# BRITISH CHEMICAL ABSTRACTS

# **B.**—APPLIED CHEMISTRY

JUNE 24, 1927

## I.-GENERAL; PLANT; MACHINERY.

Mechanical properties of moist granular solids. P. G. NUTTING (J. Washington Acad. Sci., 1927, 17, 185—191).—A theoretical consideration, preliminary to experimental work, of the mechanical properties of moist granular solids with particular reference to moulding sand. It is shown that the forces associated with the merging of the adsorbed water films on small grains at the points of contact are sufficient to account for the order of magnitude of the observed mechanical rigidity, and that the cohesional force increases with the amount of water present up to a maximum, and diminishes to zero corresponding with complete wetting, in qualitative agreement with experiment. R. W. LUNT.

Accuracy of mechanical analysis. C. K. WENT-WORTH (Amer. J. Sci., 1927, 13, 399—408).—The principal errors in the analysis of sediments by sifting are due to variations in the time of sifting and in the composition of samples divided in different ways, and to the repeated sifting of the same sample. The error due to the second cause is about seven times as great as that due to the third, but of the same order as the time error at 1 hr. of shaking. For comparative purposes, shaking periods of 5—10 min. are sufficient, but for accurate analyses the time must be greatly increased, and should be standardised for any one series. H. F. GILLBE.

#### PATENTS.

Rotary furnace. C. B. WISNER (E.P. 246,118, 29.12.25. Conv., 13.1.25).—A tubular rotating furnace which is to be heated outside its full length is provided with alternate stationary and rotating jacket sections, which form with the inner tube of the furnace an annular heating space. The tyres and gearing of the furnace are placed outside the rotating sections, and the joints between the fixed and rotating parts are made of thin overlapping strips of metal in many plies.

## B. M. VENABLES.

Furnace [walls]. E. G. BAILEY (E.P. 249,560, 22.3.26. Conv., 20.3.25).—A furnace wall is constructed of water tubes faced with refractory material, and by adjusting the thickness of the latter a judicious compromise may be made between low temperature with long life for the refractory material and high temperature with good combustion conditions; in general, the inner surface of the refractory should be just below the fusing point of the ash. Methods of attaching the refractory tiles are described. B. M. VENABLES.

Furnace settings. PLIBRICO JOINTLESS FIREBRICK Co., Assees. of I. S. PIETERS (E.P. 260,953, 17.8.26. Conv., 9.11.25).—A vessel such as an oil still is set over a furnace chamber, which has fireboxes or tunnels constructed within it, from which flame and products of combustion emerge in such a manner as not to impinge directly on the still through side openings, so that flames from one tunnel impinge on those from another, and through top openings with adjustable lids.

B. M. VENABLES.

Furnace. F. I. PEDERSEN (E.P. 269,397, 6.9.26).— In a furnace comprising a number of superposed combustion chambers or zones, a water heating pipe is embedded in the walls of the lower combustion spaces, and is continued through the flues above the combustion spaces. B. M. VENABLES.

Furnace. F. A. FAHRENWALD (U.S.P. 1,624,043, 12.4.27. Appl., 13.1.26).—The rabble arms of a roasting or other furnace are pivotted at their points of attachment to the driving shaft, and are secured in their working position by comparatively fragile means.

B. M. VENABLES.

Refrigerating machines for obtaining great differences in temperature. Soc. ANON. POUR L'Ex-PLOIT. DES PROC. M. LEBLANC-VICKERS (E.P. 259,921, 29.6.26. Conv., 14.10.25).-A refrigerating machine operating by the evaporation of water or other liquid in a vacuum is arranged with a series of evaporators fixed vertically over each other and a corresponding vertical series of condensers. The evaporator with the highest pressure and temperature is at the top, and the vapour is drawn from it by an ejector or compressor to the lowest condenser, and so on down the series of evaporators and up the series of condensers. The liquid in each series (refrigerant or cooling water, respectively) is sprayed successively in each vessel downwards of the series, and air in the condensers naturally works to the top one, owing to that having the lowest pressure, and can therefore be withdrawn at one point only. B. M. VENABLES.

Centrifugal grinding mills. B. SCHERBAUM, Assee. of A. KRAUSE (E.P. 257,947, 3.9.26. Conv., 3.9.25).—A single grinding ball is driven round within a ring, the diameter of the ball being greater than the radius of the ring. Several of these grinding elements may be arranged in line on one shaft, and when thus balanced, the shaft may be driven at a high speed, suitable for direct coupling to electric or other motors. Scraping or deflecting blades may be affixed to the rotor in the spaces between the rings, preferably with a sweep of larger diameter than the rings, also within the rings, at parts of the rotor not occupied by the balls.

B. M. VENABLES. Crushing and grinding machine. A. C. HAMEY (U.S.P. 1,627,506, 3.5.27. Appl., 13.2.26. Conv.,

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19.2.25).—A muller is rotated about a vertical shaft, and can also be reciprocated vertically. The feed is central, e.g., down the hollow shaft, and the fixed grinding plate has a central cone surrounded by one or more grooves corresponding to ridges in the muller.

#### B. M. VENABLES.

Grinding mill. J. B. C. SCHERBAUM (E.P. 266,009, 15.2.27. Conv., 15.2.26).—A grinding ring, provided with one or more loose balls within it, is oscillated, *e.g.*, by a crank and pitman, transversely to its axis, about a shaft parallel to its axis. By this means the ball is caused to run round the ring and effect grinding. Several rings may be arranged abreast in one casing with feed chutes and draught of air to remove the ground material. B. M. VENABLES.

Grinding mill. E. C. LOESCHE (E.P. 268,263, 29.12.26).—A shallow bowl is rotated on a vertical axis, and grinding rollers engage with it near its rim. The material is fed to the centre of the bowl, and works upwards and outwards by centrifugal force, passing under the rollers and flying off the edge of the bowl; the coarse is separated from the fine either by a screen surrounding the bowl, or by an air current passing up the annular space between the bowl and the fixed casing. The coarse material, which in either event drops under the bowl, is returned to its upper or grinding side, either by an external elevator or by scoops depending from holes in the bottom of the bowl near the axis.

#### B. M. VENABLES.

Grinding and separating apparatus. W. C. HOOVER, R. D. FRITZ, and E. F. CAHILL, ASSTS. to B. F. GOODRICH CO. (U.S.P. 1,626,466, 26.4.27. Appl., 21.11.23).—A gaseous current—bearing with it the material to be ground—is impelled through an enclosed grinding mill, and through a conduit which leads to the open end of a rotary screen through the meshes of which the gas and fine material pass, while the coarse material is worked back to the open (inlet) end of the screen by internal blades. B. M. VENABLES.

Rotary mixing machine. J. F. BURN and J. S. LANCASTER (E.P. 268,658, 28.9.26).—A bowl for, *e.g.*, a concrete mixer is formed with a stepped bottom or closed end, to promote perfect mixing.

#### B. M. VENABLES.

Coal and ore washing installations having a single ascending current. E. LAURENT (E.P. 254,693, 15.6.26. Conv., 4.7.25).—A coal-washing apparatus is provided on the supply side with a weir which forms a bed of waste rock, and the rough surface reduces the speed of arrival of the material. On the outlet side it is provided with an adjustable knife-edge for sending a middling product either into the waste or into the coal, or to a separate outlet for the middling alone.

#### B. M. VENABLES.

Dryer. CARBORUNDUM Co., LTD., Assees. of C. H. GREENWOOD (E.P. 260,956, 9.9.26. Conv., 9.11.25).---A rotary dryer which may be fired with oil fuel, for which a high combustion temperature is desirable, is constructed with a tunnel-like combustion chamber of silicon carbide in the bottom of a heating chamber which surrounds the shell of the dryer. The products of combustion, after losing the bulk of their heat in the latter chamber, may be conducted through the dryer. B. M. VENABLES.

Drying apparatus. K. MEISTER (E.P. 268,237, 11.10.26).—A drying apparatus for sticky substances such as brewers' grains comprises a heated feed conveyor, the early part of which is arranged to compress the material and squeeze water out, followed by a trough dryer provided with a false bottom comprising sickleshaped steam-heated elements, and with air heated by the steam jacket of the feed conveyor. The final drying is effected in a rotary cylinder dryer provided with longitudinal steam pipes inside, over which the material is showered by the rotation of the drum.

B. M. VENABLES.

Methods of handling material to be sintered. J. E. GREENAWALT (E.P. 265,533, 6.4.26. Conv., 5.2.26).—The charge car of a sintering machine of the Greenawalt type is provided with an inclined screen just below its outlet and a chute below the screen, so that the moist charge is separated into coarse and fine, and the coarse material rolls off the screen just ahead of the fine from the chute, and arrives at the sintering grate first, thus forming a first layer of coarse material upon which the fine will rest. B. M. VENABLES.

Process and apparatus for feeding solids and gases into reaction vessels under pressure. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 268,188, 12.6.26).—A number of auxiliary vessels are connected by pipes to the reaction vessel. An auxiliary vessel is cut off from the pressure, opened, filled with the powdered solid, closed, and a gas pressure rather higher than that in the reaction vessel applied which blows the solid into the reaction vessel. By using the auxiliary vessels in turn the feed may be more or less constant.

B. M. VENABLES.

Separation of minerals and other substances. T. M. DAVIDSON (E.P. 268,043, 21.9.25 and 27.3.26).— Finely-divided material is fed on to an endless inclined conveyor which is almost wholly submerged in water, and has a transverse vibratory motion imparted to it. The material is preferably fed first to an inclined tray, which is also subjected to vibration and is partially submerged. A partial separation of varying density is thus effected before deposit upon the travelling conveyor. The heavier particles passing upwardly on the conveyor may be finally washed with a water spray before discharge. C. A. KING.

Apparatus for separating solids from liquids. W. H. CAMPBELL (E.P. 268,051, 29.12.25).—An apparatus suitable for supporting brewers' grains in the wort, and discharging them after the wort has been drained off, comprises a number of superposed perforated plates, one end of each (except the lowest) is hinged so that it can be raised when charging the grains and used as a chute to distribute the grains over the whole surface of the plates. For discharging the grains the whole set of plates is tilted together and the grain discharged through an opening along one side of the bottom of the tank.

B. M. VENABLES.

Centrifugal separator. UNION A.-G. FÜR METALL-IND. (E.P. 262,096, 19.11.26. Conv., 30.11.25).— Liquid to be purified flows through a number of co-axial centrifugal chambers in succession, each one larger in diameter than the preceding, and the outlets (for liquid) from each being at about the smallest radius possible. Near the inner wall of the outermost chamber a co-axial cylindrical filter is arranged, and the liquid passes inwards through this, and is then deflected by helical guides behind the filter up to a final discharge neck or annular chamber which also contains a filter medium. The first filter keeps clean because collected solids are thrown off it by centrifugal force. B. M. VENABLES.

Method of mounting and driving centrifugal separating apparatus. F. GRIMBLE, M. N. CAIRD, and E. COOMBS (E.P. 269,218, 20.10.25).—The shaft of an underdriven centrifugal machine is provided with rigid radial bearings on each side of the driven worm at the bottom; above that is a universal joint, above that a radial bearing having a certain lateral freedom controlled by springs, and the bowl is attached to the shaft by means that allows a slight lateral oscillation.

B. M. VENABLES.

[Centrifugal] separation of liquid mixtures. J. L. TUFTS (U.S.P. 1,623,508, 5.4.27. Appl., 25.4.23). —The mixed liquid is admitted to the lower part and the heavier constituent overflows the top rim of a cage or bowl. The lighter constituent is collected from below an annular skimming ring by means of a scoop-shaped pipe also discharging over the top rim.

B. M. VENABLES.

Apparatus for purifying liquids centrifugally. W. ALEXANDER (E.P. 267,858, 11.12.26).—A mixture of oil and water or other liquids is formed into a vortex within a cylindrical vessel, the vortex being produced by admitting the mixture and/or exhausting the separated water at a suitable inclination produced either by sloping the pipes or by vanes. The separated oil is exhausted at the axis at the upper part, and the conduit may be provided with vanes giving an upward movement to the oil inside the vortex. The inlet for mixture is situated at the circumference near the top, and the outlet for water at some intermediate point (radially) at the bottom. If the last is merely a large central outlet, the vortex will be of the free or Rankine type.

B. M. VENABLES.

Emulsifying apparatus. J. McGougan and J. HUNTER (E.P. 267,681, 16.2.26).—The apparatus comprises a hemicylindrical vessel with hermetically closing lid positioned as the diameter of the cylinder and with an internal paddle positioned as a radius. The paddle is provided with a number of small apertures, and when it is oscillated by an external handle the mixture of liquids is forced through the apertures under pressure from side to side. B. M. VENABLES.

Production of permanent emulsions. P. LECHLER (E.P. 254,701, 22.6.26. Conv., 1.7.25).—An emulsion of two liquids, e.g., tar and water, is made in proportions that are known to emulsify easily, and are kept until the liquid has separated into two stable emulsions containing more and less tar. The former is drawn off for market, and the latter is used in the preparation of fresh emulsion. B. M. VENABLES.

Apparatus for deflocculating and emulsifying.

E. A. BUTLER, Assr. to COLLOIDAL EQUIPMENT CORP. (U.S.P. 1,624,037, 12.4.27. Appl., 30.4.25).—An emulsifying chamber has one fixed and one rotating wall, and on each are annular projections forming recesses which are inclined in opposite directions on opposite walls.

