation]** (U.S.P. 1,604,424).—See II.

**Protection of ferrous metals against corrosion** (E.P. 261,809).—See X.

**Alloys suitable for loading telephone and telegraph conductors** (E.P. 261,060).—See X.

**Producing "non-sag" tungsten [filament] wire** (U.S.P. 1,605,192).—See X.

**Alloy [for contacts]** (U.S.P. 1,605,432).—See X.

## XII.—FATS; OILS; WAXES.

**Rate of molecular weight increase in the boiling of linseed and tung oils.** J. S. LONG and G. WENTZ (Ind. Eng. Chem., 1926, 18, 1245—1248).—Only moderate concordance is obtained when various solvents are used in the determination of mol. wts. of thickened linseed and tung oils by the cryoscopic method, and benzene is recommended as a regular solvent. For thickened oils with mol. wt. over 2000, it is advisable to extract with hot benzene in a Soxhlet apparatus and adjust the concentration of the extract to 1%, the standard concentration adopted. The rates of mol. wt. increase of linseed oil heated alone and with various additions were determined. The presence of linolenic monoglyceride or of linolenic acid increases the rate whilst free glycerol retards it. Retardation is also induced by thickening the oil in an atmosphere of steam. These experiments, and a further one in which linseed oil was thickened in an atmosphere of carbon dioxide and steady evolution of water, acraldehyde, etc. took place coincidentally with uniform rise in mol. wt. and fall in iodine value, are proffered as evidence for the occurrence of condensation reactions. The removal of mucilage from linseed oil accelerated the increase of mol. wt. on heating. A significant fact in connexion with commercial oil boiling was the development of mucilage in a "non-breaking"

oil by the use of an atmosphere of steam, since varying amounts of water, inadvertently introduced, may develop the "break," with subsequent adsorption of driers which are thus rendered ineffective. The addition of sulphur to linseed oil caused a rapid increase of mol. wt. while the oil was heating up, but a considerably retarded rate of increase on maintaining the oil at 293°.

S. S. WOOLF.

**Rate of molecular weight increase in the boiling of linseed oil.** J. S. LONG and W. J. ARNER (Ind. Eng. Chem., 1926, 18, 1252—1253).—The effects of various driers on the rate of increase of the mol. wt. of linseed oil on heating are tabulated. Preliminary attempts are made to couple the various maxima of such physical properties of oil films as elongation or ultimate tensile strength with the molecular state of the oil from which the films are derived. S. S. WOOLF.

**Relationship during drying between the acid value of linseed oil and the concentration of cobalt acetate.** W. L. EVANS, P. E. MARLING, and S. E. LOWER (Ind. Eng. Chem., 1926, 18, 1229—1230).—The acid value of linseed oil films containing varying concentrations of cobalt acetate, drying under standard conditions, is an increasing function of the time. The acid value is also expressed as a function of the cobalt acetate concentration, graphically and by a series of exponential equations. S. S. WOOLF.

**Rate of polymerisation of perilla oil.** M. TOCH and T. T. LING (Ind. Eng. Chem., 1926, 18, 1285—1287).—The polymerisation of perilla oil, examined at temperatures up to 305°, is shown to proceed with the evolution of volatile matter probably including acraldehyde. The original oil of  $d_{20}^{20}$  0.9134,  $n_D^{25}$  1.4812, acid number 3.10, and iodine value (Wijs) 201 gives at the end of 3 hrs. "bodied" oils having the characteristics  $d_{20}^{20}$  0.9672 and 0.9722,  $n_D^{25}$  1.4914 and 1.4923, acid number 13.62 and 12.22, and iodine value 141 and 138 according as the polymerisation is conducted by heat alone or by heat and "blowing," the latter oil being much darker in colour. Tables are given showing the values obtained for samples taken from a larger scale experiment in which the heating was continued for 8 hrs. It is demonstrated that the mean mol. wt. of the oil rises uniformly with the density, refractive index, and acid value, and inversely as the iodine value, between the values 753 at the start and 1650 after 8 hrs. E. HOLMES.

**Fatty acids of whale oils. II. Fatty acids of long-headed finner (sei) whale oil.** Y. TOYAMA (Chem. Umschau, 1926, 33, 293—299, and J. Soc. Chem. Ind. Japan, 1926, 29, 531—538).—The oil under examination, of  $d_4^{20}$  0.9106,  $d_4^{15}$  0.9203, acid value 0.98, saponif. value 168.3, iodine value (Wijs) 121.3,  $n_D^{30}$  1.4699,  $n_D^{20}$  1.4736, and unsaponif. matter 2.29%, gave mixed fatty acids having the following characteristics:  $d_4^{20}$  0.84, acid value 192.2, saponif. value 194.1, iodine value (Wijs) 125.9,  $n_D^{30}$  1.4609, and ether-insoluble bromides 25.59%. By methods similar to those used in Pt. I (B., 1924, 1019) the fatty acids are separated and identified as follows. The saturated acids amount to 20% and those of the oleic series to not more than 75% of the total acids. The saturated acids consist largely of palmitic acid; myristic, stearic, and arachidic acids are present in



smaller amounts, whilst behenic acid and acids of fewer carbon atoms than 14 appear in very small amounts. Acids of the oleic series are zoomaric, oleic, an acid  $C_{20}H_{38}O_2$ , and cetoleic acid  $C_{22}H_{42}O_2$ . There is a possibility of the presence of tetradecenoic acid and of unsaturated acids having more than 22 carbon atoms in very small amount. The unsaturated acids with more than one double linking consist largely of those with 20 and 22 carbon chains, with smaller amounts of 18-carbon acids, including  $C_{18}H_{34}O_2$  and  $C_{18}H_{30}O_2$ .  $C_{20}H_{32}O_2$  occurs in the  $C_{20}$  acids, and among the  $C_{22}$  acids is  $C_{22}H_{34}O_2$ —probably clupanodonic acid—and smaller amounts of  $C_{22}H_{36}O_2$ .

E. HOLMES.

**Dibromides of oleic and elaidic acids and the purification of oleic acid.** D. HOLDE and A. GORGAS (Z. angew. Chem., 1926, 39, 1443—1446).—Crude oleic acid, iodine value (Hanus) 92.14, is treated with bromine, and the resulting product dissolved in light petroleum (b.p. 30—50°). By successive fractional crystallisations at —21° and —78° oleic acid dibromide is obtained as a solid, m.p. 28.5—29°. Elaidic acid, m.p. 43—44°, similarly treated yields the dibromide, m.p. 29—30°, the crystalline form of which is obviously different from that of its isomeride, mixed m.p. 20°. Oleic acid obtained by reduction of its dibromide distils at 200°/0.6 mm., has iodine value (Hanus) 89.96, and m.p. 13.5° after cooling at 0° for  $\frac{1}{4}$  hr., 14.0° after 3 hrs. The results obtained confirm the findings of Nicolet (A., 1921, i, 390) that the addition and subsequent removal of bromine cause no *cis-trans* isomerisation in this series.

E. HOLMES.

**Fatty acids of whale oils. IV. Fatty acids of Californian grey whale oil.** Y. TOYAMA (J. Soc. Chem. Ind. Japan, 1926, 29, 538—546, and Chem. Umschau, 1927, 34, 19—25).—The fatty acids of Californian grey whale oil of  $d_{4}^{15}$  0.9290, acid value 0.50, saponification value 191.0, iodine value 166.7,  $n_D^{20}$  1.4788, unsaponifiable matter 1.55%, were examined. The mixed fatty acids contain about 10% of saturated acids and the unsaturated acids about 60% or less of acids of the oleic series. Amongst the saturated acids, palmitic acid preponderates, with myristic and stearic acids in smaller amount, and a trace of arachidic acid. The presence of a small amount of saturated acids having less than 14 carbon atoms is also indicated. The acids of the oleic series consist mainly of zoomaric and oleic acids.  $C_{20}H_{38}O_2$  and  $C_{22}H_{42}O_2$  are also present. A tetradecenoic acid,  $C_{14}H_{26}O_2$ , is present in a small amount. The acids more unsaturated than those of the oleic series consist chiefly of  $C_{20}$  and  $C_{22}$  acids, mainly  $C_{20}H_{32}O_2$  and  $C_{22}H_{34}O_2$ , also  $C_{20}H_{30}O_2$ ,  $C_{24}H_{38}O_2$ , and  $C_{22}H_{36}O_2$ .  $C_{16}$  and  $C_{18}$  acids ( $C_{16}H_{26}O_2$ ,  $C_{16}H_{24}O_2$ ,  $C_{18}H_{28}O_2$ ,  $C_{18}H_{30}O_2$ ) also occur in the acids giving ether-insoluble bromides.

K. KASHIMA.

**Fatty oils as substitutes for ethyl alcohol in citrus flavours.** H. A. SCHUETTE and B. P. DOMOGALLA (Ind. Eng. Chem., 1926, 18, 1254—1257).—In a search for substitutes for ethyl alcohol in the preparation of orange and lemon flavours the following fatty oils have been examined: sweet almond, corn, cottonseed, neutral lard, olive, peanut, rape, sesame, and soybean oils, all of minimum acidity. The solutions are analysed by a polariscopic method in a 100 mm. tube,

the appropriate factors for lemon and orange oils being 1.7 and 2.7 respectively. Baking tests on flavours consisting of 5% by volume of the essential oils in the various fatty oils show them to be equal to, if not better than, the alcoholic extracts, but the lack of stability of such flavours militates against their general use. Acid numbers of these solutions show in 54 months an increase of 25—55% for the lemon flavours, and 19—100% for the orange flavours.