Manufacture of an emulsion for use in the disincrustation of, or removal of scale from, boilers, hot wells, condensers, and the like. FILTRATORS, LTD., and V. SAKS (E.P. 268,665, 13.10.25).—In the preparation of an anti-scale emulsion as described in E.P. 176,294 (B., 1922, 280A) by the action of live steam on, e.g., linseed residue, the mucilage or emulsion, instead of discharging direct to the boiler, is discharged through a thermostatic release valve (similar to a steam trap) at atmospheric pressure into the hot well or other desired place. B. M. VENABLES.

Liquor circulating means for apparatus wherein articles, materials, or substances are treated with liquids, or wherein liquids are mixed or treated. MELLOR, BROMLEY & CO., LTD., T. C. BROMLEY, and C. H. GREEN (E.P. 269,241, 12.1.26).—The dye vat or other vessel to be heated and/or agitated is provided with duplicate sets of air- and steam-jets adapted to swirl the liquid in either direction according to the set of jets used. Valves for changing the direction are automatically operated by a rocking device comprising either a pair of buckets which will overbalance either way when filled by a stream of water above the fulcrum or a paddle wheel in the vat turned by the swirl of the liquid which will lift and overbalance a weighted lever, through the medium of worm gear. B. M. VENABLES.

Method of treating one liquid with another. S. J. DICKEY, ASST. to GENERAL PETROLEUM CORP. OF CALIFORNIA (U.S.P. 1,625,195, 19.4.27. Appl., 14.5.24). —Oil is treated with a heavier liquid reagent in an apparatus comprising several closed compartments one above the other, in which the oil flows upwards and the reagent downwards; the latter collects on the bottom of each compartment and flows by gravity to the bottom of the next, but the oil is forced through jets in each compartment with sufficient velocity to effect mixing.

B. M. VENABLES.

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Fractional distillation. THERMAL INDUSTRIAL & CHEMICAL (T.I.C.) RESEARCH CO., LTD., and W. J. CHADDER (E.P. 269,090, 23.11.26).—The vapours from a primary still pass to a dephlegmator, the condensate from which drops to a secondary still underneath, in which a bath of molten metal is maintained at a temperature just below that at which the desired condensate boils. The vapour returns to the dephlegmator, and, together with the remainder of the original vapour. may pass to other dephlegmator-still units for collection of other fractions. B. M. VENABLES.

Distilling apparatus. J. H. BURLINGHAM, Assr. to TEXAS Co. (U.S.P. 1,626,223, 26.4.27. Appl., 18.10.21). —The level of the liquid in a still for inflammable liquids is determined by means of an indicator spaced from the still, and which only functions on depression of a trigger, both conducting passages from indicator to still being kept automatically closed. C. O. HARVEY.

B. M. VENABLES.

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Separating by vacuum distillation the most volatile constituents of a mixture of liquids. A. SCHMALENBACH (E.P. 269,052, 15.9.26. Conv., 9.6.26. Addn. to E.P. 244,736; B., 1926, 968).—The process described in the original patent is modified by the use of direct steam for distillation and a moderate vacuum. B. M. VENABLES.

Exchange of heat between gaseous, vapour-like, or liquid materials. MASCHINENFABE. ING. H. SIMMON (E.P. 252,373, 18.5.26. Conv., 19.5.25).—The apparatus comprises a rotor through which one medium is passed while the other medium passes between the rotor and a surrounding casing. The rotor comprises a cylinder with deep ribs perpendicular to the axis, and several methods are described of constructing such a rotor. B. M. VENABLES.

Apparatus for bringing liquids and gases into contact. [Rectification of liquid air.] L'AIR LIQUIDE Soc. ANON. FOUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (E.P. 262,042, 11.5.26. Conv., 28.11.25).—In a rectifying column the trays are constructed to hold an amount of liquid roughly in proportion to the viscosity and surface tension of the gas bubbles at that region; e.g., in a liquid air column the trays in the upper or nitrogen-rich end are made larger and/or deeper than in the lower end, because nitrogen bubbles take so much longer to burst than those of oxygen. B. M. VENABLES.

Apparatus and process for leaching, filtering, and absorbing gases and the like. H. M. STARK (U.S.P. 1,625,831, 26.4.27. Appl., 24.2.23).—A gas filter is constructed with pairs of stamped sheets side by side, with the cut portions so bent that a number of V-shaped capillary spaces are formed between the two plates.

B. M. VENABLES.

Process and apparatus for treating liquids with purifying and decolorising agents. J. N. A. SAUER (U.S.P. 1,627,343, 3.5.27. Appl., 25.5.23. Conv., 26.5.22).—See E.P. 198,366; B., 1924, 321.

Furnaces [for preheating etc.]. A. SMALLWOOD and J. FALLON (E.P. 266,923, 6.4.26. Addn. to E.P. 255,938 ; B., 1926, 792).

[Device for removing the lids of] furnaces. BRITISH THOMSON-HOUSTON CO., LTD., and A. N. OTIS (E.P. 255,479, 16.7.26. Conv., 16.7.25).

Heating or cooling viscous liquids. GRISCOM-RUSSELL Co., Assees. of R. C. JONES (E.P. 256,579, 21.6.26. Conv., 5.8.25).

[Basket cover for] hydro-extractors or centrifugal machines. A. F. DUNSMORE (E.P. 268,701, 30.12.26).

Process and apparatus for charging a liquid with radioactive emanations or gases. A. LEPAPE and E. LEVOUX (E.P. 267,764, 3.6.26).

Flotation apparatus (U.S.P. 1,624,559).-See X.

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

Primary decomposition of coal. I. Temperature of initial decomposition. J. G. KING and R. E.

WILLGRESS (Dep. Sci. Ind. Res., Fuel Res., 1927, Tech. Paper No. 16. 19 pp.).—A series of coal samples (20 g. each), from peat to anthracite, of 10-20-mesh size, were mixed with freshly-ignited sand (40 g.) of the same size and heated in a vertical tube placed in an electric furnace and connected with a cooled U-tube. The temperature of this mixture was raised 10° per hr., while an inert gas was passed through the tube, and the appearance of moisture and oil was observed. When the inert gas was nitrogen the temperature of initial appearance of oil from peat was 180°, from lignite 250°, and from bituminous coal 215° (for caking coal) to 240° (for noncaking coal). Substantially the same figures were obtained when using carbon dioxide or hydrogen, or working in vacuo, but no definite value was obtainable with anthracite. Decomposition continued for some hours at least if the coal was maintained at the critical temperature. With bituminous coals the temperature of initial decomposition appears to increase with increasing oxygen content and decreasing carbon content. The evolution of water from coal in the absence of air is a gradual process, but with certain coals there are temperatures at which the rate of evolution is accelerated, the first acceleration of moisture evolution coinciding, for bituminous coals, with the appearance of oil vapour. The temperatures of initial decomposition here recorded are much lower than those found by previous investigators. W. T. K. BRAUNHOLTZ.

Inflammation of coal dusts : effect of the chemical composition of the dust. T. N. MASON and R. V. WHEELER (Safety in Mines Res. Bd., 1927, Paper No. 33. 20 pp.) .-- Mixtures of coal dust and fuller's earth, both containing about 85% passing through 200-mesh, were spread on the floor of a gallery 38 in. in diameter and 308 ft. long, closed at one end. Ignition of the dust was effected by a cannon, aided by an "impetus tube." The inflammability of the dusts of a series of British bituminous coals of equal fineness, measured by the amount of incombustible matter which must be present in the mixed dust in order to prevent continued propagation of flame, was related to the content of volatile matter in the coal (ash-free and dry basis), the dusts from coals with a high content of volatile matter tending to be more readily inflammable. It is not suggested, however, that the content of volatile matter directly determines the inflammability of the coal dust. No difference was found between the inflammability limits of fresh and weathered dusts from a given coal. The effects on flame propagation of the concentration of dust in the gallery and "impetus tube" and of varying the cannon charge were also studied.

W. T. K. BRAUNHOLTZ.

Upper Silesian blast-furnace coke. F. BÖNNE-MANN (Glückauf, 1926, 62, 1551—1557; Chem. Zentr., 1927, I, 543).—A systematic study has been made of the coking properties of coals from various seams, none of which, by itself, yields a good coke. By determining the most suitable particle size, width of oven, temperature, and mixture ratios, it has been possible to produce a coke which works smoothly in the blast furnace, giving a 60% increase in the yield of iron and an improvement also in the quality of the latter. A. B. MANNING.  Purification of town gas in relation to corrosion. J. PARKER (Gas J., 1927, 178, 361-362).-Corrosion inside gas mains is mainly attributed to the condensation of water vapour from the gas in the presence of oxygen, carbon dioxide, etc. This can be prevented if the saturation of the gas is reduced below 40% by washing with a strong solution of calcium chloride in a "brush" horizontal rotary washer. The solution is continuously circulated through the washer via a cooler to extract the heat evolved in the washing process, the diluted solution being reconcentrated by means of exhaust steam from the engine driving the washer. The cost of the process when operated in conjunction with that of naphthalene extraction does not exceed 0.2d. per 1000 cub. ft. of gas treated. Tests carried out on the dried gas have given no indication of the presence of iron carbonyl. H. D. GREENWOOD.

Apparatus for determining the content of solvent vapours in gases, particularly of benzene hydrocarbons and gas benzine from the carbonisation of coal. A. WEINDEL (Brennstoff-Chem., 1927, 8, 136— 138).—The tin adsorption tube containing active charcoal is inserted into a thick-walled aluminium cylinder heated by gas. Steam is superheated by passing, on its way to the tin tube, through a tube bored out of the wall of the cylinder, in which also a thermometer is inserted. The temperature of the aluminium cylinder is raised to 350° (corresponding to a temperature of 320—330° in the tin tube), and the expulsion of the solvent is complete in 25—35 min. Only 100—150 g. of steam are required. W. T. K. BRAUNHOLTZ.

Products from the cracking of lignite producer tar oils and petroleum gas oils. G. VARGA and A. ERDÉLY (Brennstoff-Chem., 1927, 8, 133-136).-One litre of oil was rapidly heated in an autoclave till the internal pressure reached a given figure, and the cracked products were withdrawn while that pressure was maintained. The yields of oils boiling up to 200° were about 45-57% from various gas oils, and 32-47% and 28-32% from refined and crude lignite producer tar oils, respectively. At 10-20 atm. pressure the lignite producer tar oils began to decompose at 370-390°, and the maximum yield of benzine was obtained at about 420°, whilst above this temperature coke and gas formation predominated. The corresponding temperatures for the Roumanian, Russian, and Texas gas oils examined lay about 20° higher. With gradually increasing cracking temperature the sp. gr. of the benzine from the lignite oils first sank and then rose. There was no material difference as regards boiling range, sp. gr., and solubility in 100% sulphuric acid between the benzine obtained from petroleum and that from refined lignite producer tar oils. W. T. K. BRAUNHOLTZ.

Utilisation of gas coke. E. W. SMITH (J. Soc. Arts, 1927, 75, 572-596).

Spalling of refractories. GREEN and DALE.—See VIII.

Use of super-refractories in gas manufacture. KNOLLMAN.—See VIII.

Coke-oven construction and refractories. VICKERS and GREEN.—See VIII. Discoloration of glass by coal gas. OFFE.—See VIII.

#### Gallium in flue-dust. RAMAGE.—See X.

#### PATENTS.

Decomposition of coal and hydrocarbons by heating with hydrogen under pressure. Soc. INTERNAT. DES COMBUSTIBLES LIQUIDES (F.P. 606,189 and 606,191, 12.2.25).-(A) The reaction is carried out in two stages, the material being hydrogenated in the first and decomposed into separate fractions in the second. By heating a mixture of powdered bituminous coal and tar oils, in the proportion 2:1, directly at 440-475° in the presence of hydrogen at 100 atm. pressure, no liquid product is obtained, but by heating first to 420° within 24 hrs., and then at 440° for 2 hrs., a 56.9% yield of oil boiling to 330° is obtained. The amount of hydrogen taken up increases with the pressure, whilst the amount of decomposition varies inversely with the pressure. The yield and quality of the low-boiling fraction of the oil are better the greater the hydrogen absorption during the first stage. When a mixture of equal parts of coal and coal-tar residues is heated in the autoclave at 210° it becomes liquid, free carbon and ash settling out, but the yield of oil of low b.p. is small. If, however, this product is again hydrogenated after the addition of 5% of alkaline iron oxide a good yield of oil boiling to 230° is obtained. (B) The starting materials are pumped under pressure into the reaction chamber, which is provided with stirrers. The liquid reaction product can be discharged through a tube which dips below its surface, into a container fitted with a valve. The separation of the solid, liquid, and gaseous products is carried out under normal pressure. A. B. MANNING.

Production of fuel. C. J. GREENSTREET, ASST. to AMERICAN COALINOIL CORP. (U.S.P. 1,623,241, 5.4.27. Appl., 13.9.22).—Coal is ground and agitated with a mixture of water and fuel oil of density greater than  $1 \cdot 18$ . The impurities are thus separated from the coal, which is dispersed in an emulsion of oil and water. The product, which is of value as a fuel, can be rendered more stable by blowing it with air to oxidise and thicken the oil. T. S. WHEELER.