E. HOLMES.

**Detergent action of soaps.** R. M. WOODMAN (Chem. News, 1926, 133, 339—341, 353—356).—Phenol, hydrogenated phenols, and cresylic acid are much more soluble in soap solutions than in pure water, and addition of these substances greatly increases the solvent power of soap solutions for paraffin oil, anthracene oil, coal-tar creosote, etc. Vegetable oils and oleic acid were found to be quite insoluble. Pickering (B., 1917, 462) has shown that the more concentrated a soap solution the greater is the solvent action per unit of soap. The present measurements show, however, that the influence of concentration is not of such importance when phenolic substances are added to the soap. The presence of phenols, hydrogenated phenols, or cresols renders soaps much more soluble, and more easily soluble, in water, yielding clear solutions. Further, besides possessing greater solvent and emulsifying powers for hydrocarbon oils, solution and emulsification are much more rapid, thus tending to increase detergent action.

J. S. CARTER.

**Apparatus for the determination of moisture [in soap] by distillation with xylene.** PRITZKER and JUNGKUNZ.—See I.

**Effects of ultra-violet light on paint vehicles.** STUTZ.—See XIII.

**Function of oils and fats, and emulsions of oil and water in breadmaking with special reference to gluten formation and modification.** BENNION.—See XIX.

#### PATENTS.

**Refining and decolorising oils and fats.** A. S. QUICK (E.P. 261,440, 18.6.25).—Gases rich in oxygen are passed through the oil or fat to be refined, in the presence of water and a radioactive material, e.g., monazite sand. The addition of very small quantities of alkaline substances and/or substances capable of absorbing pigments, e.g., charcoal, fuller's earth, etc., is sometimes necessary. Temperature and duration of treatment depend on the nature and proposed use of the oil treated. (Cf. E.P. 161,813; B., 1921, 397.) S. S. WOOLF.

**Manufacture of butter fat products (U.S.P. 1,605,009).**—See XIX.

**Process of producing edible fat (U.S.P. 1,605,108).**—See XIX.

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

**Application of the statistical method in testing paints of durability.** J. H. CALBECK (Ind. Eng. Chem., 1926, 18, 1220—1222).—Results of exposures on the N. Dakota test fences (1915 and 1921) are statistically analysed, enabling the influence on durability of particular



variables to be clearly defined. The significance of the "volume ratio" (percentage by volume of the pigment phase in the dried paint film) is established, 28% being the probable minimum compatible with satisfactory durability, but the supposed existence of an optimum percentage of zinc in mixed lead-zinc paints is not confirmed. The extent of "chalking" after one year's exposure of a paint is not a reliable indication of its durability.

S. S. WOOLF.

**Accelerated weathering.** H. A. NELSON and F. C. SCHMUTZ (Ind. Eng. Chem., 1926, 18, 1222—1227).—The destructive agencies made use of in laboratory accelerated weathering cycles are correlated with their counterparts in the actual weathering process. From consideration of the effects of ultra-violet light, alone and in conjunction with heat, humidity, and oxygen or ozone-enriched atmospheres, the solvent and erosive functions of water sprays, and the varying action of temperature changes according to the original elasticity of the film under test, a cycle is devised which successfully simulates the weather conditions prevailing in a particular locality (Palmerton, Pa., U.S.A.).

S. S. WOOLF.

**Cause and prevention of staining on white paint.** H. T. MORGAN and J. H. CALBECK (Ind. Eng. Chem., 1926, 18, 1227—1228).—The staining of white paints in contact with iron or copper is shown to be due to the presence of zinc oxide, which, unlike other white pigments, precipitates colloidal metal hydroxides from solution.

S. S. WOOLF.

**Effects of ultra-violet light on paint vehicles.** G. F. A. STUTZ (Ind. Eng. Chem., 1926, 18, 1235—1238).—The exposure of linseed, perilla, poppy seed, soyabean, and castor oils, in mass, to the action of ultra-violet light at 50° (with the passage of a slow stream of air, oxygen, or nitrogen through the oil) produced similar results to the application of heat or air-blowing, *i.e.*, decrease in iodine value, and increase in viscosity, mol. wt., refractive index, and acid value. The degree to which ultra-violet light (over the wave-length interval from 3655—2300 Å) is absorbed by oils was observed by measuring the light transmitted through a cell containing the oil under test, using an ultra-violet spectrophotometer. This was carried out for the various oils, before and after treatment with ultra-violet light, as well as for several commercial samples of treated and bodied linseed oils, and various fatty acids etc. When the degree of absorption shown by the oil is expressed by the relationship  $I = I_0 \cdot 10^{-Kt}$ , where  $I$  = intensity of transmitted light,  $I_0$  = intensity of incident light,  $t$  = film thickness, and  $K$  = absorption coefficient,  $K$  is found to increase with mol. wt. and viscosity in all cases. Exposure to light has, therefore, a cumulative effect by making the oil more absorbent for ultra-violet rays.

S. S. WOOLF.

**Developments in controlling tung oil varnishes.** W. W. BAUER (Ind. Eng. Chem., 1926, 18, 1249—1251).—A résumé of current views on the solidification or "crystallising" of tung oil is given. The "burnt coal gas" test for producing frosting of tung oil varnish films is criticised, and it is established that the presence

of oxygen is essential for "crystallisation," whereas both the formation of a solid modification and the production of frosted films have been achieved in the absence of light (contrary to previous statements). The rate and extent of "crystallisation" are increased by factors which permit an increased rate of oxygen absorption, oxides of nitrogen being found to act catalytically in this sense. A test method is described, according to which tung oil varnish films remain for 1 hr. at ordinary temperatures in a uniformly distributed atmosphere supplied by bubbling air through concentrated nitric acid. Varnishes that do not frost under these conditions may be expected to dry glossy under all normal conditions. The tendency to "crystallise" in varnishes is marked by the development of a surface gel, which may re-dissolve.

S. S. WOOLF.

**Analysis of lacquers.** B. J. OAKES (Ind. Eng. Chem., 1926, 18, 1232—1233).—In a general discussion of lacquer analysis the importance of preliminary physical tests is stressed, such phenomena as "sweating" or discoloration under heat, brittleness on bending, etc. affording useful guidance in the subsequent stages. For actual analysis, a recommended procedure is as follows:—100 g. of lacquer are stirred into about 800 c.c. of toluene. The pigment stays with the gelled nitrocellulose, and by repeated extractions with benzene the other ingredients are removed practically entirely and further examined. The nitrocellulose is separated from the pigment by extraction with acetone and centrifuging. Synthesis as a confirmation of analytical results is necessary.

S. S. WOOLF.

**Physical constants and individual properties of cellulose lacquer solvents, thinners, and plasticisers.** C. P. VAN HOEK (Farben-Ztg., 1926, 32, 624—628).—The following particulars of over 200 organic substances are tabulated:—formula, b.p./760 mm., m.p.,  $d^{15}_4$ ,  $n^{20}_D$ ,  $\alpha_D$ , inflammability, odour, physiological action, solubility, and solvent action.

S. S. WOOLF.

**Interpretation of stress-strain curves on lacquer and lacquer components.** W. A. McKIM (Ind. Eng. Chem., 1926, 18, 1239—1242).—From consideration of the stress-strain curves of experimental lacquer films of various ages, subjected to variations in temperature, humidity, and light, the influence of specific constituents, *e.g.*, plasticisers, high-boiling solvents, resins, pigments, on strength and elasticity is discussed.

S. S. WOOLF.

**Varnish and stoving lacquers for preserve cans.** H. SERGER (Farben-Ztg., 1926, 32, 685—686).—The requirements (legal and general) of, and methods of testing, protective coatings for the inside of preserve tins are described. Temperature of stoving is shown to be of importance. An electrochemical explanation is offered of the observed fact that corrosion is more severe in cases of damage to the stoved lacquer film than if no varnish had been applied.

S. S. WOOLF.

**Determination of metallic lead in pigments.** MCINTOSH.—See X.

**Boiling of linseed oil and tung oil.** LONG and WENTZ.—See XII.

**Boiling of linseed oil.** LONG and ARNER.—See XII.



**Drying of linseed oil.** EVANS and others.—See XII.

**Polymerisation of perilla oil.** TOCH and LING.—See XII.

#### PATENTS.

**Preparation of titanium compounds [for pigments].** P. SPENCE & SONS LTD., W. B. LLEWELLYN, and S. F. W. CRUNDALL (E.P. 261,051, 8.5.25).—Precipitated hydrated basic titanate phosphate is obtained in a filterable and washable form by slowly adding a solution of sodium phosphate or phosphoric acid, while stirring, to a solution of titanium sulphate or chloride containing less than 10%  $\text{TiO}_2$ , and free hydrochloric acid, at 80–100°, in the presence of finely divided barium sulphate.

H. ROYAL-DAWSON.

**Manufacture of a brown pigment containing titanium sludge.** DEUTSCHE GASGLÜHLICHT-AUER-GES.M.B.H. (E.P. 257,229, 22.6.26. Conv., 22.8.25).—The sludgy residues obtained by treating titaniferous ores with sulphuric acid are neutralised, washed, dried, calcined, and ground. The final stage of neutralisation is effected by the use of zinc oxide, strong alkalis being used up to this point. The colour of the finished pigment is determined by the temperature of calcination.

S. S. WOOLF.

**Producing a titanium [oxide] pigment.** R. H. MONK, Assr. to J. IRWIN (U.S.P. 1,605,851, 2.11.26. Appl., 21.3.25).—Precipitated titanium hydroxide, free from iron, mixed with a filler, such as barium sulphate, and sawdust or other carbonaceous material (8%), is calcined at about 800°, when a pure white titanium oxide pigment is obtained.

T. S. WHEELER.

**[Cellulose] varnish.** E. LAYADOUX (E.P. 261,589, 5.2.26).—To a cellulose varnish comprising nitrocellulose, castor oil, amyl or ethyl acetate, and benzene or benzine, is added "Oriental essence," a suspension of fish scales in aqueous ammonia. The mixture yields a flexible, resistant, glossy, and permanent film.

S. S. WOOLF.

**Lacquer for shoe heels etc.** F. G. EDBROOK (U.S.P. 1,607,516, 16.11.26. Appl., 11.7.23. Conv., 26.7.22).—A lacquer containing equal proportions of cellulose acetate and dichlorohydrin, and a solvent consisting of acetone, together with benzyl alcohol, industrial alcohol, and benzene.

H. ROYAL-DAWSON.

**Manufacture of resinous compositions.** METROPOLITAN-VICKERS ELECTRICAL CO. LTD., Assees. of H. C. P. WEBER (E.P. 253,519, 7.6.26. Conv., 10.6.25).—A phenol-furfuraldehyde resin is mixed with a "glyptal" (glyceryl phthalate) resin (both being in either the A- or B-stage of condensation) with or without fillers or the use of a solvent. The composite resin is "cured" to any required stage, and retains the good insulating and resistant properties of the "glyptal" resin, together with the ease of moulding of phenol-furfuraldehyde resins. The improved rate of conversion to the C-stage suggests a mutual condensing action of the two types of resin.

S. S. WOOLF.

**Synthetic resins.** CHEM. FABR. DR. K. ALBERT G.M.B.H., A. AMANN, and E. FONROBERT (E.P. 261,522, 12.10.25).—The intermediate condensation product of phenols and aliphatic, aromatic, or mixed ketones is treated at a temperature not above 80° with an aldehyde

in an alkaline medium. The resin subsequently precipitated with acid may be converted into an infusible, insoluble product by drying or heating.

S. S. WOOLF.

**Hardening fusible phenolic resins.** T. S. HUXHAM, Assr. to AMERICAN INSULATOR CORP. (U.S.P. 1,606,943, 16.11.26. Appl., 27.3.24; cf. Mains and Phillips, B., 1921, 357 A).—Cold-moulding compositions suitable for the manufacture of insulators etc. are prepared by dissolving a fusible phenol-aldehyde resin (100 pts.) in a suitable high-boiling hydrocarbon solvent, and adding up to 90 pts. of the resinous product formed by the interaction of equimolecular proportions of furfuraldehyde and aniline, or other aromatic amine, and suitable amounts of fillers and pigments. The mixture is hardened when required by being heated at 150° for several hours.

T. S. WHEELER.

**Condensation product of acrolein and process of producing the same.** C. MOUREU and C. DUFRAISSE (U.S.P. 1,607,293, 16.11.26. Appl., 29.3.22. Conv., 31.3.19).—See E.P. 141,058; B., 1921, 858.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

#### PATENTS.

**Production of rubber isomer.** H. L. FISHER, Assr. to B. F. GOODRICH CO. (U.S.P. 1,605,180, 2.11.26. Appl., 26.12.24. Conv., 24.9.24).—See E.P. 249,172; B., 1926, 453).

**Caoutchouc composition.** A. BIDDLE, Assr. to UNITED PRODUCTS CORP. OF AMERICA (Reissues 16,476—7, 16.11.26, of U.S.P. 1,437,487, 5.12.22).—See B., 1923, 107 A.

**Manufacture of a rubberised fibre composition** (U.S.P. 1,606,595).—See V.

**Production of lampblack** (U.S.P. 1,606,380). See II.

**[Rubber] diaphragm for electrolytic cells etc.** (E.P. 238,870).—See XI.

### XV.—LEATHER; GLUE.

**Maximum reactivity of the hide protein in its isoelectric zone.** K. H. GUSTAVSON (J. Amer. Chem. Soc., 1926, 48, 2963—2971).—This paper deals with a new type of protein reactions, which cannot be explained on the basis of a salt formation by means of primary valency. The salt studied was *cis*-sodium dioxalato-diaquochromate  $\text{Na}[(\text{H}_2\text{O})_2\text{Cr}(\text{C}_2\text{O}_4)_2]$  (cf. Werner, A., 1915, i, 375). Solutions of this salt were adjusted by addition of *N*-sodium hydroxide so that four series of solutions, containing 5.0, 10.0, 24.0, and 55.0 g. of  $\text{Cr}_2\text{O}_3$ /litre, were obtained, each over a range of  $p_{\text{H}}$  1—8. These solutions were aged for more than six months. Weighed amounts of American standard hide powder were shaken with measured volumes of these solutions for 48 hrs., the solutions were filtered, and the  $p_{\text{H}}$  values of the filtrates determined electrometrically. The residues were analysed for chromium and protein. The fixation of the anionic chrome complexes from the oxalatochromate was found to take place on both sides of the isoelectric point of the hide protein, with a pronounced



maximum in the region of that point. Since, at the isoelectric point, the formation of protein salts of primary valency nature is, as a rule, not possible, the production of molecular compounds by means of secondary valency forces from the basic protein groups to the central atom in the anion complex appears to be the most reasonable explanation of these results. Experiments with the oxalatochromate solutions and de-aminised hide powder indicated that the chrome fixation reaction was practically independent of the extent of ionisation of the hide protein, since only a slight shift of the maximum zone was observed, and that the basic protein groups were chiefly involved in the anionic chrome fixation, a retardation of chrome fixation being obtained. This appears to indicate that in the fixation of cationic chromium the acidic protein groups are concerned (cf., Thomas and Kelly, B., 1926, 600). A number of facts are adduced to support this view of the dual nature of chromium fixation. For example, cationic chrome-tanned hide powder gives, upon treatment with solutions of oxalatochromate, the same additional chrome fixation as a regular hide powder, but does not adsorb chromium from a cationic chromium salt. F. G. WILLSON.

**Adsorption of acids by hide in relation to swelling.** II. P. N. PAVLOV and G. G. TIMOCHIN (Kolloid Z., 1926, 40, 129—135. Cf. B., 1926, 958).—The hide was immersed in solutions of succinic or acetic acid for 24 hrs., and the initial and final concentrations of acid were determined by titration with phenolphthalein as indicator. From the apparent adsorption curves the true adsorption isotherms can be deduced. The adsorption coefficients of succinic and acetic acids are independent of the volume of the solution and mass of the adsorbent, and a partition of the acid between the aqueous and collagen phases evidently occurs.

E. S. HEDGES.

**Influence of hydrogen-ion concentration and of neutral salts on the degree of aldehyde tanning.** O. GERNGROSS and R. GORGES (Z. angew. Chem., 1926, 39, 1432—1435).—Hide powder is tanned for 5 hrs. in 0.95% formaldehyde solution of  $p_H$  range 3 to 12, and the degree of tanning of the resulting leathers determined as the "water-stability" number =  $WB = 100(a - a')/a$ , where  $a$  is the amount of hide substance originally present in the test sample, and  $a'$  the hide substance lost by extraction of the resulting leather with hot water under standard conditions. The method gives results reproducible to  $\pm 1$  unit. Tables of results and a graph demonstrate the following points: (a) The tanning, which is scarcely perceptible at  $p_H$  3, rises slowly and uniformly to  $p_H$  6.3; (b) between  $p_H$  6 and 7 there is a relatively enormous rise in the amount of tanning; (c) between  $p_H$  8 and 12 the degree of tanning is constant at the high value,  $WB = 78$ . Common salt solution  $4/5$  saturated and 0.9N- to 0.09N-potassium thiocyanate caused a sensible decrease in the degree of formaldehyde tanning of lamb skin, whilst the common salt itself in solution of  $p_H$  4.5 has no appreciable effect. Saturated salt solution of  $p_H$  2.5 to 3.6 suppresses all tanning, whereas a strongly alkaline solution,  $p_H$  8.6 to 11.2, gives a beautiful leather of  $WB = 87$ . When lamb skin is tanned with formaldehyde solution to

which has been added egg yolk the resulting leather is improved both in appearance and pliability.

E. HOLMES.

**Quantitative determination of gelatin.** E. LENK (Biochem. Z., 1926, 177, 434—448).—The determination of gelatin in commercial samples and in glue cannot be effected by the viscosity method, since the latter is influenced by several factors, e.g.,  $p_H$ , ageing, salt content, homogeneity, etc. A new method is described, the percentage concentration of gelatin ( $y$ ) being given by the relation  $y = -0.55x + 1.8$ , where  $x$  = logarithm of the gelatinisation time in min. The maximal error of the method is 2%, with times of gelatinisation ranging from 10 to 60 min. Commercial gelatin contains 70—100%, and glue 10—40% of gelatin. The curve showing the relationship between the time of gelatinisation and  $p_H$  has a sharp turning point between  $p_H$  4.6 and 5.9; the freer the material from ash, the nearer is this point of inflexion to  $p_H$  4.6. Between  $p_H$  7.0 and 7.5 the curve becomes practically parallel with the abscissæ. Determinations of gelatin by this method are therefore carried out at this  $p_H$  (i.e., after neutralisation to litmus).

P. W. CLUTTERBUCK.

PATENT.

**Process of removing hair from hides or skins.** H. C. ROSS and H. C. MARRIS, Assrs. to W. WALKER & SONS, LTD. (U.S.P. 1,605,988, 9.11.26. Appl., 13.11.24).—See E.P. 241,666; B., 1926, 23.