Production of a binding fuel material. T. NAGEL (U.S.P. 1,626,208, 26.4.27. Appl., 24.5.24).—Pulverised fuel is incorporated in suitable proportions with a mixture of alcohol slops and sulphite liquor, then pressed, and heated at 250–500°, to convert the mixture into a hard insoluble smokeless binder. H. ROYAL-DAWSON.

[Fuel] pulveriser. SYRACUSE PULVERIZER CORP., Assees. of A. J. BRIGGS (E.P. 258,896, 6.10.25. Conv., 8.8.25. Cf. E.P. 256,562; B., 1926, 999).—A pulveriser for fuel which draws sufficient air for combustion, and can be placed close to the furnace where the fuel is burnt, is constructed with a disintegrator of the hammer type surrounded by one of the intercalating tooth type, the powdered fuel being drawn off by a fan on the same shaft. Control devices for air and fuel are provided. B. M. VENABLES.

Gas generating apparatus. F. UMPLEBY (E.P. 269,269, 29.1.26).—The generator consists of a chamber of refractory material and having uneven (grooved) walls.

The gasification is effected by the heat generated by surface combustion on the walls. The air or oxygen and the material to be gasified are admitted to the chamber by regulatable valves, and the gas being generated can be modified by the admission of steam, carbon dioxide, etc. Steam is also admitted in the form of a screen through which the ash or residue from gasification must fall and is cooled. Catalysts may be used, also a rotary motion imparted to the fuel. The direction of flow of the generator and burning gases may be reversed in order to assist the separation of the heavier particles of ash as residue. The fuel may be introduced in the form of a spray. A suitable device is provided for the discharge of may also be made of a heat-resisting nickel alloy or nickel itself. A series of chambers for a graduated gasification the ash. The reaction chamber, valves, and other parts process may be provided. The gas may be generated from the volatile matter of carbonaceous material, and the fine coke-like residue fed to the furnace. The plant may operate in conjunction with a pulverised fuel or briquetting plant. R. A. A. TAYLOR.

Generation of combustible gas. J. E. HACKFORD, and HAKOL, LTD. (E.P. 268,899, 16.11.26).—The process described in E.P. 217,613 (B., 1924, 703) is modified in that the air admitted to the chamber is directed by several different suitable means round the walls of the chamber so that the fuel is prevented from coming into contact with the walls and depositing carbon on them. R. A. A. TAYLOR.

Manufacture of water-gas. H. NIELSEN and B. LAING (E.P. 269,234, 6.1.26).—The endothermic nature of the water-gas reaction is counterbalanced by the admission with the steam (which may be superheated) of a quantity of more or less inert gas admixed with carbon dioxide. The gas and steam may be heated in a chequer brickwork chamber and then brought into a second chamber to react with coarsely pulverised semi-coke. The sensible heat of the watergas made can be used to effect the carbonisation of coal. The material to be carbonised is allowed to fall down a vertical space between superposed shelves.

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

Coke ovens. N. V. SILICA EN OVENBOUW MIJ., Assees. of C. OTTO & Co., G.M.B.H. (E.P. 259,968, 13.10.26. Conv., 14.10.25).—A vertical chamber oven has two regenerators on one side only. They are separated by the chimney-flue common to both, and are connected with the heating-wall on the one side and with the flue, gas-main, or air on the other. The products of combustion thus follow a C-shaped path. The air and gas may be distributed so as to meet only at the top or bottom of the heating-flues. Mains for the distribution of rich or lean gas or both may be provided, together with suitable regulation of the openings from the distributing-flue. R. A. A. TAYLOR.

[Lampblack from natural] gas. STANDARD DEVELOP-MENT Co. From J. P. GARNER (Can.P. 261,588, 26.5.24). —Natural gas is cracked to produce lampblack, giving at the same time a low-grade gas. The latter is enriched by the addition of a hydrocarbon which is not liquid at the ordinary temperature, and which suffices to bring the calorific value up to that required for ordinary use.

A. B. MANNING.

Acetylene and lampblack from liquid hydrocarbons, e.g., petroleum. C. LONGHI (F.P. 612,036, 2.3.26. Conv., 7.3., and 21.9.25; cf. E.P. 248,830; B., 1926, 525).—The known reaction whereby acetylene is produced by the action of the electric arc under the surface of a liquid hydrocarbon rapidly ceases owing to the formation of a layer of vapour round the electrodes. This is avoided if the arc is extinguished immediately after its formation and re-formed at once in another place. Thus the oil is contained in a cylindrical vessel in which a disc rotates rapidly in such a manner that arcs are continually being formed and broken between a large number of points serving as electrodes on the disc and corresponding electrodes on the walls of the vessel. The same result is attained by the use of two discs without points which rotate in opposite directions. A. B. MANNING.

Treating oils. G. EGLOFF and W. R. HOWARD, Assrs. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,627,164, 3.5.27. Appl., 8.6.21. Renewed 5.2.27).—Heated oil is passed into an enlarged reaction chamber through a pipe, the open end of which is situated at the end of the chamber remote from the point of entry of the pipe. This end of the chamber carries a downward extension, and the vapours are withdrawn at a point remote therefrom. C. O. HARVEY.

**Treating impure crude oil.** J. F. WRIGHT (U.S.P. 1,627,072, 3.5.27. Appl., 26.1.24).—The oil is sprayed against a rotating member and comes in contact with a spray of hot water. Separation is effected in vertical chambers. C. O. HARVEY.

Treatment of petroleum emulsions. C. C. AVERILL, ASST. to W. S. BARNICKEL & Co. (U.S.P. 1,617,737-1,617,741, 15.2.27. Appl., [A-E], 2.9.24, 12.10.25, 21.11.25, 3.5.26, 26.6.26).-(A-C) Oil-water emulsions are broken by emulsification with water followed by chemical demulsification. (D) Water-in-oil emulsions are homogenised and subsequently subjected to electrical dehydrating action. (E) Water-in-oil emulsions are treated with a chemical demulsifying agent, homogenised to reduce the size and increase the uniformity of the water droplets, and dehydrated electrically. C. O. HARVEY.

Purification of benzol, petrol, and the like. K. Cox and P. J. MCDERMOTT (E.P. 269,242, 12.1.26).— The oil or spirit is heated with ferric sulphate and, if necessary, with a micro-porous material, and the distillate is then treated with alkali to neutralise its acidity. R. A. A. TAYLOR,

Refining oils. H. O. PARKER, Assr. to MATHIESON ALKALI WORKS (U.S.P. 1,627,338, 3.5.27. Appl., 18.6.25).—The oil is passed through a succession of mixing and settling operations, an alkaline aqueous solution of hypochlorite being supplied to one of the mixing chambers, allowed to settle out in the succeeding settling chamber, and then passed back to similar mixing and settling chambers preceding those to which the aqueous solution is first supplied. C. O. HARVEY.

Purification of waste acid from crude oil refining. W. DEMANN (G.P. 436,242, 10.9.25).—The waste acid is warmed or diluted with water, mixed with heavy benzine, tetralin, or other hydrocarbon of aliphatic CL. III.-TAR AND TAR PRODUCTS. CL. IV .- DYESTUFFS AND INTERMEDIATES.

or polymethylene character, and the mixture then separated by centrifuging. A. B. MANNING.

Purifying hydrocarbon oils. J. C. MORRELL and S. COMAY, ASSTS. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,627,055, 3.5.27. Appl., 22.6.25).—The oil is treated with a solution of copper hypochlorite.

F. G. CROSSE.

Distilling hydrocarbon material. E. M. CLARK, Assr. to STANDARD DEVELOPMENT Co. (U.S.P. 1,625,984, 26.4.27. Appl., 17.1.21).—The pitch residues resulting from the steam-distillation of hydrocarbon oils are heated, while supplying steam, until still wax distils over, when the distillation is continued without steam to the coking stage. C. O. HARVEY.

Reactivation of desulphurising agents for hydrocarbon oils. J. B. RATHER and F. S. SHEPARD, Assrs. to STANDARD OIL CO. OF NEW YORK (U.S.P. 1,622,671, 29.3.27. Appl., 16.4.24. Renewed 12.8.26).—Copper oxide which has been employed for the desulphurisation of hydrocarbon vapours is regenerated by treatment with superheated steam to remove adhering oil, and then with air at 260° to convert the sulphide present into oxide.

T. S. WHEELER.

**Removal of sulphuretted hydrogen from gas.** E. W. SMITH and T. C. FINLAYSON, Assrs. to WOODALL-DUCKHAM, LTD. (U.S.P. 1,628,477, 10.3.27. Appl., 14.11.25. Conv., 10.12.24).—See E.P. 245,575; B., 1926, 230.

Destructive distillation of solid carbonaceous materials. P. DVORKOVITZ (U.S.P. 1,627,321, 3.5.27. Appl., 16.10.22. Conv., 17.11.21).—See E.P. 192,816; B., 1923, 301 A.

Free-discharge separators of plant for washing coal and other minerals by means of liquid streams. A. FRANCE (E.P. 269,017, 28.6.26).

Separation of liquids and solids of low melting point (G.P. 437,482).—See I.

Fractional distillation (E.P. 269,090).—See I. Furnace settings (E.P. 260,953).—See I.

Separation of minerals etc. (E.P. 268,043).—See I. Coal washing installation (E.P. 254,693).—See I.

Washing minerals (E.P. 268,262).—See X.

Fatty acids from hydrocarbons (U.S.P. 1,627,452). --See XII.

Manufacture of methyl alcohol and hydrocarbons (E.P. 247,178).—See XX.

## III.—TAR AND TAR PRODUCTS.

Cracking of lignite producer tar oils. VARGA and ERDÉLY.—See II.

#### PATENTS.

Permanent emulsions (E.P. 254,701).-See I.

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

Dyes in wine. VALENTINI.-See XVIII.

#### PATENTS.

Production of 1-naphthol-8-carboxylic acid. R. HERZ and F. SCHULTE, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,623,678, 5.4.27. Appl., 12.7.26. Conv., 29.11.24).—1-Cyanonaphthalene-8-sulphonic acid is hydrolysed with 60% sulphuric acid at 70° to 8-sulpho-1naphthoic inner anhydride, m.p. 151—152°, which product on fusion with potassium hydroxide at 200° yields 1-naphthol-8-carboxylic acid. T. S. WHEELER.

Manufacture of arylamides of o-hydroxycarboxylic acids and of intermediate products for azo dyes. BRITISH SYNTHETICS, LTD., and E. B. HIGGINS (E.P. 268,877, 7.1.26).—Quantitative yields of arylamides are obtained by adding an arylamine gradually to an o-hydroxycarboxyl chloride stirred in a solvent or diluent (benzene) maintained at 60°. Hydrogen chloride is evolved and only 1 mol. of the amine is necessary. Examples are 2:3-hydroxynaphthoic anilide and  $\beta$ naphthylamide. C. HOLLINS.

Preparation of stable diazo compounds. I. G. FARBENIND. A.-G. (F.P. 610,261, 29.1.26).-Solid aromatic diazo compounds, with the exception of those derived from the aryl or aralkyl ethers of o-aminophenols, are mixed with metallic salts of aromatic sulphonic acids, with or without the addition of copper salts, aluminium salts, or alkali chromates. Numerous examples are given, the stabilising compounds being : technical sodium naphthalene-1: 6-disulphonate; the same with anhydrous aluminium sulphate ; sodium naphthalene-triand -tetra-sulphonates from naphthalene and 4 mols. of sulphuric acid monohydrate; sodium benzenesulphonate and tetralinsulphonate ; sodium naphthalene-2:7-disulphonate and crystallised copper sulphate. A moist 72% paste of p-diazobenzenesulphonic acid and sodium naphthalene-1: 5-disulphonate is dried without difficulty at 40-50°. In some examples diazo salts already stabilised as zinc chloride double salts or in the form of their *p*-chlorobenzenesulphonates etc. are mixed with aromatic sulphonates. C. HOLLINS.

Preparation of stable diazo compounds. CHEM. FABR. GRIESHEIM-ELEKTRON (F.P. 600,311, 3.7.25).— Aqueous solutions of nuclear substituted benzenediazonium chlorides, excepting sulphonic acids and azo derivatives, give stable zinc chloride double salts, 2RN<sub>2</sub>Cl,ZnCl<sub>2</sub>, which may be salted out with sodium chloride, and dried either at 50—70° or by admixture with partly anhydrous aluminium sulphate etc.

C. HOLLINS.

**Production of a [vat] dye [of the anthraquinone series].** W. G. WOODCOCK, H. A. E. DRESCHER, E. G. BECKETT, J. THOMAS, and SCOTTISH DYES, LTD. (E.P. 268,537, 3.10.25).—A blue vat dye, brighter in shade and faster to chlorine than that obtained from the product of reduction of 2:4-dichloro-1-aminoanthraquinone, is prepared by the condensation of 2-chloro-1-amino-anthraquinone, m.p. 196°, obtained by the method of E.P. 264,916 (B., 1927, 246) and purified by precipitation as *sulphate* from 70% sulphuric acid. The condensation takes place in presence of sodium acetate (etc.) and copper (etc.) in hot nitrobenzene, naphthalene, etc.