## XVI.—AGRICULTURE.

**Studies on contact insecticides.** I. Introduction and methods. II. Toxicity of *Tephrosia Vogellii*, Hook, to *Aphis rumicis*, L. III. Insecticidal action of chloro-, nitro-, and hydroxy-derivatives of benzene and naphthalene. IV. Toxicity of certain plants and plant products to *Aphis rumicis*, L. F. TATTERSFIELD, C. T. GIMMINGHAM, and H. M. MORRIS (Ann. Applied Biol., 1925, 12, 61—65, 66—76, 218—262; 1926, 13, 424—445).—I. Methods adopted to obtain quantitative data with regard to the insecticidal properties of a number of groups of synthetic compounds and natural plant products are described. Adults of *Aphis rumicis* (black bean aphid) and eggs of *Selenia tetralunaria* (purple thorn moth) were used as test subjects. The insects were sprayed under controlled conditions, and afterwards kept under observation, counts being made of the numbers affected by the treatments. By testing compounds at a series of concentrations, the results can be graphed, plotting the concentrations against the percentages killed. II. Aqueous and alcoholic extracts of the leaves and seeds of the leguminous plant *Tephrosia Vogellii*, which occurs in many parts of the tropics, and in some districts is used as a fish poison, were highly toxic to *A. rumicis*. Aqueous extracts at concentrations equivalent to 1% or more of the leaves and seeds, and emulsions containing 0.1% or more of the dried alcoholic extract, were completely toxic. Extracts of the stems were less poisonous. III. The toxicity of a number of chloro-, nitro-, and hydroxy-derivatives of benzene and naphthalene to *A. rumicis* and eggs of *S. tetralunaria* was investigated. The introduction of chlorine or of a nitro-group into the benzene ring increases toxicity, and



a second nitro-group still further accentuates it. The following order of toxicity was noted: benzene < toluene < xylene < chlorobenzene < *p*-dichlorobenzene < *o*-dichlorobenzene < trichlorobenzene < nitrobenzene < *m*-dinitrobenzene. The nitro-phenols and -cresols are all more toxic than the parent substances. The dinitro-compounds are more toxic than the mono- or trinitro-compounds. The order of toxicity of the phenols was: phenol < *o*-nitrophenol < *m*- and *p*-nitrophenol < 2:4-dinitrophenol > trinitrophenol; and the same order applies to the cresols and their derivatives. With few exceptions, the relative toxicities to the aphides and to the eggs were approximately in the same order.  $\alpha$ -Chloronaphthalene was the most toxic naphthalene derivative tested. 2:4-Dinitrophenol and 3:5-dinitro-*o*-cresol showed specially powerful insecticidal properties; the latter was completely toxic to the eggs at concentrations between 0.2 and 0.1%. It is suggested that compounds of this type, although injurious to foliage, may have practical value as winter spray fluids for fruit trees in dormant condition. (Cf. succeeding abstract.) IV. The toxicity to *A. rumicis* of extracts of a number of plants, including some tropical fish poisons, was studied. Alcoholic extracts of the roots and stems of white haiari and the stems of black haiari (species of *Lonchocarpus* from British Guiana), the roots of *Tephrosia toxicaria*, and the leaves of *T. Vogelii* all possessed high insecticidal properties. Certain substances isolated from these plants were also tested. The most toxic derivative obtained from the haiaris was shown to be identical with tubatoxin, the crystalline poison found in *Derris elliptica*. Tubatoxin was more toxic to *A. rumicis* than nicotine. Of a number of alkaloids tested, cytosine, lobeline, and eserine were rather less toxic than nicotine. None of the other alkaloids tried approached nicotine in toxicity.

C. T. GIMINGHAM.

**Toxicity of 3:5-dinitro-*o*-cresol and other compounds to insect eggs, under laboratory and field conditions.** C. T. GIMINGHAM, A. M. MASSEE, and F. TATTERSFIELD (Ann. Applied Biol., 1926, 13, 446—465. Cf. preceding abstract).—Further laboratory experiments on the toxicity of 3:5-dinitro-*o*-cresol and its sodium salt to eggs of the moth *Selenia tetralumaria* are reported. The results confirmed the high toxicity of these compounds. The sodium salt was shown to be slightly less toxic than the dinitrocresol itself. Spray fluids containing 0.25 and 0.15% of dinitrocresol or the sodium salt were tested under field conditions, and showed a high efficiency against the eggs of the hop-damson aphid on plum trees. Counts of eggs on selected shoots on sprayed and control trees, before and after spraying, gave a reliable numerical measure of the effect of the treatments. No injury to the trees, due to the use of these spray fluids, was observed.

C. T. GIMINGHAM.

**Determination of phosphoric acid in soils.** D. J. HISSINK and M. DEKKER (Rijkslandbouwprefstation, Groningen, 21.11.24; Bied. Zentr., 1926, 55, 436—437).—For the determination of acid-soluble phosphate, 12.5 g. of air-dried and finely powdered soil are gently heated for 5 hrs. in a platinum crucible, with occasional stirring. The soil is then transferred to a flask and heated with 150 c.c. of 12.5% nitric acid on a glycerin

bath (150°) for 75 min. After cooling, 5 c.c. of concentrated sulphuric acid are added, and the mixture is cooled and diluted to 250 c.c. and filtered. 50 c.c. of the filtrate are treated with 50 c.c. of sulphate-molybdate reagent (Lorenz), and, after 24 hrs., filtered (Neubauer-Tiegel method), and the precipitate is washed with ammonium nitrate solution and acetone, and dried for 30 min. *in vacuo*. Citric-soluble phosphate is determined by shaking 55 g. of air-dried, finely powdered soil with 500 c.c. of 2% citric acid solution for 48 hrs. at room temperature. 200 c.c. of the filtered extract (100 c.c. only if soil is rich in chalk) are evaporated to dryness with 50 c.c. of concentrated nitric acid. The residue is dissolved in a sufficiency of hot water containing 10 c.c. of 10% nitric acid, filtered, evaporated to about 15 c.c., treated with 35 c.c. of nitric-sulphuric acid solution and 50 c.c. of sulphate-molybdate solution, according to the Lorenz method, by which the analysis is completed.

A. G. POLLARD.

**Action of lime on clay soils.** D. J. HISSINK (Landbouwkundig Tijds., 1925, 37, 442—444; Bied. Zentr., 1926, 55, 484—486).—From determinations of the amounts of calcium carbonate and exchangeable calcium in limed soils, the author has calculated the percentage efficiency of dressings of lime on three different soils to be 80%, 3%, and 44% respectively. The differences are attributable partly to the character of the soils and partly to the mode of application of the dressings.

G. W. ROBINSON.

**Action of lime on fen soils.** D. J. HISSINK (Landbouwk. Tijds., 1926, 38, 448—449; Bied. Zentr., 1926, 55, 486—488. Cf. preceding abstract).—Data are given to show the fate of calcareous dressings applied to fen soil. The proportion of calcium oxide appearing finally in the form of "humus lime" was 87.5%, 95.1%, and 49.7% respectively in three cases, the last two being in different parts of the same field.

G. W. ROBINSON.

**Action of lime on a Roodoorn soil.** D. J. HISSINK (Landbouwk. Tijds., 1925, 37, [45]; Bied. Zentr., 1926, 55, 447—8).—The soil was a stiff clay with high humus content. The effect of liming on the saturation capacity, replaceable calcium, and  $p_H$  value of this soil is recorded. Liming was found to increase the availability of phosphates for plants.

A. G. POLLARD.

**Determination of easily soluble soil phosphates.** M. GERLACH and O. NOLTE (Mitt. Deut. Landw.-Ges., 1926, 10; Bied. Zentr., 1926, 55, 448—450).—Comparison is made of results of phosphate determinations by a number of existing methods. Generally speaking, crop increases following the application of phosphatic manures, decreased with increasing amounts of citric-soluble phosphate in the original soils. Exceptions to this were frequent, however, and it was judged unsafe to use the method for evaluating fertiliser requirements. By employing the Neubauer method, it was observed that soils having a high "root-soluble" phosphate content were usually those benefiting least by treatment with phosphate fertilisers. No definite relationships were apparent, and exceptional cases were numerous. In the majority of soils examined, low citric-soluble phosphate contents were coincident with low "root-soluble" phosphate content. It is considered that field



trials alone offer a suitable basis for the determination of the phosphate deficiency of soils. A. G. POLLARD.

**Phosphate solubility and the growth of *Azotobacter*.** H. NIKLAS, K. SCHARER, and A. STROBEL (Landw. Jahrb., 1926, 63, 387—410; Bied. Zentr., 1926, 55, 488—490).—The authors have studied the development of *Azotobacter* in cultures containing increasing amounts of various phosphates. The utilisation of the different phosphates as shown by the magnitude of the optimal additions can be correlated with their chemical solubility in different solvents.

G. W. ROBINSON.

**Determination of the manurial requirements of soils.** M. GERLACH (Landw. Jahrb., 1926, 63, 339—368; Bied. Zentr., 1926, 55, 481—484).—A critical review of the present position of the problem of determining the manurial requirements. G. W. ROBINSON.

**Further manurial experiments with superphosphate, basic slag, Rhenania phosphate, and dicalcium phosphate on four different types of soil.** H. NIKLAS, A. STROBEL, and K. SCHARER (Landw. Jahrb., 1926, 63, 607—625; Bied. Zentr., 1926, 55, 493—495).—In field experiments on two neutral, one alkaline, and one slightly acid soil, respectively, over two seasons, superphosphate and Rhenania phosphate proved the most efficient phosphatic fertilisers. Dicalcium phosphate was less, and basic slag least effective. The high availability of the Rhenania phosphate may be correlated with its physical condition.

G. W. ROBINSON.

**Adsorption capacity of soils.** D. J. HISSINK (Chem. Weekblad, 1926, 23, 511—515).—The view of adsorption as an exchange of cations between the solution and the soil, and the significance of the replacement of calcium and magnesium in the soil by sodium and potassium from the solution, are discussed. The following values for soil analysis are proposed:—(1) The milligram-equivalent of replaceable base in 100 g. of clay ( $S$  clay) and in 100 g. of humus ( $S$  humus); (2) saturation level of the soil,  $V$ , i.e., ratio of replaceable bases to the total quantity the soil can bind,  $T$ ; thus  $V = 100 S/T$ ; (3) lime factor ( $Kf$ ), the quantity of lime which the humus must adsorb to give a neutral reaction ( $p_H = 7$ ), and the relations between them considered. From the values of  $T$ , the average equivalent weights of clay and humus, respectively, are given as 1225 and 175. The relation between the amount of lime necessary to flocculate a soil suspension, the content of clay and humus, and the saturation level  $V$  is discussed.