C. HOLLINS.

Manufacture of intermediates of the anthraquinone series. BRITISH DYESTUFFS CORP., LTD., and W. W. TATUM (E. P. 268,891, 11.1.26).—The preparation of 1:4-diamino- or 1:4-dialkyldiamino-anthraquinones in the form of their leuco-compounds is performed in a 438 CL. V.—FIBRES; TEXTILES; CELLULOSE; PAPER. CL. VI.—BLEACHING; DYBING; PRINTING; FINISHING.

single operation by treating a 1:4-dihydroxy- or 1-amino-4-hydroxy-anthraquinone with ammonia or an alkylamine in the presence of sodium hyposulphite and water, in an autoclave, if desired, or in an open vessel. Quinizarin, sodium hyposulphite, and aqueous ammonia at 70—90° give leuco-1:4-diaminoanthraquinone. Similarly leuco-1:4-dimethyldiaminoanthraquinone is prepared from 1-amino-4-hydroxyanthraquinone, aqueous methylamine, and hyposulphite at 30—80°; leuco-5-amino-1:4-dimethyldiamino-8-hydroxyanthraquinone from diaminoanthrarufin; leuco-1:4-dimethyldiamino-7:8-dihydroxyanthraquinone from 1:4:7:8-tetrahydroxyanthraquinone. The last two products after oxidation give greenish-blue dyes for acetate silk. C. HOLLINS.

Preparation of nitriles of the benzanthrone series. M. P. SCHMIDT and W. NEUGEBAUER, ASSTS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,628,280, 10.5.27. Appl., 23.2.26. Conv., 17.11.24).—See E.P. 243,026; B, 1927, 101.

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

Transverse sections of artificial silk. Y. KAMI and S. NAKASHIMA (J. Cellulose Inst., Tokyo, 1927, 3, 87-94).-Photography of cross-sections of threads is used as the safest method of controlling artificial silk manufacture and for judging the nature of the product. Comparisons are shown between sections and the coagulation of the cellulose derivative solution (i.e., composition of the spinning bath, bath temperature, drying of the raw silk, etc.). Four methods of preparing the sections are described and compared, and the effects of these methods in showing differences between various kinds of artificial silk are illustrated. Differences in the degree of swelling of sections prepared by the four methods are shown by measurements of their area, and the ratio area (sq. microns): denier of a single fibre is termed the softness factor of the silk. B. P. RIDGE.

#### PATENTS.

Cooking vegetable fibre. S. D. WELLS (U.S.P. 1,626,171, 26.4.27. Appl., 29.6.26).—Fibrous material is softened by treatment with a solution of sodium carbonate and sulphur. H. ROYAL-DAWSON.

Manufacture of a cellulose-fibre product treated with a size embodying soya-bean flour. G. DAVIDSON, H. F. RIPPEY, C. N. CONE, I. F. LAUCKS, and H. P. BANKS, ASSTS. to I. F. LAUCKS INC. (U.S.P. 1,622,496, 29.3.27. Appl., 3.5.26).—Soya-bean flour dispersed in water, with or without addition of weakly alkaline salts, is employed as a size for paper or the like. T. S. WHEELER.

Spinning process. H. L. J. CHAVASSIEU, ASST. to Soc. POUR LA FABR. DE LA SOIE RHODIASETA (U.S.P. 1,622,368, 29.3.27. Appl., 26.3.26. Conv., 20.1.25. Cf. E.P. 246,430).—Aqueous solutions of inorganic and organic thiocyanates and thiocarbimides (*iso*thiocyanates) readily dissolve cellulose esters and ethers. The solutions so obtained are applied to the production of filaments of artificial silk by spinning into water in the usual way. T. S. WHEELER.

Tasteless, odourless, water- and fat-soluble printings on parchment paper. H. WREDE (G.P. 438,075, 19.12.25).—Solutions of cellulose or cellulose derivatives into which colouring matters, metal powders, etc. have been incorporated, are used for printing on parchment paper, the colouring matters etc. being such as are unattacked by the chemicals used in parchmentising. Alternatively, the incorporated substances may be such as give in the parchmentising bath insoluble or uncoloured, dyeing or non-dyeing, precipitates; or the dyes may be applied in colloidal form; or in place of cellulose and its derivatives, substances may be used which give parchment-like precipitates with the usual parchmentising agents; or the prints may be made before parchmentising. C. HOLLINS.

Production of wood pulp. W. D. MOUNT (E.P. 269,256, 13.10.25).—See F.P. 607,726; B., 1927, 185.

Drying apparatus (E.P. 269,064).—See I.

Wool oil emulsions (E.P. 246,867).—See XII. Printing inks (E.P. 268,966).—See XIII.

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

Dyeing textile materials. H. B. SMITH (U.S.P. 1,623,709, 5.4.27. Appl., 29.8.23).—Cotton in the form of cloth or skeins with portions of varying degrees of hardness is immersed in a bath containing a restricted amount of a cotton dyestuff and an assistant of dyeing, *e.g.*, sodium chloride. The soft and loosely twisted portions of the cotton are rapidly dyed, and the bath is exhausted before the hard portions are coloured. The bath is then heated at 60° to fix the colour. The undyed parts of the material are bleached by immersion in an alkaline oxidising bath. A variegated material with good contrast between the dyed and undyed portions is obtained. T. S. WHEELER.

Dyeing acetate silk. BRITISH DYESTUFFS CORP., LTD., A. SHEPHERDSON, and A. DAVIDSON (E.P. 268,933, 1.3.26).—Acetate silk is dyed directly in presence or absence of dispersing agents by means of coramidines, the anthraquinoneacridines obtained from  $\alpha$ -arylaminoanthraquinones by the methods of G.P. 126,444 and 262,469. Examples are the coeramidines from 1-anilinoanthraquinone (greenish-yellow), 1-anilino-2methylanthraquinone (golden-yellow), 1:5-dianilinoanthraquinone (pink), 1: 4-di-p-toluidinoanthraquinone (brownish - red), 1-amino-4-p-toluidinoanthraquinone (bluish-red), 1-amino-5-p-toluidinoanthraquinone (violet). The 1-amino-5-p-toluidinoanthraquinone is prepared from 1-chloro-5-aminoanthraquinone and p-toluidine in presence of sodium acetate. C. HOLLINS.

Dyeing cellulose esters and ethers. I. G. FARB-ENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 245,790, 8.1.26. Conv., 9.1.25).— Cellulose esters or ethers are dyed with monoazo dyes made by coupling a diazo compound containing not more than one sulphonic group with *m*-aminoacetanilide or its homologues or derivatives which are capable of coupling. *m*-Aminoacetanilide is coupled with diazotised 2chloroaniline-5-sulphonic acid (yellow) and with diazotised 3-nitroaniline-4-sulphonic acid (golden-yellow).

C. HOLLINS.

Dyeing furs. BRITISH DYESTUFFS CORP. LTD., and R. S. HORSFALL (E.P. 268,952, 24.3.26).—Fur is dyed

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from an acid bath with N-alkyl- $\omega$ -sulphonic acid derivatives of aminoanthraquinones, aminoazo and diaminoazo compounds, no other sulphonic group being present. Thus *p*-nitrobenzeneazophenylaminomethane- $\omega$ -sulphonic acid acid gives an orange-scarlet shade. The affinity of these dyes for fur is better than that of ordinary acid dyes, and they possess other advantages. C. HOLLINS.

Dyeing animal fibres and fabrics of a protein nature. S. W. WILKINSON, Assr. to ZAIR SYNDICATE, LTD. (U.S.P. 1,628,484, 10.5.27. Appl., 10.9.25. Conv., 11.9.24).—See E.P. 242,027; B., 1926, 11.

Liquor circulating means for treating materials with liquids (E.P. 269,241).—See I.

Dyeing of leather (U.S.P. 1,628,160).—See XV.

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

Analysis of concentrated and fuming sulphuric acids by thermometric titration. T. SOMIYA (Proc. Imp. Acad., Tokyo, 1927, 3, 76-78).-The water content of concentrated sulphuric acid may be determined by titration with fuming sulphuric acid containing 25% of free sulphur trioxide, the titration being carried out in a large test tube placed in a Dewar flask ; the end-point is ascertained by plotting the temperature of the acid against the c.c. of fuming acid added and noting the point of intersection of the two straight lines thus obtained, the temperature ceasing to rise when all the water is converted into sulphuric acid. Fuming sulphuric acid is analysed in a similar manner; an excess of 80% sulphuric acid is added to combine with all the sulphur trioxide, and the mixture then titrated with the standard fuming acid. A. R. POWELL.

Analysis of commercial acetic anhydride containing little or no sulphuric acid by thermometric titration. T. SOMIYA (Proc. Imp. Acad., Tokyo, 1927, 3, 79-81).—Acetic anhydride, when free from sulphuric acid, is accurately determined by thermometric titration (cf. preceding abstract) with a 25% solution of aniline in acetic acid. The aniline solution may be standard-1sed by thermometric titration with sulphuric acid or with a standard acetic anhydride. Alternatively, the acetic anhydride to be analysed is titrated with an unstandardised aniline solution, a second portion is partially hydrolysed with a known small quantity of water, and the remaining anhydride titrated against the aniline solution; if a is the volume of aniline used in the first titration for 1-g. of the anhydride, m the weight of anhydride used in the second test and, after hydrolysis by n grams of water, b the volume of aniline used in the second titration, then the percentage of acetic anhydride in the commercial sample is 10,210  $am/\{18 \cdot 02(an - b[n + m])\}$ . If the sample contains sulphuric acid (present as sulphoacetic acid) the endpoint is reached when the following reactions have taken place: (a)  $Ph \cdot NH_2 + (CH_3 \cdot CO)_2 O = Ph \cdot NH \cdot CO \cdot CH_3$  $+ CH_3 \cdot CO_2H$ , and (b)  $Ph \cdot NH_2 + HSO_3 \cdot CH_2 \cdot CO_2H =$ Ph·NH<sub>2</sub>,HSO<sub>3</sub>·CH<sub>2</sub>·CO<sub>2</sub>H. In a second portion of the sample the sulphoacetic acid is removed by addition of lead acetate and, without filtration, the acetic anhydride is determined alone by titration with aniline. Alternatively, the sample is titrated directly with a solution of aniline hydrochloride in acetic acid, the sulphoacetic acid present not interfering in this case. The last-namcd acid may be determined directly in commercial acetic anhydride by thermometric titration with barium acetate, the following reaction taking place:  $Ba(OAc)_2 + 2HSO_3 \cdot CH_2 \cdot CO_2H = Ba(SO_3 \cdot CH_2 \cdot CO_2H_2)$ + 2AcOH. A. R. POWELL.

Treatment of pollucite and preparation of cæsium chloride. A. KASTLER (Bull. Soc. chim., 1927, [iv], 41, 428-434).-The cæsium in American pollucite, a non-radioactive mineral containing only traces of potassium, rubidium, and calcium, is extracted as follows :- The mineral is treated with concentrated sulphuric acid and extracted with warm water. On cooling, the extract deposits crystalline cæsium alum, which is decomposed by excess of barium hydroxide. The solution of cæsium hydroxide thus obtained is treated with carbon dioxide, filtered, and the filtrate evaporated to dryness. The residue of cæsium carbonate is purified by extraction with alcohol (which dissolves only the cæsium salt) and dissolved in hydrochloric acid. The volatility of cæsium chloride first becomes apparent at 700°. S. K. TWEEDY.

Acid-resistance of asbestos. A. CALMON (Gummi-Ztg., 41, 1861—1865).—The acid-resistance of asbestos is determined by measuring the loss in weight of asbestos fibres, less than 1 cm. in length, after treatment with acid under specified conditions. Hornblende asbestos, e.g., blue Cape or amosite asbestos, is more resistant to the attack of mineral acids, either cold or hot, than serpentine asbestos, e.g., Canadian, Rhodesian, or Russian asbestos. Asbestos in general resists the attack of weak acids, e.g., acetic acid, hornblende asbestos being unattacked even at the b.p. The effect of acid on the mechanical properties of the material is also described.

B. W. CLARKE.

Properties of tungsten carbide,  $W_2C$ . K. BECKER and R. HÖLBLING (Z. angew. chem., 1927, 40, 512—513). —Evidence for the existence of the ditungsten carbide,  $W_2C$ , prepared by heating an intimate mixture of tungsten or tungsten carbide, WC, with carbon in stoicheiometric proportions at temperatures over 1600°, is obtained by examination of the X-ray diagram. The diagram shows only faint lines corresponding with free carbon, tungsten, or tungsten carbide. The compound is readily attacked by chlorine at 400° with formation of tungsten hexachloride and graphite, whereas tungsten and tungsten carbide of the same grain size are almost unattacked. The conclusion of Westgren and Phragmén (A., 1926, 1084) that the material is a solid solution of carbon in tungsten is therefore negatived.

#### L. M. CLARK.

[Preparation of] pure oxides and salts of tungsten and molybdenum. E. K. JENCKES (Trans. Amer. Electrochem. Soc., 1927, 51, 299—308).—Ammonium paratungstate and tungstic acid containing less than 0.05% of impurity are obtained commercially from wolframite or scheelite. Wolframite is decomposed with sodium hydroxide and calcium tungstate is precipitated from the solution by addition of calcium chloride. The calcium salt is treated with hydrochloric acid, and the resulting tungstic acid is dissolved in ammonia solution. From this solution the pure ammonium salt is obtained. Pure tungstic oxide is obtained by dehydration of the acid. For the production of molybdic oxide, molybdenite is roasted, the sulphur content finally being lower than 0.5%. The roast is leached with ammonia solution, iron, copper, and calcium are then removed from solution, and ammonium molybdate is obtained by crystallising or seeding.