S. I. LEVY.

**What happens to the lime when soil is limed?** D. J. HISSINK (Proc. 2nd Comm. Int. Soc. Soil Sci., 1926, A, 174—197; Chem. Abstr., 1926, 20, 3204).—The following data were obtained for experimental fields:  $p_H$ ,  $K$  (humus) = g. humus — lime per 100 g. of humus,  $K$  (clay),  $V$  = degree of saturation, and the lime remaining as calcium carbonate in the soil. Four new values are employed: (1) Actual adsorption, i.e., g. of lime adsorbed by the clay-humus substance per 100 g. of soil. (2) Potential adsorption, i.e., g. of lime that the clay-humus substance per 100 g. of soil can take up under the prevailing climatic conditions, calculated (for

Dutch conditions) on the assumption that the highest values of  $K$  are 5.2 (humus) and 1.1 (clay). (3) Coefficient of utilisation of the lime applied, i.e., the actual adsorption as a percentage of the potential adsorption. (4) Liming ratio, i.e., the ratio of g. of lime applied per 100 g. of soil to the potential adsorption. The largest possible actual adsorption in the shortest possible time with the smallest possible liming ratio is desirable. The actual adsorption will be the greater the finer-grained is the lime and the more thoroughly it is mixed with the soil immediately after liming. Soils with low  $K$  and  $V$  values adsorbed practically all the lime as clay-humus lime within about 1 yr., little remaining as calcium carbonate. The  $K$  and  $V$  values are predominant factors in determining the actual adsorption.

A. A. ELDRIDGE.

**Relation between the  $p_H$  value, degree of saturation, and humus of some humus soils. Equivalent weight of the humus substance.** D. J. HISSINK (Proc. 2nd Comm. Intern. Soc. Soil Sci., 1926, A, 198).—For 15 soils with humus content 6.7—50%, the  $p_H$  varies from 3.3 to 7.0, the degree of saturation ( $V$ ) from 0.09 to 36.0, and the  $S$  (humus) from 6 to 214. Average values at  $p_H 7$  were  $V = 35.8$ ;  $S$  (humus) = 206. The average equivalent weight of the humus substance is 176.

A. A. ELDRIDGE.

**Systematic methods for the recognition of plant extracts.** GRIMME.—See XX.

#### PATENTS.

**Process for preparing soil.** A. WIDDIS (U.S.P. 1,608,917, 30.11.26. Appl., 17.12.24).—A layer of an emulsion of bentonite is placed at the bottom of the soil, previously loosened to a point below the usual depth of the plough.

H. ROYAL-DAWSON.

**Manufacturing colloidal phosphate fertilisers.** W. DE HAËN (U.S.P. 1,607,666, 23.11.26. Appl., 20.1.25. Conv., 16.7.21).—Phosphatic material is treated with humus colloids at a low temperature and with moderate grinding in the presence of water.

H. ROYAL-DAWSON.

**Ammonium salt [sulphate for fertiliser].** E. W. HARVEY (U.S.P. 1,608,738, 30.11.26. Appl., 16.8.22. Renewed 5.10.25).—The condition of commercial ammonium sulphate is improved by the addition of a mixture of a basic calcium compound and a semihydrated calcium sulphate.

H. ROYAL-DAWSON.

**Methods of making plant foods.** H. F. BLACKWELL (U.S.P. 1,606,015, 9.11.26. Appl., 21.4.22).—A basic plant food element unavailable to plants is added to vegetable matter and decomposed by means of bacteria forming humic acid, which combines with the basic element forming a water-soluble humate which is recovered.

H. ROYAL-DAWSON.

**Dressing seeds.** FARBERWERKE VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 241,568, 14.10.25. Conv., 15.10.24).—The emission of dust caused by using dry fungicide composition in dressing seeds is avoided by the addition of 3% of paraffin oil to such composition consisting of 30 pts. of copper carbonate, 30 pts. of a resin sulphate, 5 pts. of phenylarsinic acid, and 32 pts. of kieselsol.

F. G. CROSSE.



Controlling the temperature of heated air in drying and like operations [for crops etc.] (E.P. 261,446).—See I.

Composition for exterminating weeds (E.P. 261,241).—See XXIII.

## XVII.—SUGARS; STARCHES; GUMS.

Apparatus for the determination of moisture [in sugar] by distillation with xylene. PRITZKER and JUNGKUNZ.—See I.

Acid in potato flour and syrup. TRYLLER.—See XIX.

### PATENTS.

Production of a cannery syrup for the canning and preserving of fruits and the products thereof. E. S. COCHRAN, Assr. to J. T. McCROSSON (U.S.P. 1,606,827, 16.11.26. Appl., 19.8.24).—Without preliminary liming, the raw juice from the sugar cane or beetroot is strained through plate strainers and forced at a pressure of 20–30 lb./sq. in. through a closed heater at 104–110°. While under pressure it is filtered and further refined by the addition of 0.5% of porous infusorial earth and 1% of bone black, followed by a second filtration. The clear syrup, which contains the bulk of the sucrose and of the reducing sugars of the raw juice, is then concentrated. F. R. ENNOS.

Mixing methods and apparatus [for liming sugar juices] (E.P. 247,541).—See I.

Filtering device [for sugar solutions] (E.P. 261,204).—See I.

## XVIII.—FERMENTATION INDUSTRIES.

Wort composition. H. L. HIND and N. C. BEETLESTONE (J. Inst. Brew., 1926, 32, 540–551).—The increase in extract gained by acid addition is considerable when an alkaline mashing liquor is used. A greater extract results from the addition of vinegar than from sulphuric acid, but diastatic action is favoured more by the sulphuric acid and a greater proportion of reducing sugars is produced than when vinegar is added. In general, an unusually high proportion of buffer substances is undesirable, but the converse must not be pushed to extremes. Excess of buffers in the wort acting between  $p_H$  5.5 and 4.0, by tending to inhibit the increase of acidity during fermentation, favours an unstable beer. Admixture of raw grain with malt containing excessive nitrogen increases the stability of the beer by producing a wort containing less buffer substances than an all-malt wort, and consequently does not hinder to the same extent the increasing production of acidity during fermentation. As sparging continues and as the worts become weaker the concentration of buffer substances decreases, whereas the relative proportion of buffers as compared to fermentable extract from the goods increases and a point may be reached when the disadvantages from excessive buffering may overbalance any advantage realised by further extraction of fermentable matter. C. RANKEN.

Yeast maltase. V. J. JSAIEV (J. Inst. Brew., 1926, 32, 552–573).—Maltase is most active at a  $p_H$  of 6.1–6.7, the optimum  $p_H$  being independent of the temperature. The optimum temperature for its activity is 30–35°, and the temperature coefficient is 1.26. Even

under aseptic conditions, its activity is greatly reduced by alcohol, acetone, and water, but is preserved better if kept in sterile solution in concentrated glycerol. Provided the period of action is short, the activity of maltase inhibited by contact with acids or alkalis may be restored. A more prolonged action of the acids or alkalis is followed by the destruction of the maltase. With low concentrations of maltase, the velocity of the hydrolysis of maltose is directly proportional to its amount, but proportional to the square root of the amount at higher concentrations. Dextrose and laevulose retard hydrolysis, whereas galactose has no effect. The mechanism of the reaction cannot be expressed by means of a single mathematical equation. Although maltose and dextrose form readily dissociated compounds with maltase, the affinity of the latter to maltose is much weaker than to dextrose. The enzymic hydrolysis of maltose is a genuine reversible reaction, and reaches an equilibrium at 80–85% of hydrolysed maltose. Maltose, revertose, and gentiobiose are the products of the action of yeast extract on dextrose. C. RANKEN.

Examination of very old cold-stored hops. A. C. CHAPMAN (J. Inst. Brew., 1926, 32, 574).—Alameda (Californian) hops of the 1896 growth and placed in the cold store in Feb., 1897, gave on analysis in Dec., 1926, the following results: moisture 12.05%, soft resins 10.0%, hard resins 8.8%, and tannin 2.67%. The preservative power determined by the Chapman biological method (cf. B., 1925, 185) and by the lead precipitation method of Ford and Tait (cf. B., 1924, 568) gave the values 26 and 32, respectively. The  $\alpha$ -acid had completely disappeared and the whole of the preservative property was resident in the  $\beta$ -resin. C. RANKEN.

Vitamin content of grapes and grape wines. A. MERJANIAN (Z. Unters. Lebensm., 1926, 52, 307–311).—Bezssonov's vitamin test was applied to grape juice and grape products. Positive tests were given by grape juice and some wines. The vitamin content of wines appears to decrease with age. Wine yeasts contained no vitamin and the must only minute quantities. Traces of vitamin were detected in pure wine vinegar.

A. G. POLLARD.

Boiling point apparatus for determining strength of wines. G. FILAUDEAU (Ann. Falsif., 1926, 19, 531–536).—The Malligand ebullioscope gives satisfactory indications of the alcoholic strength of wines.

D. G. HEWER.

Determination of alcohol in spirits by distillation. E. FEDER and L. RATH (Z. Unters. Lebensm., 1926, 52, 292–294).—In the distillation process for the determination of alcohol, certain losses of alcohol occur when the liquid under examination has a high alcohol content. This was traced to the carrying over of uncondensed alcohol by expelled air during the initial warming up of the apparatus, and could only be trapped by immersing the end of the condenser in water. A simple method of arranging a pycnometer and funnel for this purpose was successfully adopted. Results obtained in this manner were approximately 0.1% higher than by the official (German) method, and were more closely in agreement with figures calculated from the density of the extract-free liquor (Berg).

A. G. POLLARD.



**Preparation of dilute alcohol and liquids of definite alcoholic strength, and methods of determining alcohol.** G. TELLERA (Atti R. Accad. Lincei, 1926, 65, 705—710).—Formulæ are given for calculating the proportions of water to be added to aqueous alcohol or to a liquor having a certain alcoholic content to reduce it to any required content. For the determination of alcohol, Chabot's method (B., 1926, 210; see also Widmark, A., 1922, ii, 789) is recommended and details of the procedure are given. T. H. POPE.

**Acetic acid and vinegar.** KREUTZ and BÜCHNER.—See XX.

#### PATENTS.

**Manufacture of bacterial preparations.** M. L. LANDMAN (E.P. 261,455, 7.8.25).—A solution of 1 pt. of agar-agar in 24 pts. of water is gradually mixed with an equal volume of heavy mineral oil. A milk or whey-broth culture of the bacilli is then stirred in until an emulsion is formed, with the oil as the continuous phase, and the aqueous solution and the culture the disperse phase (for *Bacillus acidophilus* the distribution in the final product should be about 350 million/c.c.).

F. R. ENNOS.

**Method of making plant foods** (U.S.P. 1,606,015).—See XVI.

**Manufacture of butter fat products** (U.S.P. 1,605,009).—See XIX.

### XIX.—FOODS.

**Colours in foodstuffs.** H. DRAKE-LAW (J.S.C.I., 1926, 45, 428—434 T).—The essential properties of food-stuff colours are described, and reference is made to the different causes of harmfulness. Toxicity, antisepticity, the irritating action on the skin are mentioned, and the dangerous limits of harmful metallic impurities are given. The various properties required for confectioners' colours are outlined, such as fastness to light, heat, acids, alkalis, sulphur dioxide, and metals. The methods used for testing are described. The essential properties of the best known colours are given in detail, and the uses to which they are applied are set forth, special attention being given to export trade. An extensive list of colours which are now used, or which have been used in the past, is shown, and the best known methods of identification of these are fully described. Consideration is given to the diluents commonly associated with confectioners' colours, and the method of testing for strength of diluted colours is outlined. The manner of determining the chief metallic impurities is explained, and reference is made to the many processes of analysis now in common use. The need for a standard method of analysis of all confectioners' products is emphasised, especially in connexion with export trade. Attention is directed to the growing necessity for the international standardisation of methods of analysis of the raw materials used in foodstuffs, especially in view of the more stringent foreign regulations now being imposed on imported goods.

**Influence of peptic digestion in the determination of total carbohydrates in cereal products.** B. G. HARTMANN and F. HILLIG (J. Assoc. Off. Agric. Chem., 1926, 9, 482—484).—In the case of products

containing large amounts of proteinaceous matter, the determination of carbohydrates by conversion into dextrose by means of diastase and acid hydrolysis is slow and not always reliable. By digesting the cereal with pepsin at room temperature overnight, boiling, cooling to 65°, and adding malt infusion, the conversion into dextrose is usually complete in 30 min.

F. R. ENNOS.

**Function of oils and fats, and emulsions of oil and water in breadmaking with special reference to gluten formation and modification.** E. B. BENNION (J.S.C.I., 1926, 45, 435—438 T).—Oil and emulsions of oil and water were used in gluten washing determination, and it was found that there is a distinct action, especially on grades of flour containing a high natural percentage of gluten, such as American flour and straight-run home-milled flours. Emulsions of oil in water increase the amount of gluten in the flours to a slightly less extent than oil itself. The colour of the glutes, dried in hot-air ovens at 105° to constant weight, which were obtained from doughs made up with emulsions of oil in water was of a much lighter character, being a creamy shade instead of dark brown as normally. A standard method of gluten determination is given which was employed in the research. Oil and emulsions of oil retarded fermentation to a slight extent as judged by cylinder tests. Baking tests were carried out, and the loaves containing the emulsion possessed the best coloured crumb and pile, the colour being whiter than the crumb of the loaf containing oil alone, and much whiter than the control loaf. Oil will keep the crumb of a loaf moist, but an emulsion is more effective. A table of moisture contents of loaves made by different methods is given. The use of emulsions in the manufacture of gluten bread is also considered.

**Differentiation between flours by means of iodine absorption and rate of sedimentation.** N. A. TROFIMUK (Z. Unters. Lebensm., 1926, 52, 311—318).—The nature of flours is definitely indicated by the iodine adsorption of their aqueous extracts. The filtered extracts are titrated with iodine solution in the presence of starch to match a standard colour. The cereal flours can readily be distinguished from each other by this means. A marked difference exists between the range of iodine absorption of flours from mono- and di-cotyledonous seeds. Somewhat similar differences occur in the rates of sedimentation of various flours. Measurements are made of the rates of increase of depth of sediment in a cylinder of flour suspension. Admixture of 10% of rye, of 15% of oat, or of 20% of barley flour to wheaten flour may be detected in this way.

A. G. POLLARD.

**[Phosphoric] acid in potato flour and syrup and its determination.** H. TRYLLER (Z. Spiritusind., 1926, 49, 342—343, 355—356, 365—366; cf. B., 1925, 221).—As contrasted with prima potato flour, no definite conclusions as to the acidity of the secondary and tertiary flours can be drawn from titration with sodium hydroxide and phenolphthalein and with hydrochloric acid and methyl orange. The acidity is greater than is accounted for by the phosphoric acid in the ash and is probably due to amino-acids derived from albuminous matter. Comparison of acidities is best made by determination



of the hydrogen-ion concentrations. The  $p_H$  value of a starch is the same as that of the liquid with which it is in equilibrium. For the prima flour the liquid used is a buffer solution consisting of 25 c.c. of a mixture of 6 pts. of 0.1M-disodium phosphate and 4 pts. of 0.1N-monopotassium phosphate diluted to 1 litre, but for the less pure secondary and tertiary flours distilled water only is required. The hydrogen-ion concentration is then determined in the liquid by the colorimetric or the electrometric method. In the case of the prima flour the  $p_H$  value may also be arrived at by titration with acid and alkali, all three methods giving concordant results. Comparison of the electrometric neutralisation curves of the prima flour with those of various acids, confirms the fact already established that the acid present is entirely phosphoric, and that it is not combined with starch as an ester. The curves for the secondary and tertiary flours show no similarity to that of phosphoric acid, but resemble either that of albumen from the sludge or of the amino-acid glycocoll. By determining the  $p_H$  values of successive wash waters it is possible to follow the course of the washing of starch free from acid, and to determine the exact point at which to stop to obtain a neutral product. The physico-chemical method is also applicable to the syrup. In order to get concordant  $p_H$  values it is necessary to work with solutions of the same concentration, approximating to 1 pt. of syrup in 2–4 pts. of water. F. R. ENNOS.

**Determination of fat in malted milk.** E. S. ROSE (Amer. J. Pharm., 1926, 98, 595–596).—A modified Werner-Schmid method in which a mixture of 2 pts. of "benzin U.S.P." and 1 pt. of washed ether is used as the extraction medium. F. R. ENNOS.

**Preservation of milk samples with formaldehyde and trioxymethylene.** LETOURNEUR-HUGON and VALIN (Ann. Falsif., 1926, 19, 544–547).—Two drops of 40% formaldehyde and about 1 g. of trioxymethylene are used to preserve about 250 c.c. of milk with more satisfactory results than with dichromate, and with entire elimination of gaseous fermentation. Such preserved samples may be analysed by ordinary methods, except that lactose should be determined polarimetrically and not by Fehling's solution. In using the Gerber method for fat it is necessary to heat at 70–80° on a water bath for some time before centrifuging to ensure re-solution of the coagulum. The Adam method is not suitable. D. G. HEWER.

**Physico-chemical basis of the density of milk.** G. KOESTLER (Z. Unters. Lebensm., 1926, 52, 279–287).—The density of milk can be represented as a mathematical summation, based on the densities of its constituents determined in the dispersed colloidal condition. Methods for determining these values are outlined. Mean values recorded are:  $d_{15}^{15}$  for fat, 0.920; dicalcium caseinate, 1.450; lactose, 1.607; salts, 2.619. Using these values, calculated and experimental values for the densities of a number of milk samples were in much closer agreement than when the usually accepted values of Fleischmann were employed. For a sample of "pathological" milk, calculated and observed values were more divergent. A. G. POLLARD.

**Determination of the freshness of eggs.** E. DINSLAGE and O. WINDHAUSEN (Z. Unters. Lebensm.,

1926, 52, 288–291).—Alterations in the weight and density of eggs with time depend upon the method of storage, temperature, and moisture content of the surrounding atmosphere. The rate of decrease of density of eggs with age is lowered by increased moisture content of the surrounding air. In the absence of information as to method of storing and the original density of the egg, it is impossible to decide its age with any reasonable degree of accuracy.

A. G. POLLARD.

**Specific characters of regenerated preserved peas.** J. FROIDEVAUX (Ann. Falsif., 1926, 19, 536–544).

—The proportion of the water-insoluble portion of dried peas was found to be unaffected by the temperature of sterilisation employed, and by the method of preparation previous to boxing. The commercial value of preserved peas is inversely proportional to the amount of desiccation before boxing, and quality is higher the lower the nitrogen and higher the water content in the insoluble portion. D. G. HEWER.

**Composition and nutritive value of pumpkin-seed cake.** A. ZAITSCHEK and E. JALOWETZ (Kisérlet. Közlem., 1924, 27, 26–31).—Low-grade cake contained 41% of crude fibre (8% digestible); high-grade, 21% (none digestible). A large number of samples contained 40–50% of protein, 11–30% of fat, and had a starch value of approximately 90%. A. A. ELDRIDGE.

**Vitamin-containing food preparations and their content of anti-beri-beri vitamin.** E. KOMM (Z. Unters. Lebensm., 1926, 52, 303–307).—Animal nutrition experiments are described indicating the presence of vitamin-B in a number of proprietary food preparations. A. G. POLLARD.