L. M. CLARK.

Industrial absorption of nitrogen oxides in the synthetic nitric acid industry. C. TONIOLO (Chim. et Ind., 1927, 17, 546-553).—See B., 1927, 251.

#### PATENTS.

Production of persulphuric acid and its soluble salts from sulphuric acid by electrolysis. OESTER-REICHISCHE CHEM. WERKE G.M.B.H. (E.P. 265,141, 27.10.26. Conv., 28.1.26).—Sulphuric acid containing about 500 g./litre of concentrated acid is electrolysed at 20°, a current concentration of 400 amp./litre being used. A yield of over 70% is obtained. The per-salts are made from the corresponding sulphates.

W. G. CAREY.

Manufacture of hydrocyanic acid. O. LIEB-KNECHT, ASST. to ROESSLER & HASSLACHER CHEMICAL CO. (U.S.P. 1,626,648, 3.5.27. Appl., 24.6.24).—A mixture of carbon monoxide and ammonia is passed in contact with a catalyst composed of a porous non-acid charcoal, the temperature of the reaction being maintained at 400—800°. H. ROYAL-DAWSON.

Concentration of ammonium nitrate solutions. AZOGENO S.A. PER LA FABR. DELL' AMMONIACA SINTE-TICA E PROD. DERIVATI, and C. TONIOLO (E.P. 247,229, 8.2.26. Conv., 7.2.25).—The latent heat of ammonium nitrate is utilised for concentrating solutions' thereof by dissolving the dry salt in a concentrated solution of ammonium nitrate, heating at 85—125°, and spraying the product into dry air. W. G. CAREY.

Manufacture of basic chromium salts [for tanning]. I. G. FARBENIND. A.-G., Assees. of FARBWERKE VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 251,267, 15.4.26. Conv., 24.4.25).—Basic chromium salts which are easily soluble in water and tan rapidly are made by combining a basic chromium salt, or the hydroxide, with an oxide, hydroxide, or salt of an alkaline-earth metal or of zinc, magnesium, or aluminium, with the addition of acid or alkali to produce the desired basicity.

W. G. CAREY.

Production of metal nitrides, hydrides, etc. É. TILCHE (F.P. 611,139, 27.5.25).—In processes such as the production of iron nitride and subsequent conversion of it into iron and ammonia by treatment with hydrogen, the gases are subjected to the action of radioactive material or an emanation, and the iron is raised to a high electrical potential and heated to redness during treatment with the nitrogen. L. A. COLES.

Process and apparatus for preparation of carbon disulphide. H. SCHULZ (G.P. 438,037, 3.4.25. Addn. to G.P. 407,656; B., 1926, 823).—The prior process is modified (a) by feeding sulphur vapour into the upper part of the oven and withdrawing carbon disulphide from the lower part, or (b) by removing the carbon disulphide at slightly increased pressure, or (c) by adding the sulphur from two containers or tubes attached to the floor of the oven. C. HOLLINS.

**Preparation of hydrogen sulphide.** A. HENWOOD, Assr. to R. M. GAREY (U.S.P. 1,623,942, 5.4.27. Appl., 9.11.25).—A mixture of sulphur (36%), paraffin b.p. above 110° (14%), and asbestos fibre (50%) evolves hydrogen sulphide in a regular stream when heated.

T. S. WHEELER.

Manufacture of stable hydrogen peroxide. A. L. HALVORSEN (U.S.P. 1,627,325, 3.5.27. Appl., 2.7.21).— The peroxide of an alkali-forming metal is fed into a mixture of alcohol and sulphuric acid, forming hydrogen peroxide and a sulphate; the latter is collected on a filter, and the greater part of the solution is distilled in one operation, the vapours from which are condensed separately, forming a solution rich in hydrogen peroxide, and another rich in alcohol. H. ROYAL-DAWSON.

Stable mixtures for generating carbon dioxide. J. F. SCHWARZLOSE SÖHNE G.M.B.H., Assees. of SEYDEL (G.P. 437,113, 24.12.25).—Mixtures of carbonates with succinic anhydride, benzoic anhydride, cinnamic anhydride, tannin, etc. are used for the preparation of compressed medicaments, mouthwashes, etc.

S. S. WOOLF.

Production of concentrated sulphur dioxide. GES. FÜR LINDE'S EISMASCHINEN A.-G. (G.P. 437,910, 14.3.25).—The temperature attained during the combustion of sulphur in oxygen is kept at a minimum by using an excess of sulphur vapour, and, if necessary, by carrying out the combustion under raised pressure.

L. A. Coles.

Dissolving liquid chlorine. G. ORNSTEIN (G.P. 437,689, 12.4.25).—Liquid chlorine is added in the desired quantity to a flowing stream of a solvent, such as water, or an absorbent, such as an alkaline solution.

L. A. COLES. Lime kilns (E.P. 265,654 and U.S.P. 1,627,215).— See IX.

Nitric oxide from air (E.P. 269,046).—See XI. Fungicides (G.P. 436,923).—See XVI.

## VIII.—GLASS; CERAMICS.

Immiscibility in silicate melts. J. W. GREIG (Amer. J. Sci., 1927, [v], 13, 1-44, 133-154).-Experiments in which binary mixtures of silica with various metallic oxides were heated at various high temperatures and the phases present after quenching determined by microscopical examination, show that silica-rich mixtures of magnesium, calcium, strontium, manganous, zinc, ferrous, nickelous, or cobaltous oxides with silica melt to two immiscible liquids, one of which is almost pure silica. The equilibrium relations in the affected parts of the systems : MgO-SiO<sub>2</sub>, CaO-SiO<sub>2</sub>, SrO-SiO<sub>2</sub>, and FeO-SiO<sub>2</sub> have been investigated, and the equilibrium diagrams constructed. In all four cases the temperature of equilibrium between cristobalite and the two liquid phases is of the order 1690-1700°, and the silica ends of the equilibrium diagrams are markedly

similar. In each case cristobalite crystallises out from the less siliceous liquid so rapidly on cooling as to make the quenching to a clear glass very difficult. Experiments with barium oxide-silica mixtures show that these oxides are apparently completely miscible, although the shape of the liquidus of cristobalite is indicative of the proximity of an area of immiscibility. This type of liquidus has not hitherto been encountered in silicate studies. Beryllium, stannic, lead, aluminium, sodium, and potassium oxides are completely miscible. Investigations on the systems : MgO-CaO-SiO<sub>2</sub>, MgO-FeO-SiO<sub>2</sub>, MgO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>, MgO-Na<sub>2</sub>O-SiO<sub>2</sub>, CaO-Al<sub>2</sub>O<sub>3</sub>-SiO2, CaO-Na2O-SiO2, and MgO-K2O-SiO2 show that in ternary systems containing two of the metallic oxides which form two immiscible liquids with silica, the range of compositions of mixtures which melt to form two liquids extends across the corner of the diagram, whilst for those ternary systems containing an oxide of the "miscible " type, there is only a very narrow range of compositions which melt to form two liquids. Cristobalite is the first crystalline phase to form when any of these liquids is cooled. Mixtures of boric and silicon oxides of varying composition were made into clear homogeneous glasses, there being no evidence of immiscibility. The quartz dissolved very slowly, and this is believed to be the reason why silica has been reported to be insoluble in fused boric oxide. This slow dissolution together with the settling of the heavier quartz through the melt probably explains the two layers reported by Dimbleby, Hodkin, Parkin, and Turner (B., 1923, 551 A). In so far as liquid miscibility with metallic oxides is concerned, silica is remarkably similar to boric oxide. All oxides found to be incompletely miscible with silica were found by Guertler (A., 1904, ii, 614) to be incompletely miscible with boric oxide. In each case, moreover, one of the liquids was found to be almost pure boric oxide. The m.p. of cristobalite is  $1713\pm5^{\circ}$ . The equilibrium temperature between cristobalite and the two liquids in the system  $CaO-SiO_2$ ,  $1698 \pm 5^\circ$ , is suggested as a fixed thermometric point. The exact ratio of lime to silica is of little importance, provided that it lies within the range of formation of two liquids. A mixture 15 CaO, 85 SiO<sub>2</sub> is suitable. Purity of materials is, however, of the utmost importance. The application of the present data to the problems of silica refractories and their bearing on petrology are discussed. J. S. CARTER.

Influence of foreign matter on the thermal expansion and transformation of silica. J. F. L. WOOD, H. S. HOULDSWORTH, and J. W. COBB (Trans. Ceram. Soc., 1926, 25, 289-308).-Additions (generally 2%) of various substances (mostly having relatively low m.p., and including lime for comparison purposes) were made to a Welsh (Bwlchgwyn) quartzite, ground to pass a 100-mesh I.M.M. sieve. Test pieces made from the various mixtures were fired to cones 9, 12, and 14. Data concerning the reversible thermal expansion, density, porosity, and refractoriness of these test pieces were obtained. It was found that boric acid, potassium carbonate, potassium chloride, potassium chromate, biotite, sodium felspar, lithium chloride, and ferric chloride facilitate the conversion of the quartz, the effects of boric acid, potassium carbonate, biotite, and borocalcite

being most pronounced in modifying the reversible thermal expansion. The true sp. gr. is not a safe criterion of the reversible thermal expansion of the different mixtures. Apparently the solution of the quartz in the matrix varies, some of the silica maintaining the expansion characteristics of a glass. Thus, the densities of mixtures made with 5% of boric acid and with 2% of lime are not widely different, but they have very pronounced differences in reversible thermal expansions. The refractoriness of the different mixtures is not appreciably inferior to that of the limebonded mixture. Some mixtures show an increase, others a decrease of porosity with increase of firing temperature. A. T. GREEN.

Influence of iron oxide in promoting the inversion of silica. W. J. REES (Trans. Ceram. Soc., 1926, 25, 314-320).-Analyses and microsections of the dark, ferruginous patches on well-fired silica brick containing iron oxide were contrasted with those of a normal portion of the brick. The photomicrograph of the medium-brown portion shows the presence of a large proportion of tridymite. The crystals, besides being more numerous, are also larger than those observed in the normal brick. The dark-brown portion of the patches shows still greater development of tridymite. Although the presence of iron oxide in a silica brick batch favours quartz conversion and accelerates the rate of tridymite formation, there is no indication that the ferruginous patches possess appreciably lower refractoriness. The presence of 2-3.5% of iron oxide in silica bricks for use in the open-hearth process may be advantageous, as the increased tridymite content will result in greater volume stability and reduced spalling tendency. Iron oxide in silica bricks for coke ovens is A. T. GREEN. deleterious.

Behaviour of felspar and flint with acids and bases. E. W. SCRIPTURE, JUN. (J. Amer. Ceram. Soc., 1927, 10, 238-242).-20% suspensions of felspar and of flint were made up in a series of tall test tubes, treate d with varying quantities of sodium hydroxide and hydrochloric acid, and allowed to remain for 18 hrs. The absorption of the acid or alkali was determined either by the titration of a portion of the clear liquid or by comparison with standard indicators. Flocculation or deflocculation was measured by three observations, viz., the height of the clear liquid, the height of the sediment, and the turbidity as determined by the evaporation of a 20 c.c. portion withdrawn from a level of 16 cm. below the surface (the total height of the column being With large additions of alkali, flocculation, 26 cm.). analogous to the so-called "salting-out" effect noted with clay suspensions, occurs. With smaller additions of alkali and passing over to the side of small acid additions, a wide deflocculation zone with constant  $p_{\rm H}$  of suspension is obtained. This state is characterised by compact sediment and a turbid liquid above. More acid additions result in pronounced flocculation, the sediment being loose and the liquid clear, corresponding with the ordinary flocculated condition of clay suspensions. Within the usual range of  $p_{\rm H}$ , clay causes a decrease, whilst the flint and felspar cause an increase of alkalinity, the effect being greater with felspar. A. T. GREEN.

Variation of soda and boric oxide in ground-coat enamels. R. R. FUSSELBAUGH (J. Amer. Ceram. Soc., 1927, 10, 270-274).-On the basis of a ground-coat enamel already in use three others were evolved, (a) with 5% of soda and 25% of boric oxide ; (b) with 15% of both soda and boric oxide; and (c) with 25% of soda and 5% of boric oxide. The batches were carefully milled and aged, and other precautions taken to get identical circumstances. Three thicknesses of enamel were tested, and these were fired at 870° for  $4\frac{1}{2}$ , 3, and  $2\frac{1}{2}$  min. respectively. The results show that there are definite limits to soda and boric oxide contents which can be used in this type of ground coat. An increase of soda at the expense of boric oxide produces an enamel liable to form "copperheads," but reduces the tendency to " blister " and " fishscale." The converse happens when boric oxide is increased at the expense of soda. The phenomenon of A. T. GREEN. blistering is discussed.

Agreement of ground coat and enamel. A. MALINOVSZKY (J. Amer. Ceram. Soc., 1927, 10, 275—277). —Staley's ratio is considered an admirable device for the control of enamelling. To obtain this factor, a "refractory value" of the ground coat, which is the ratio of the percentage of refractory constituents (in the ultimate analysis) in the ground coat to the percentage of fluxing constituents, is first determined. A "fluxing value" of the enamel, which is the inverse of the refractory value, is next obtained. Staley's ratio is found by dividing the refractory value of the ground coat by the fluxing value of the enamel. Good control has been obtained by keeping this factor at a constant value ranging between 1.33 and 1.66. A. T. GREEN.