**Viscosity of pectin sols.** A. OHN (Ind. Eng. Chem., 1926, 18, 1295–1298).—A pectin preparation obtained by alcoholic precipitation from a water extract of the white inner rind of oranges turns yellow at 155°, decomp. 158°, has  $d_{20}^{20}$  1.180, and is precipitated by soluble copper and lead salts. The relative viscosity of sols made from this preparation, citric acid, cane sugar, and distilled water depends upon the proportions of these constituents, but, within limits and when the hydrogen-ion concentration is sufficient, an increase in the amount of pectin or sugar gives a noticeable increase in relative viscosity. If the pectin is deficient, increase in the other factors gives no appreciable change in the relative viscosity. The optimum ratio for a good jelly is 0.40 g. of pectin and 62.5 g. of sugar per 100 c.c. at  $p_H$  2.60, the relative viscosity of this sol at 103° then being 0.560, compared with a glycerol standard ( $d_{20}^{20}$  1.220) at 60°. There is a large increase in relative viscosity below 90° of sols having sufficient pectin, acid, and sugar, this increase being greater below 70°. Within limits the surface tension of these sols varies inversely as the viscosity, this variation depending mainly on the hydrogen-ion concentration. E. HOLMES.

**Apparatus for the determination of moisture [in butter] by distillation with xylene.** PRITZKER and JUNGKUNZ.—See I.

**Lacquers for preserve cans.** SERGER.—See XIII.

**Acetic acid and vinegar.** KREUTZ and BÜCHNER.—See XX.



**Citrus flavours.** SCHUETTE and DOMOGALLA.—See XII.

#### PATENTS.

**Process of producing edible fat.** G. GRINDROD, Assr. to CARNATION MILK PRODUCTS Co. (U.S.P. 1,605,108, 2.11.26. Appl., 12.5.19).—Edible vegetable fats are hydrogenated, in carefully-regulated stages, to remove toxic constituents and substances likely to produce rancidity, and to avoid hardening of the fat. The material is first hydrogenated in the presence of a catalyst at 70° to decomposed aldehydes and ketones, and then under 250—600 lb. pressure at 90° to decompose the hydroxy-fats. F. R. ENNOS.

**Manufacture of butter fat products.** T. A. SPAETH, Assr. to MATTHEWS SELECTED DAIRIES Co. (U.S.P. 1,605,009, 2.11.26. Appl., 1.5.26).—Sweet cream is mixed with a lactic culture at about 24°, and an acidity of approximately 0.9% of lactic acid allowed to develop. A smaller quantity of a solution of gelatin in milk is mixed with the acidified cream and the whole is left in the cool for about 12 hrs. No further souring will occur for an extended period. F. R. ENNOS.

**Preservation of eggs.** A. J. BELLAMY, and A. BELLAMY & Co., LTD. (E.P. 261,194, 13.5.25).—After removal of the shells, the remainder or the yolk only is mixed with 2½—15% of glycerin, and the mixture concentrated in shallow trays by exposure to dry air until the pasty residue contains not more than 40% by weight of moisture. F. R. ENNOS.

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

**Identification of acetic acid in lavender oil.** P. LANGLAIS and J. GOBY (Perf. Essent. Oil Rec., 1926, 17, 520—521).—Examination of a blended lavender oil ( $d$  0.892,  $\alpha_D$  -6° 20', ester number 121.42, esters calculated as linalyl acetate 42.5%) has shown that, contrary to the statements of Dalton (*ibid.*, 432—3, 476—7), the predominating acid is acetic acid. By incomplete steam-distillation of the acidified potassium salts obtained on saponification, acetic acid corresponding to 9.2% of the oil was isolated and identified by conversion into acetanilide, *p*-bromoacetanilide, and ethyl acetate. E. H. SHARPLES.

**Acetic acid and vinegars containing formic acid.** A. KREUTZ and C. BÜCHNER (Z. Unters. Lebensm., 1926, 52, 295—298).—The popular prejudice against synthetic vinegars containing acetic acid prepared by the carbide process is unfounded. The amount of formic acid present in such vinegars is too small to be injurious. Determination of formic acid in vinegar by the reduction of permanganate is not indicative of the source of the vinegar. Many artificial vinegars are coloured with burnt sugar. The latter contains reducing substances which render the permanganate test for formic acid valueless. A. G. POLLARD.

**Detection of alcohol adulterants.** J. M. HALEY (Ind. Eng. Chem., 1926, 18, 1312—1313).—Detailed methods for the rapid but certain detection of methyl alcohol, acetone, benzene, alkaloids, phenol, and ethyl phthalate are given, whereby the complete test for these six adulterants may be carried out in 1 hr. E. HOLMES.

**Furfuraldehyde in industry.** D. H. KILLEFFRE (Ind. Eng. Chem., 1926, 18, 1217—1219).—A general account of the transition of furfuraldehyde from a laboratory curiosity to an industrial raw material, of its present and possible uses, together with a detailed description of the present commercial method of preparation. E. HOLMES.

**Colour reaction of saccharin.** L. EKKERT (Pharm. Zentr., 1926, 67, 821—822).—The known colour reactions of saccharin are summarised, and the following additional reaction is described. To 0.02—0.05 g. of saccharin in 1—2 c.c. of water are added 3—6 drops of 30% hydrogen peroxide solution, followed by 0.01—0.04 g. of sodium nitrite, and the mixture is well shaken. The solution first acquires a yellow colour, which is changed on addition of alkali into a vivid brown-red. The reaction is also given by other phenols, such as resorcinol, and by morphine, but these substances give a yellow solution on treatment with sodium nitrite and dilute sulphuric acid, whilst saccharin under the same conditions yields a colourless solution. W. J. POWELL.

**[Assay of] *Cascara sagrada*.** J. B. BERARDI and M. C. CANAN (J. Amer. Pharm. Assoc., 1926, 15, 189—193).—On extraction of fluid extracts of cascara, aloes, frangula, or rhubarb with ethyl ether or ethyl acetate, the residues were inert; the colours produced by the extracts with ammonia were not characteristic. Of two colour-reacting constituents in *Cascara sagrada*, one, insoluble in water, gives a deep cherry-red colour with ammonia; the other, soluble in water, gives an orange-brown colour. The non-bitter principle is soluble in water and is therapeutically active. The bitter principle is soluble in water, accompanies or is the brown-colour-reacting constituent and accompanies or is part of the therapeutically active principle, probably an anthraquinone derivative. A. A. ELDRIDGE.

**Industrial conversion of coal into organic products of technical use.** PATART.—See II.

#### PATENTS.

**Manufacture of solvents [from petroleum hydrocarbons].** C. P. BYRNES (E.P. 259,293, 6.7.25).—The partial oxidation products of petroleum hydrocarbons, obtained as in E.P. 138,113 (B., 1921, 636), 173,750 (B., 1923, 818), and 209,128 (B., 1924, 210), are extracted with mixtures of alcohol and water in such proportions that the more highly oxidised components (esters, aldehydes, and ketones) are removed. The alcohol is distilled off, preferably after dehydrating the solution with, e.g., anhydrous sodium sulphate, and the residual oil is fractionated. The fractions boiling from 100° to about 250° are suitable solvents for nitrocellulose and varnish gums in the preparation of lacquers and lacquer paints. Any free acids present in the partially oxidised oils may be esterified, before the extraction with aqueous alcohol, by addition of a suitable catalyst, such as hydrogen chloride. Again, all or part of the aldehydes or ketones may be removed from the alcoholic solution by extraction with sodium bisulphite. A. DAVIDSON.

**Vanadium compound [of 8-hydroxyquinoline].** H. HAHN and W. KROPP, Assrs. to WINTHROP CHEM. Co., INC. (U.S.P. 1,607,196, 16.11.26. Appl., 25.9.25. Conv.



24.12.24).—8-Hydroxyquinoline (30 pts.) and vanadium trioxide (10 pts.) are heated with water (300 pts.), and the brownish-green powder obtained is washed, and dried. It has the probable formula  $V(OC_6H_4N)_3$  and is of therapeutic value in the treatment of spirochaetal diseases. Other oxides of vanadium and derivatives of 8-hydroxyquinoline can also be employed.

T. S. WHEELER.

**Manufacture of acyl halides.** BRIT. DYESTUFFS CORP., LTD., and S. COFFEY (E.P. 261,240, 15.3.26).—Acyl halides are prepared pure and in good yield by treating a mixture of fatty acid and anhydride with a phosphorus trihalide. No hydrogen halide is formed. *E.g.*, a mixture of 27.3 pts. of 100% acetic acid, 18.7 pts. of acetic anhydride, and 35.5 pts. of phosphorus trichloride is warmed. An 85% yield of pure acetyl chloride distills over.

B. FULLMAN.

**Disinfectant, antiseptic, and medicament.** RHENANIA VEREIN CHEM. FABR. A.-G., Assees. of O. GERNGROSS and K. RÜLKE (U.S.P. 1,606,359, 9.11.26. Appl., 2.1.25. Conv., 28.1.24).—The disinfectant properties of heavy metal salts, such as mercuric chloride, mercuric thiocyanate, or silver cyanide, are greatly increased by the addition of a large excess of an alkali or alkaline-earth thiocyanate. Suitable mixtures, of special value in disinfecting albuminoid matter, comprise mercuric thiocyanate (1 pt.) and potassium thiocyanate (124 pts.), or potassium silver cyanide (1 pt.) and potassium thiocyanate (99 pts.), each mixture being used in about 3% solutions.

T. S. WHEELER.

**Making *m*-nitro-*p*-hydroxyphenylarsinic acid.** I. OSTROMISLENSKY, Assr. to OSTRO PRODUCTS CORP. (U.S.P. 1,607,299, 16.11.26. Appl., 22.7.22).—A mixture of arsanilic acid (*p*-aminophenylarsinic acid; 250 g.), nitric acid *d* 1.3 (275 c.c.), and water (750 c.c.) is treated with sodium nitrite (133 g.), the temperature being maintained at 70°. The mixture is then heated near its b.p. under reflux for 6–9 hrs. Diazotisation, replacement of the diazo-group by hydroxyl, and nitration successively occur, and on cooling *m*-nitro-*p*-hydroxyphenylarsinic acid separates in 80% yield. It can be purified by dissolving in boiling 15% sodium hydroxide solution, and precipitating the sodium salt with ethyl alcohol.