Influence of iron oxide and iron sulphides on the rate of quartz conversion in commercial silica brick manufacture. W. HUGILL and W. J. REES (Trans. Ceram. Soc., 1926, 25, 309-313).-Silica bricks were made on a commercial scale from (a) marcasiteincrusted ganister and (b) normal ganister free from iron sulphides, with lime-emulsion additions to give 1.5% CaO in the finished product. These bricks were fired to cone 15-16. The results show that iron sulphides help the quartz conversion considerably, and appear to increase the mechanical strength of the product. Iron oxide was removed from a portion of a batch of ganister by means of treatment with acids, and bricks were made of the treated and untreated ganisters. It was thus shown that the presence of iron oxide in the brick favourably influences the rate of quartz conversion and increases the mechanical strength of the resultant product.

A. T. GREEN.

Hydrogen-ion control in ceramics. W. A. TAYLOR (J. Amer. Ceram. Soc., 1927, 10, 243-258).—The meaning of the  $p_{\rm H}$  of solution and the factors influencing it are demonstrated, and a description of the indicator method of its evaluation is given. A. T. GREEN.

Testing refractory materials for resistance to slag corrosion and erosion. A. J. DALE (Trans. Ceram. Soc., 1926, 25, 326—338).—Many testing methods concerning slagging have been evolved, but there is little fundamental data. A discussion of the testing methods is given. These are classified, and include the following measurements: (1) depth of penetration of

slag into brick; (2) viscosity of the slag brick solution; (3) volume of brick dissolved or otherwise removed during a definite heat-treatment in contact with slag; (4) effect of impact of powdered and heated slag on heated refractory material; (5) change in ultimate chemical composition of the slag or of the brick, after a definite period of reciprocal interaction; (6) progressive alteration in temperature gradient through a wall, one face of which is exposed to the solvent action of the corrosive melt; and (7) a deductive method based on the micro-examination of the layer between the slag and brick. It is suggested that laboratory methods which would evaluate the progressive rate of corrosion and erosion of different refractories by slags at various temperatures should provide results of definite industrial value. A. T. GREEN.

Thermal properties of refractories, and factors influencing them. A. T. GREEN (Trans. Ceram. Soc., 1926, 25, 361—385).—A résumé of the data concerning the sp. heat, heat capacity, temperature diffusivity, thermal conductivity, and surface emissivity of refractories is given. Some of the factors governing these properties are discussed. The average surface emissivity for a firebrick surface between 200° and 500° is given as 0.72. A. T. GREEN.

Trend of design in modern coke-oven construction and its bearing upon refractory materials. A. E. J. VICKERS and A. T. GREEN (Trans. Ceram. Soc., 1926, 25, 407-427).-The principles involved in modern coke-oven design, chiefly including the supply, utilisation, and transmission of heat, the velocity of the gases in the heating flues, the recovery of heat, and certain structural features, are discussed. A résumé of the properties of silica bricks, with special reference to coke-ovens, is given. Silica bricks are shown to possess certain advantages over fireclay and siliceous refractories, particularly those relative to mechanical strength at high temperatures, temperature diffusivity, thermal conductivity at high temperatures, and resistance to corrosion. A. T. GREEN.

Spalling of refractory materials. A. T. GREEN and A. J. DALE (Trans. Ceram. Soc., 1926, 25, 428-470).-Spalling is defined as the inability of a refractory material to withstand without disrupture the stresses induced by temperature fluctuations. A fundamental equation shows the spalling tendency to be directly proportional to the coefficient of expansion and inversely to the maximum shearing strain and the square root of the coefficient of temperature diffusivity. The constitutional factors influencing the spalling of silica, siliceous fireclay, magnesite, chromite, sillimanite, and zirconia products are discussed. The relation between the thermal expansion and the constitution of refractories, particularly silica and fireclay products, is considered at length. The methods which have been formulated for the testing of spalling are reviewed. It is stated that the crux of the solution of the problem lies in the study of the elasticity, visco-elasticity, and plasticity of different refractories over a complete range of temperature. The influence of iron oxide in promoting what is apparently a type of spalling encountered in the carbonising industries receives attention. A. T. GREEN.

Abstraction of sulphur dioxide from flame gases by glasses and glazes. FABER.—See II.

#### PATENTS.

Heat-resisting glass. R. F. BRENNER, Assr. to H. C. FRY GLASS Co. (U.S.P. 1,623,301, 5.4.27. Appl., 9.5.22).—A heat-resisting glass contains silica 65—80%, boron trioxide 10—20%, colloidal aluminium oxide 2—10%, alkaline oxides 5—10%, and alkaline-earth oxides 0.5—2%. T. S. WHEELER.

Blowing and moulding of silica articles. QUARTZ & SILICE (E.P. 263,766, 22.11.26. Conv., 29.12.25).— An electrically heated furnace for softening silica is provided with a balance arm supporting the silica blank and a counterweight, the movement of which indicates the deformation and therefore the softening point of the blank. Below the furnace is situated a mould, to which the softened blank is rapidly transferred, and in which it is blown to the required shape by means of compressed air. No subsequent machining or superficial fusion is necessary to produce a smooth finish.

#### B. W. CLARKE.

Manufacture of clay-bonded products by means of a casting clay slip. SCHEIDHAUER & GIESSING A.-G. (E.P. 262,383, 24.6.26. Conv., 4.12.25. Addn. to E.P. 253,947; B., 1926, 254).—In the manufacture of clay-bonded products according to the main patent, other non-plastic refractory substances in a fine state of division, such as sillimanite, calcined alumina, magnesite, etc., which are suitable for clay-bonding, are substituted for the chamotte and mixed with a small proportion of bonding clay for use as a slip during the casting process. B. W. CLARKE.

Manufacture of decorated tiles. V. LEFEBURE (E.P. 268,851, 7.12.25).—A mixture of an alkaline silicate, colloidal silicic acid, or silica itself with a metallic oxide or salt, one of the ingredients being in a syrupy condition, is moulded to a sheet or applied to an asbestos cement or other backing. The sheet thus formed is heated at below 350° to harden the silicate layer. Pigments and other fillers may be incorporated if required. B. W. CLARKE.

Making and moulding hard porcelain. A. M. MCINTOSH (U.S.P. 1,627,245, 3.5.27. Appl., 15.7.22).— The finished porcelain is heated until it is plastic, then pressed and moulded. H. ROYAL-DAWSON.

Preparation of ceramic products. W. VERSHOFEN (U.S.P. 1,628,522, 10.5.27. Appl., 26.9.23. Conv., 27.9.22).—See G. P. 380,623; B., 1924, 295.

Treatment of metallic oxides (E.P. 248,360).—See X.

#### IX.—BUILDING MATERIALS.

Evaporation of water and salt solutions from surfaces of stone, brick, and mortar. A. P. LAURIE and J. MILNE (Proc. Roy. Soc. Edinburgh, 1926, 47, 52-68).—Experiments were made to test the hypothesis that if in a porous substance (whether stone, brick, mortar, or brick or stone with mortar) containing an equal amount of water distributed through it the evaporation from one part of the surface is slower per unit surface than from the other, then there will be a passage of water from the slow-drying to the

This quick-drying portion during the drying-out. was confirmed in the case of a porous sandstone, and it was also shown that in the case of untreated stone and stone treated with "silicon ester" two stages of evaporation are apparent. In the first stage, when the stone is wet and the surface is probably kept flooded by capillary attraction, stones of equal area evaporate at approximately the same rate, the rate for each stone being roughly proportional to the amount of water left. In the second stage there is a much faster removal of residual water from the untreated stone than from the treated stone. In the case of brick in contact with mortar, decay of the brick is due only to crystallisation below the brick surface of calcium sulphate, the sulphuric acid being derived from the air and rain. The principal source of lime is in the mortar, the acid-charged rain passing into the mortar and through the brick in contact with the mortar, and forming calcium sulphate. During evaporation the solution of calcium sulphate is drawn into the brick and crystallises there, owing to the rate of evaporation at the brick surface being faster than the rate at the mortar surface. Study of a portion of an old priory which had been pointed with a mixture of lime and Portland cement led to the following conclusions: In pointing old buildings consisting of sandstones containing calcite, care must be taken to use a sand free from salt. Portland cement is quite unsuitable for such a purpose as it makes a dense mortar with a very low rate of evaporation. The practice of washing the surface of the mortar and not leaving it with the skin formed by the trowel is probably wise, as the skin probably diminishes the rate of evaporation. Before re-pointing a building, after the joints have been cleaned out, a suitable preservative should be sprayed on the surface of the brick or stone. It is essential that both mortar and stone should not be treated with a preservative, as this reduces the rate of evaporation for both surfaces. Experiments throwing light on the conditions promoting local crystallisation below the surface are described. W. CLARK.

Painting cement and plaster. GARDNER.-See XIII.

#### PATENTS.

**Process and apparatus for burning cement.** J. S. FASTING (E.P. 268,868, 6.1.26).—A rotary kiln is provided with a series of tubes or channels arranged on the outside of the drying zone kiln tube and which communicate freely with the body of the kiln. The raw material to be dried and the combustion gases pass in opposite directions through these tubes, which, owing to their increased cross-sectional area, will reduce the velocity of flow of the combustion gases and give more efficient heat transference. The material after being dried is subjected to a grinding process by passing below a device located in the tubes, thereby facilitating the subsequent burning process. B. W. CLARKE.

Kilns [for burning limestone]. E. W. PATTISON. From W. D. MOUNT (E.P. 265,654, 12.8.25).—A vertical kiln comprises the following parts in order downwards : Charging hopper with bell, magazine for limestone, outlets for gases, upper part of kiln with peep holes, constricted part of kiln with downwardly-inclined gas tuyères just below the narrowest part, lower part of kiln, rotating cone bottom discharging burnt lime. Air for combustion enters through the burnt lime, and is thus preheated; the gas inlets are inclined downwards in order to make the flame reach the centre and prevent overburning up the sides. B. M. VENABLES.

Lime kiln and method of burning lime. A. E. TRUESDELL, ASST. to DOHERTY RESEARCH CO. (U.S.P. 1,627,215, 3.5.27. Appl., 23.4.23).—The kiln is connected with the vertical kiln of a furnace for burning fuel under such conditions that the products of combustion are obtained at a temperature in excess of that required for burning limestone, and delivered to the kiln. The waste gases leaving the kiln are re-introduced into the furnace between the point where combustion takes place and that of delivery of the products of combustion to the kiln. H. ROYAL-DAWSON.

Concrete and similar materials. K. P. BILLNER (E.P. 268,893, 12.1.26).—Cement is mixed with from 70 to 100% of water and from 0.03 to 0.5% of powdered aluminium or zinc together with fine aggregate and colouring materials if required, sufficient water being added so that the concrete can be applied by brushing or spraying to brickwork, ordinary concrete, and structural steelwork. B. W. CLARKE.

Manufacture of hydraulic cement and the like. O. GERLACH (U.S.P. 1,627,170, 3.5.27. Appl., 9.10.24).— A mixture of calcium carbonate, zinc retort ashes, and carbon is calcined at 950—1000°. Zinc is volatilised and burns to oxide which is deposited on the lime, the product being finally pulverised. F. G. CROSSE.

Artificial stone or marble. L. E. WELCH (U.S.P. 1,626,577, 26.4.27. Appl., 17.8.26).—Magnesium oxide and a filler are mixed with a suitable proportion of a liquid consisting of  $1\frac{3}{8}$  gals. of zinc sulphate solution  $(d \ 2 \cdot 1)$  and 100 gals. of magnesium chloride solution  $(d \ 1 \cdot 8 - 2 \cdot 4)$  together with acetic acid.

B. W. CLARKE.

[Granulation] treatment of slags by fluids under pressure. M. MAGUET, ASST. to SOC. ANON. DES CHAUX ET CIMENTS DE LAFARGE ET DU TEIL (U.S.P. 1,627,982, 10.5.27. Appl., 6.10.23. Conv., 26.10.22).—See E.P. 206,132; B., 1925, 176.

[Mould for] the production of artificial stone blocks. H. SCHAEFER (E.P. 268,587, 7.6.26).

**Roofing material.** FLINTROTE Co., Assees. of C. E. RAHR and R. E. DRAKE (E.P. 255,477, 16.7.26. Conv., 16.7.25).

Cement for electrodes (G.P. 437,552).-See XI.

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

Alloys of iron research. W. ROSENHAIN. V. Preparation of pure chromium. F. ADCOCK. VI. Preparation of pure manganese. (Miss) M. L. V. GAYLER. VII. Preparation of high-purity silicon. N. P. TUCKER (Iron and Steel Inst., May, 1927. Advance copy. 52 pp. Cf. B., 1924, 832; 1925, 829).—V. Methods for the preparation of very pure chromium are described. The starting point is electrolytic chromium made by the electrolysis of an aqueous solution containing

30% of pure chromic acid and 1% of sulphuric acid. Commercial "pure" chromic acid is unsuitable, as it contains sulphate. Full details of the electrolysis are given. Three processes have been developed, all using lead anodes in connexion with (i) a porous cell and tin cathode, (ii) a square steel bar cathode, (iii) a rotating steel tube cathode. Of these, (i) gives a metal of high purity in dendritic form, unsuitable for melting in the induction furnace, whilst excessive electrical energy is used. Method (ii) is the simplest for obtaining small quantities of pure metal in a compact form, and the electrical energy used is only about one quarter that required by the first process. It is, however, difficult to remove the metal from the cathode. Method (iii) is the best for production on a larger scale, and gives an easilyremoved compact deposit with moderate energy consumption. The electrolytic metal contains both hydrogen and oxygen, the latter being present in a form which leaves no residue on dissolution in acid. The hydrogen is removed by melting in vacuo, but this process changes. the state of the oxygen to insoluble chromic oxide, which can only be removed by heating the solid metal in a current of pure dry hydrogen at 1500-1600°, when a product is obtained which is spectroscopically pure. It cannot, however, be melted, even in fused alumina crucibles in vacuo, without introducing oxygen. Pure chromium is quite soft, and its m.p. considerably abovethat of iron.

VI. Manganese of 99.3% purity may be prepared by the reduction of trimanganese tetroxide with aluminium in a salamander crucible lined with alumina; manganous oxide is not so suitable for this purpose. The method of Allmand and Campbell (A., 1925, ii, 305) is also suitable. Manganese with total impurities less than 0.01% can, however, be readily prepared by distilling commercial manganese in a high-frequency induction furnace under a pressure of 1-2 mm. The resulting metal, silver-grey in colour, is hard enough toscratch glass, but very brittle. Its m.p. is  $1244^{\circ} \pm 3^{\circ}$ . It shows four transformation points at the following temperatures :—(1)  $1191^{\circ} \pm 3^{\circ}$ ; (2)  $1024^{\circ} \pm 2^{\circ}$ ; (3)  $742^{\circ} \pm 1^{\circ}$ ; and (4)  $682^{\circ} \pm 1^{\circ}$ . Of these changes, (3) is accompanied by a change of crystal structure, but not (4). One or both of these is accompanied by a marked change in volume. The ingots obtained by melting in vacuo are extensively cracked owing to the transformations on cooling. The impure thermit manganese is less brittle, and has only a few cracks, whilst its m.p. and transformations are at higher temperatures. than in the pure metal.

VII. The greater part of the impurities in commercial silicon lie in the grain boundaries, and may be removed by the following process. The best quality of commercial silicon is finely powdered and just covered with water. Hydrochloric acid is cautiously added, when the silicide impurities are decomposed with evolution of spontaneously inflammable gases. The product is digested with hot aqua regia for 24 hrs., and then washed and transferred to a platinum dish, where it is treated with sulphuric and hydrofluoric acids, and heated until it fumes. After a further treatment with concentrated hydrochloric acid, it is washed and dried. The final product under favourable circumstances contains 99.94% Si, and may be melted in silica crucibles. W. HUME-ROTHERY.

Alloys of iron research. VIII. Constitution of alloys of iron and phosphorus. J. L. HAUGHTON (Iron and Steel Inst., May, 1927. Advance copy. 18 pp.). -The constitution of alloys of iron and phosphorus has been determined by thermal and microscopic analysis in the range 0-30% P. The addition of phosphorus lowers the f.p. of iron, which falls to a eutectic point at 1050°, and contains, approximately, 10.5% P; the constituents of this eutectic are the  $\alpha$  solid solution of phosphorus in iron, and the compound Fe<sub>2</sub>P. The liquidus then rises to 1166°, at which temperature the compound Fe<sub>3</sub>P is formed by a peritectic reaction between the liquid and the compound Fe2P, the latter corresponding to a maximum on the liquidus at 1380°. Beyond this point the liquidus falls to a second eutectic, the constituents of which are Fe<sub>2</sub>P and an unknown compound. The compound Fe<sub>2</sub>P forms no solid solutions, but magnetic analysis shows that it undergoes a transformation at about 420°, which is similar to the corresponding transformation in cementite (Fe<sub>3</sub>C). The compound Fe<sub>2</sub>P forms no solid solutions with excess of iron, but there may be a very slight range of solid solubility on the phosphorus side. The effect of phosphorus on the critical points is very marked. The A4 point is rapidly depressed and the A3 point raised, the two joining to form a completely enclosed area representing the existence of  $\gamma$ -iron. There is thus no boundary between  $\alpha$ - and  $\delta$ -iron, both of which form the same space lattice. W. HUME-ROTHERY.

Temper-hardening in steels. T. MATSUSHITA and K. NAGASAWA (Iron and Steel Inst., May, 1927. Advance copy. 16 pp.).-A plain carbon steel containing 0.75% C was quenched from 800°, and its magnetic hardness, Hm (measured by the coercive force in gauss), was determined after tempering for different times at various temperatures. When tempered below 400°, Hm decreases with lapse of time, reaching an asymptotic value. But between  $400^{\circ}$  and  $500^{\circ}$  the Hm-time curves first decrease, then rise to a maximum, and finally gradually diminish. The Hm-temperature curves fall as the temperature is raised to about 400°, rise to a maximum of Hm between 450° and 500°, and then fall again. The results with nickel-chromium steels are similar with different temperature values. Corresponding abnormal changes within the same temperature ranges have been found for the impact hardness, Brinell hardness, and tensile properties. The annealing of cold-worked steels produces similar abnormal changes in the same temperature zones. The cause is probably "recrystallisation," causing the disintegration or refining of the iron crystals before crystal growth begins. The magnetic hardness and mechanical hardness curves W. HUME-ROTHERY. run parallel to each other.

Fatigue strength of hard steels. J. M. LESSELLS (Trans. Amer. Soc. Steel Treat., 1927, 11, 413—424).— Tests on 0.42% and 1% carbon steels quenched and slightly tempered to give a ball hardness over 400 show that when a departure occurs from a straight line relation between tensile strength and Brinell hardness the ratio of the endurance limit to the tensile strength is also low. This effect was associated with residual compressive stresses on the outside of the specimens. T. H. BURNHAM.

Drawing of steel wire and its relation to qualities of steel. E. A. ATKINS (Iron and Steel Inst., May, 1927. Advance copy. 24 pp.).-The action of drawing wire causes a relatively quicker flow of metal in the interior than at the outside of the wire, and consequently any steel having segregations at the core of the rods leads either to complete or incipient fractures. Surface defects on wire may be caused by surface splashes or skin blow-holes on ingots, or by faulty after-treatment, e.g., production of fins or cracks during rolling. One serious source of wire "running out" or not sizing correctly has been traced to the presence of crystalline alumina in steels which have been deoxidised with aluminium. Thus one mild steel which exhibited about 40,000 specks to the sq. in. increased in diameter 10 mils during the last pass when being drawn to 14 gauge. Deterioration of wire may be caused by the action of occluded hydrogen on inclusions during the pickling process, as also on the deposited metal after galvanising, tinning, or electro-plating. Trouble has been caused occasionally through the presence of abnormal amounts of copper, tin, or nickel in wire-making steel.

C. A. KING.

Theory of the growth of cast iron repeatedly heated. C. BENEDICKS and H. LÖFQUIST (Iron and Steel Inst., May, 1927. Advance copy. 31 pp.).-Increase in volume is necessarily brought about by a difference in expansion between adjacent layers in a metal due to sudden temperature changes. No growth will occur unless cast iron is subjected to repeated heat cycles, including the critical temperature, and on account of internal bursting around the graphitic lamellæ the anomalous contraction at Ac decreases more rapidly than the anomalous expansion at Ar, thereby giving rise to a residual expansion with every heating cycle. Successively increasing internal fracturing in the metal permits the access of oxidising gases, which occasions further growth, as does also the increase in volume due to the formation of temper carbon. Growth of cast iron on heating may be reduced by stabilising cementite in white iron, as, e.g., low carbon and silicon and high manganese content, or by the introduction of 3-4% Cr. In grey iron conditions and composition which tend either to increase the tensile strength or raise the transformation point should be favoured.

C. A. KING.

Heat-resisting steels. W. H. HATTFIED (Iron and Steel Inst., May, 1927. Advance copy. 26 pp.).— The corrosion-resistance at high temperatures of a number of pure metals and ferrous alloys was investigated by measuring their increase in weight from exposure at 900° in air with various gaseous additions, and in industrial gases. A complex furnace gas was more active than a muffle atmosphere. Water vapour and sulphur dioxide in the gases increased the attack. The order of resistance of the pure metals was chromium, nickel, cobalt, copper, iron, tungsten. The protective influence of chromium on iron was greater than that of nickel, but the addition of 8% of nickel to 18% chromium steel improved the resistance of the latter, 446

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the optimum results being obtained by the further addition of 4% of silicon or tungsten. Silicon-chromium steel was strongly resistant in all the gases used. Nichrome, which was added for comparison, failed badly in sulphur dioxide, in which, however, 15% silicon iron was substantially resistant. T. H. BURNHAM.

Cold-rolled strip steel. T. SWINDEN and G. R. BOLSOVER (Iron and Steel Inst., May, 1927. Advance copy. 29 pp.).—Tests on the cold-rolling of strip steel are recorded. The steels contained 0.10, 0.34, 0.54 and 0.70% C, and were rolled from 0.30 in. to 0.10 in. in thickness. It was found that the maximum stress in tension increased with cold-working, but, for a given percentage reduction by cold-work, the increase was proportionately less as the amount of cold-work previously received by the steel increased. The proof stress rapidly approached the maximum stress and then followed more or less parallel to it; it became proportionately less as the carbon content increased. The resistance to shear increased with cold-working, but to a less degree than the maximum stress in tension. The percentage elongation value fell rapidly with initial cold-work, subsequent cold-rolling having little influence after an elongation of 10% had been reached. Brinell hardness tests on cold-worked materials are unreliable as an indication of the maximum stress in tension, but, in general, the ratio between these two tests increased with the cold-work. In general, the variation in properties was similar in the various steels, but the deadsoft steel (such as is used for cold-press work) was the least hardened by cold-work, whilst a 0.70% carbon steel, annealed before cold-rolling, work-hardened more readily. There was no indication of a fall in hardness with progressive cold-rolling. Five of these steels after reductions in thickness of approximately 33% and 66% were heated for 1 hr. at various temperatures up to 700° and the tests repeated. It was found that, except in the case of the dead-soft specimen, the proof stress rose on reheating to 300°, and the ratio of this to the maximum stress also increased; above this temperature both values fell. The rise in proof stress increased with the carbon content and with the amount of cold-work received. The maximum stress in tension increased with the reheating temperature up to 300° in all steels except the dead-soft strip, and fell with increasing temperature above this point. Annealing before cold-rolling reduced the rise obtained by reheating after rolling. The resistance to shear also increased up to 300°, the dead-soft strip being less affected than the remaining steels; the rise was again proportional to the amount of cold-rolling. The Brinell hardness rose on reheating, and the ratio to the maximum stress fell, thus giving a higher proportional rise for the Brinell number than for the maximum stress. The time of reheating beyond 1 hr. at 700° had a decided influence on the mechanical properties. Micrographs showing the change of structure in the steels are given.

## M. E. NOTTAGE.

Influence of annealing temperature on the properties of mild steel sheets. C. A. EDWARDS and J. C. JONES (Iron and Steel Inst., May, 1927. Advance copy. 36 pp.).—The effects upon the properties of steel

sheets of the temperature range 750-920° reached during "black-annealing" were investigated. The Erichsen values of several samples, varying in thickness and composition, after annealing at temperatures between 400° and 1000°, have also been determined. It is shown that most of the stresses left in the sheets from the hotrolling mills were removed by annealing at 625-650°. A further improvement generally occurred after the temperature was raised above the A3 point. In some cases a slight deterioration of the values was obtained by annealing at 700-875°, due to growth of the ferrite crystals. It is suggested that for those sheets in which the ferrite completely recrystallises at the low temperature, it may be possible so to adjust the period of annealing at 700° that small equiaxed crystals are obtained which will give results even better than those obtained by annealing above the A3 point. Tensile tests were carried out on sheets 0.4 mm. and 1.25 mm. thick, the tests being made both parallel and at right angles to the direction of rolling. It was found that after annealing below 550°, the yield-points across the grain were the higher. Annealing at 650° produced a fall in the yieldpoint, and the values across the grain became the lower. Above 700° the yield-point values decreased, possibly owing to the rapid growth of the ferrite crystals. At 850-900° a second rise in the value of the yield-point set in, which reached a maximum at the A3 point. The maximum stresses showed variations similar to the yield-points. The tensile strength measured at right angles to the direction of sheet-rolling was always the higher after annealing at the same temperature. There was a marked improvement in elongation after annealing at 600°, which reached a maximum at 650-700°; depreciation then set in, and a minimum was obtained at 800°. Annealing at still higher temperatures brought about secondary improvements, a maximum being reached at 900°; above this temperature there was a steady decrease in the elongations. At about 700° the elongations of the 0.4 mm. specimens were considerably higher when tested in the direction of the sheet-rolling, but the differences in this respect were small after annealing at 900°. The possibility of adjusting the ratio of the degree of bar-rolling to that of sheet-rolling applied to the material in directions at right angles to one another, so that the properties of the finished sheets should be equal in both directions after annealing at 700°, is suggested. The tensile properties of the 1.25 mm. specimens were more regular at right angles to the direction of the sheet-rolling, and the elongation values were also better. M. E. NOTTAGE.