T. S. WHEELER.

**Treating tobacco.** J. W. KELLAND (E.P. 261,520, 16.4.26).—Tobacco waste (stalk, offal, etc.) is softened, pulped, and kneaded until the fibres are reduced to about 1 mm. in length. The pulp is then made into sheets or slabs, 33% acetic acid solution being added for preservative purposes if necessary.

B. FULLMAN.

**Manufacture of bacterial preparations** (E.P. 261,455).—See XVIII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

### PATENTS.

**Reducing photographic negatives, positives, films, bromide, and gaslight prints.** K. E. TAESCHNER (E.P. 261,201, 7.12.25).—Reduction is effected by bathing the image in a solution of iodine and potassium iodide, and either simultaneously or previously, with a solution of alkali chromate or dichromate. The reducing solution may also contain sugar. Tanning of

the gelatin in the vicinity of the silver by the chromate or dichromate protects the lighter tones from the action of the iodine.

W. CLARK.

**Photographic papers for making prints from tracings.** H. KAMMERER (E.P. 252,012, 7.5.26. Conv., 7.5.25).—Light-sensitive papers producing positive pictures directly by the action of aromatic amines, phenols, or aminophenols, are produced by coating with a chromate or dichromate solution containing heavy metal salts of organic acids, especially ferric salts such as ferric ammonium citrate, oxalate, or tartrate, to which may be added a salt of manganese, or mixtures of such salts. The amines etc. required for development may be applied to the paper before exposure in a dry state or in solution in spirit, development being by means of a damp sponge, or by steam. Paper prepared in the manner described keeps much better than that usually prepared for the aniline process.

W. CLARK.

**Manufacture of diffraction gratings for spectroscopic, optical, and like purposes.** K. KONDO (E.P. 262,020, 29.7.26).—A replica of a diffraction grating is obtained by pressing the original against a plane dry plate coated with bichromated gelatin, suitably exposing to light, developing the plate in hot water, and silvering. An original plane grating is reproduced on a concave plate in the above manner by employing a parallel beam of light. Gratings suitable for investigating the ultra-violet region are made by electrolytically depositing nickel on the silver coating of concave gratings prepared as above.

A. COUSEN.

**X-Ray screens.** H. S. SAWFORD (E.P. 255,831, 15.6.26. Conv., 24.7.25).

## XXII.—EXPLOSIVES; MATCHES.

**Nitrometric determination of tetryl.** L. DESVERGNES (Ann. Chim. Analyt., 1926, [ii], 8, 353–357).—Tests of Lehmstedt's method for the determination of tetryl by measuring the nitric oxide evolved on reduction with mercury and sulphuric acid (B., 1926, 388) always gave very low results when pure tetryl was used. Tetryl is insoluble in cold 90–100% sulphuric acid, and the reduction of 0.3 g. requires at least 4 hrs., whilst at 120° partial decomposition takes place with the formation of trinitrophenol, ammonia, and carbon dioxide. 2:4:6-Trinitrophenylethylnitroamine behaves in an analogous manner, so that the method is untrustworthy for the determination of either compound.

A. R. POWELL.

## XXIII.—SANITATION; WATER PURIFICATION.

**Purification of sewage by activated sludge at Essen-Rellinghausen.** F. SIERP (Z. angew. Chem., 1926, 39, 1521–1534).—A detailed account is given of experiments started in 1912 which have led to the installation of an activated sludge plant for a sewage containing acid waste. It was found that mechanical agitation was more effective and less costly than aeration alone, as the former brings the sludge more into contact with the sewage and the colloid-absorbing properties of the sludge can be utilised to the greatest advantage. Aerobic conditions, however, are essential and now aeration is combined with agitation, the air being introduced through filter plates as a fine blast, this having been found more effective than through perforated lead pipes. The speed



of agitation is important; it must be fast enough for the sludge to be broken up, but not so fast that the sludge does not settle to the bottom and so escapes the action of the air. Paddles rotating 7 times per minute are found to be most effective. For good working of the process the condition of the sludge must be watched so that its flocculating properties are utilised, and the extent of oxidation of the effluent must be tested. Great importance is attached to the breaking of the surface film by agitation to ensure direct absorption of the air. The working of the plant is also watched by determining the "oxygen consumed," by noting the transparency of the effluent, and by the free-ammonia decrease, and ratio of nitrite to nitrate. Recently, hoods have been provided for collecting the gas evolved, which is subsequently burnt.

W. G. CAREY.

**Biochemical oxygen demand of raw and treated sewage.** C. E. KEEPER and R. T. REGESTER (Eng. News-Rec., 1926, 97, 870—873).—A study of several thousand Baltimore sewage analyses to ascertain the relationship existing between the biochemical oxygen demand and the solids in raw and treated sewage collected in a separate system of sewers. Generally, the oxygen demand of the sewage is low during the winter and gradually increases until the summer months. The oxygen demand of sewage and sedimentation tank effluent increases with the suspended and settling solids, but, in the case of the former, the rate of increase is less as the amount of solids becomes greater. One explanation for this condition is that as the solids in the sewage increase, the relative amount of inorganic to organic material is greater. Settling tanks lower the oxygen demand about as much as the trickling filters; during 1919—25, the settling tanks reduced the oxygen demand from 174 to 104 p.p.m., and the trickling filters from 104 to 29 p.p.m., or 70 and 75 p.p.m., respectively. 40.3% of the oxygen demand is removed by settling tanks, 43% by trickling filters, and 0.2% by humus tanks. Such figures, however, do not give a complete and comparative picture of the rôle played by the three treatment units in purifying sewage, the trickling filters being considerably more efficacious than the settling tanks, in that large volumes of dissolved and nitrate oxygen are added to the sewage. A study, having regard to the change in oxygen content as well as to the reduction in oxygen demand, of the comparative efficiencies of the settling tanks, trickling filters, and humus tanks of eleven sewage works, showed the comparative efficiencies to be 33.5%, 62.3%, and 4.2%, respectively.

W. T. LOCKETT.

**Biochemical oxygen demand test.** R. E. GREENFIELD, A. L. ELDER, and R. E. McMURRAY (Ind. Eng. Chem., 1926, 18, 1276—1279).—During the absorption of oxygen at 2° by sewage diluted with distilled water the bacterial count first increases slowly, the absorption also being very slow (B., 1926, 422), then both increase much more rapidly. If the sewage is initially heavily inoculated with the culture of the organisms the period of slow absorption is absent, so that it seems that, as previously suggested (*loc. cit.*), the lag period in the absorption is due to the slowness of the development, at the prevailing low temperature, of the bacteria effecting deoxygenation. Polluted stream water yields absorption curves showing a second period of acceleration,

which does not appear, however, to be due to the death of plankton organisms alive at the commencement of the absorption, since it is not eliminated by destroying the plankton by heat at the outset. Experiments with sewage diluted at  $p_H$  7.7 with water containing inorganic salts gave two-stage absorption curves, and a much increased oxygen demand. In the second period nitrification, which hardly occurs at all in absence of inorganic salts, was rapid and extensive. Reduction of the  $p_H$  of the diluted sewage to 6.4 decreased the oxygen demand, but a rise to 8.1 had little effect.

R. CUTHILL.

**Effect of hydrogen-ion concentration in revivifying zeolites.** O. R. SWEENEY and R. RILEY (Ind. Eng. Chem., 1926, 18, 1214—1216).—The base-exchange capacity of zeolites used for water-softening is nearly always influenced—usually adversely—to a considerable extent by the presence of traces of acid or alkali in the sodium chloride solution used for revivification. This phenomenon does not appear, however, to be due to a solvent action. The  $p_H$  of the water being softened also seems to be of considerable importance. R. CUTHILL.

#### PATENTS.

**Compositions for exterminating insects and weeds.** GRAESSER-MONSANTO CHEMICAL WORKS, LTD., and H. MAXWELL-LEFROY (E.P. 261,241, 14.5.25).—Insects, flies, fly larvæ, and weeds in ships, houses, gardens, etc., may be exterminated by direct exposure to a composition prepared by emulsifying monochloronaphthalene (or monobromonaphthalene) with water with the aid of an agent, *e.g.*, soft soap. For the extermination of insects in fabrics a 10% solution of monochloronaphthalene in benzene may be used, for insects on ponds or solid surfaces, a 10% solution of monochloronaphthalene in a paraffin hydrocarbon of high flash point, and for insects on sheep, a mixture composed of monochloronaphthalene (10 pts.) and ordinary sheep dip (90 pts.), or a spray consisting of a mixture of monochloronaphthalene and a soothing oil, *e.g.*, cottonseed oil.

W. T. LOCKETT.

**Extermination of insects.** GRAESSER-MONSANTO CHEMICAL WORKS, LTD., and H. MAXWELL-LEFROY (E.P. 261,055, 14.5.25).—A lethal concentration of monochloronaphthalene or monobromonaphthalene is produced locally or within the space to be treated, by volatilisation of the compound by heat from a solid body, *e.g.*, kieselguhr impregnated with the liquid, or by atomising a mixture of the monohalogenated naphthalene and a hydrocarbon oil of high flash point. The monohalogenated naphthalenes are specific poisons for insects, and especially flies and fly larvæ, even at such low concentrations as one part by volume per million parts of air.

W. T. LOCKETT.

**Method of fumigation [with hydrogen cyanide].** W. S. LANDIS and G. H. BUCHANAN, Assrs. to AMERICAN CYANAMID Co. (U.S.P. 1,607,261, 16.11.26. Appl., 8.7.21).—The removal of hydrogen cyanide from a space which it has been employed to fumigate, is accomplished by injecting into it a mixture of formaldehyde and steam.

T. S. WHEELER.

**Method and apparatus for treating refuse.** F. C. EVANS (E.P. 261,119, 24.8.25).

**Disinfectant, antiseptic, and medicament** (U.S.P. 1,606,359).—See XX.