Alloys of iron and manganese containing low carbon. R. HADFIELD (Iron and Steel Inst., May, 1927. Advance copy. 65 pp.).—A series of thirteen alloys with manganese content ranging from 0.06 to 38.9% were prepared, in which the carbon averaged 0.08%, together with two alloys containing 61.5% and 83.5% Mn. Tensile, bending, hardness, impact, and hammer-hardening tests were made on the alloys in three conditions, as cast or forged, annealed, and waterquenched. The electrical resistance, heating and cooling curves, and corrodibility of the alloys were determined, and photomicrographs of the series are given. The alloys are divided into four groups :--0-4% Mn, soft and tough ; 4-10% Mn, hard and brittle ; 10-15% Mn, decreasing in hardness ; and 15-39% Mn, possessing to a limited extent the characteristics of manganese steel. Manganese progressively reduces the magnetisability of the alloys, which between 16 and 17% become nonmagnetic. Contrary to manganese steel, the specific magnetism is not increased by annealing at 500° for 60 hrs. The electrical resistance increases progressively with the manganese content. The loss of magnetism on heating is little influenced by the manganese percentage, but on cooling the intensity of the transformation diminishes, vanishing at 16-17% Mn before ordinary temperatures are reached. Between 1.68% and 38.9% Mn the microstructure of the forged alloys changes progressively-pearlitic, troosto-martensitic, martensitic, martensitic and austenitic, austenitic. No alloy possesses useful corrosion-resisting properties, and up to the present none has shown valuable properties T. H. BURNHAM. for industrial application.

Determination of manganese in cobalt steels. A. HALLBAUER and P. KRÜGER (Z. angew. Chem., 1927, 40, 513—514).—The presence of cobalt interferes with the direct determination of manganese in an alloy steel, and separation of manganese as dioxide by treatment of a solution of the steel with the usual oxidants is not completely satisfactory. More consistent results are obtained by precipitation of iron, chromium, tungsten, etc. from a nitric acid solution of the steel with zinc oxide. Cobalt is removed from the filtrate by precipitation as potassium cobaltinitrite. Manganese can then be determined by the usual method after removal of nitrous acid with carbamide. L. M. CLARK.

Gallium in flue-dust. H. RAMAGE (Nature, 1927, 119, 783).—Notable quantities of gallium have been detected in flue-dusts. The following elements, derived from South Yorkshire coal, have been detected in a gas-works flue-dust: lithium, sodium, potassium, rubidium, cæsium, copper, silver, calcium, strontium, zinc, aluminium, gallium, indium, thallium, carbon, titanium, silicon, lead, vanadium, phosphorus, arsenic, antimony, bismuth, oxygen, chromium, molybdenum, sulphur, manganese, chlorine, iron, and nickel.

A. A. ELDRIDGE.

Intercrystalline corrosion of metals. H. S. RAWDON (Ind. Eng. Chem., 1927, 19, 613-619).-Intercrystalline corrosive attack of metals when due to corrosion alone can usually be related to some structural features peculiar to the metal. Thus pure tin shows little tendency to become brittle when corroded, but if traces of aluminium are present it becomes embrittled upon very slight corrosion, the attack being confined to the micro-constituent containing aluminium. A relationship of this kind has not been established in the case of the severe intercrystalline corrosion occurring with commercially pure lead. In general, metals become embrittled at a quicker rate when under high stress, and any practical remedy seems to include the reduction of the stress acting upon the metal, either externally or internally, considerably below the yield-point of the metal, and the application of protective coatings to prevent corrosion as far as possible. The work of other observers relating to the embrittlement of tin, lead, copper, brass, and iron is reviewed. C. A. KING.

Mechanism of the production of zinc. M. BODENSTEIN (Trans. Amer. Electrochem. Soc., 1927, 51, 449—456).—The reduction of zinc oxide to zinc in presence of carbon is shown to proceed according to the mechanism (i)  $ZnO + CO = Zn + CO_2$ , and (ii)  $CO_2 + C = 2CO$ . The velocity of the reaction is determined by the comparatively slow rate of the second stage, and thus depends largely on the physical condition of the charcoal, coke, or graphite which is acting as reducing agent. L. M. CLARK.

Electro-deposition of tin. W. FRAINE (Metal Ind. [New York], 1926, 24, 463; Chem. Zentr., 1927, I, 647).—For obtaining thick, smooth deposits of tin, the following electrolytic baths are recommended: (a) a solution of stannous hydroxide in fluoboric or fluosilicic acid; (b) a solution of 113 g. of stannous chloride crystals. 283 g. of sodium hydroxide, and 226 g. of dextrose in  $4 \cdot 5$  litres of water; and (c) a solution of 680 g. of sodium stannate, 7 g. of stannous chloride crystals, and  $0 \cdot 1$  g. of resin in  $4 \cdot 5$  litres of water. A. R. POWELL.

Principles of electrolytic studies on corrosion. W. BLUM and H. S. RAWDON (Trans. Amer. Electrochem. Soc., 1927, 51, 46-490).-From a discussion of the principles of corrosion it is shown that, if the corrosion is essentially electrolytic in character, it may reasonably be accelerated by the application of an E.M.F., and that, in this case, the potential required to produce a certain current density or loss in weight is a much more accurate measure of the relative corrodibility of various metals than is the loss in weight produced by a given current in a given period. In the latter case, the relative losses in weight of different metals do not even approximately correspond with those obtained by simple immersion in corroding media. For studying electrolytic corrosion, it is suggested that two plates of the metal should form two opposite ends of a hard rubber box, and that the loss in weight of a third plate of the same metal immersed midway between the other plates should be measured when different E.M.F. are applied between the centre and outside plates. The results may be plotted as loss in weight-potential curves or as current densitypotential curves. Examples of the use of the method A. R. POWELL. are given.

Influence of iron oxide on silica [bricks]. REES.— See VIII.

Corrosion of metals by waters. LÜHRIG.—See XXIII.

PATENTS.

Production of steel. C. H. WILLS, Assee. of J. K. SMITH (E.P. 245,716-7, 5.5.25. Conv., 10.1.25).--(A) Iron produced from ore according to E.P. 215,400 (B., 1924, 601) is separated from accompanying dross and the metallic particles are pressed into briquettes which form the charge for a crucible process. (B) Pig iron may be added to the briquette charge in the proportion of 15-25%. C. A. KING. Production of iron and other carbon-binding metals and alloys thereof having a very low percentage of carbon. H. G. E. CORNELIUS (E.P. 252,017, 10.5.26. Conv. 11.5.25).—Iron containing less than 0.05% C is produced by reducing a mixture of ore and carbonaceous material in briquette form in an electric furnace. The quantity of reducing material must be at least 5% less than the theoretical amount required. Suitable materials for regulating the viscosity and conductivity of the slag may be incorporated in the briquettes, and briquettes containing the ore of desired alloying elements may be introduced. C. A. KING.

Manufacture of steel and treatment of same for the manufacture of edge tools. A. E. G. T. VON VEGESACK (E.P. 268,616, 24.7.26).—Steel for the manufacture of edged tools contains 0.7 - 1.1% C, 10 - 16% Cr, and 0.75 - 2% Mn. After air-hardening, the steel is heated at 400-500° for 5-30 min. C. A. KING.

Steel alloy. POLDIHÜTTE (E.P. 245,792, 8.1.26. Conv., 8.1.25).—A steel alloy of high tensile strength and which maintains considerable resistance at high temperatures contains 4-19% W,  $3\cdot5-7\%$  Cr, and  $0\cdot5-10\%$  Ni.

**Protection of ferrocerium.** P. J. JOSEPH (F.P. 608,402, 26.12.25).—Ferrocerium rods are protected from atmospheric oxidation by coating them with an impermeable varnish or with a red-lead mixture.

A. R. POWELL.

Wear-resisting alloy. B. E. FIELD and R. FRANKS, Assrs. to HAYNES STELLITE Co. (U.S.P. 1,626,726, 3.5.27. Appl., 24.12.25).—A ferrous alloy, suitable for making articles subject to abrasion, contains 1—12% W, 25—35% Cr, 0.10—1.0% B, and 1—3.5% C.

F. G. CROSSE.

Manufacture of chromium-iron alloys. B. D. SAKLATWALLA (U.S.P. 1,623,757, 5.4.27. Appl., 5.6.26). —For the manufacture of low-carbon chrome steel, silicon is added to the metal layer of a bath of molten steel, and a mixture of chromite and chromium trioxide is added to the slag layer. The reaction between chromium trioxide and silicon evolves sufficient heat to promote reaction between silicon and chromite without additional external heating of the bath.

T. S. WHEELER.

Manufacture of magnetic alloys. V. B. BROWNE (U.S.P. 1,627,269, 3.5.27. Appl., 19.3.25).—Silicon iron of definite silicon content is melted in relatively large quantity and maintained in a molten condition until solid inclusions have floated to the surface. The process may be assisted by agitating the molten metal.

C. A. KING.

Copper-aluminium alloys. METALLBANK U. METALLURGISCHE GES. A.-G. (E.P. 268,654, 21.9.26. Conv., 15.4.26).—The strength of copper-aluminium alloys when hot is increased while still being malleable if the composition falls between the limits  $5-9\cdot5\%$  Al, 1--6% Mn, 0--3% Ni,  $0\cdot1--3\%$  Fe, and  $0\cdot01--2\%$  Sn. The tensile strength of a particular alloy containing 84% Cu,  $1\cdot5\%$  Fe, 5% Mn, 9% Al, and  $0\cdot5\%$  Sn was 80 kg./mm.<sup>2</sup> C. A. KING.

Production of zinc. F. KRUPP GRUSONWERK A.-G. (E.P. 255,482, 16.7.26. Conv., 20.7.25).—Zinciferous material is distilled until the zinc content is reduced to 10-15%. The residue is reduced and heated in a current of air to volatilise zinc oxide, which is recovered and treated in a muffled distilling furnace.

C. A. KING.

Recovery of zinc from dross obtained in melting zinc during refining. E. C. R. MARKS. From AMERICAN SMELTING AND REFINING CO. (E.P. 269,321, 7.4.26).—The dross is roasted so as to volatilise the contained chlorides of zinc and ammonia, which are collected and returned to the melting furnace. The calcined residue is leached for the further recovery of soluble zinc compounds. C. A. KING.

Metallurgical process [for treating copper ores]. W. E. GREENAWALT (U.S.P. 1,618,187, 22.2.27. Appl., 9.3.26. Cf. U.S.P. 1,572,115; B., 1926, 330).—Copper ore containing zinc is crushed and concentrated, the concentrate being roasted and leached with a dilute acid solution to dissolve copper, zinc, and iron salts. The solution, after treatment with sulphur dioxide to reduce ferric salts, which interfere with electrolysis, is electrolysed to recover copper and oxidise ferrous iron, and finally is treated with crude zinc oxide to precipitate iron and residual copper, and yield a solution from which zinc can be recovered. T. S. WHEELER.

Conversion of matte containing nickel. O. LELLEP, Assr. to INTERNAT. NICKEL CO. (U.S.P. 1,623,797, 5.4.27. Appl., 22.3.26. Conv., 7.7.26).—Matte containing nickel is blown with air in a converter to which sand is added, and to promote reaction a flame is directed on the material in the converter. When the sulphur has been reduced to a point at which oxidation of the metal begins, the air in the blast is replaced by waste gas from the converter, containing about 3% of oxygen, which completes the oxidation of the sulphur.

T. S. WHEELER.

Production of aluminium alloys having a high silicon content. T. GOLDSCHMIDT A.-G. (E.P. 255,103, 12.7.26. Conv., 10.7.25).—Substances, e.g., alkali chlorides, metallic oxides, etc., known to produce alloys of aluminium and silicon having a fine granular eutectic structure are added to the mixture of aluminium and silicon compounds prior to electrolytic fusion in a fluoride bath. C. A. KING.

Solder for aluminium and its alloys. H. TOTTMAN (E.P. 269,771, 10.8.26).—The solder consists of an alloy of equal or approximately equal parts by weight of block tin, granulated tin, and cadmium. Stearic acid is a suitable flux. F. G. CROSSE.

Treatment of antimonial ores, more especially tin ores containing antimony. ZINNWERKE WILHELMS-BURG G.M.B.H. (E.P. 268,278, 5.7.26. Conv., 29.3.26).— Tin ores containing antimony are reduced either previous to or simultaneously with their extraction with an acid. *E.g.*, the ore may be mixed with 5—10% of carbon, roasted, and leached with hydrochloric acid (d 1·16), or treated directly with acid in the presence of a ferrous or cuprous salt. C. A. KING.

Production of hafnium from its ores. SIEMENS & HALSKE A.-G., Assees. of B. FETKENHEUER (G.P. 437,352, 17.2.24).—The ore is decomposed by treatment with

chlorine and a reducing agent such as carbon, and hafnium chloride, which is subsequently worked up to hafnium, is separated from other gaseous products by diffusion. The separation may be effected with the aid of carbon tetrachloride vapour. L. A. COLES.

Washing minerals by means of liquid streams. A. FRANCE (E.P. 268,682, 22.11.26).—In a washing plant consisting of a series of launders in cascade formation, and in which the products delivered from the lower launders are returned for re-treatment, the product from the end of the uppermost launder is discharged on to a vibrating screen on which it is washed with clean water. The suspension of loam thereby formed is utilised to obtain a muddy liquor of suitable density for the washing plant. C. A. KING.

Flotation apparatus. T. M. OWEN and M. P. DALTON (U.S.P. 1,624,559, 12,4.27. Appl., 5.7.24).— A series of cells, each divided into impeller and elevator chambers, is arranged in line, so that all the impelling and elevating means can be mounted on a single shaft. The bottom of the head end of each impeller chamber communicates with its own elevator chamber, and the pulp is discharged from the tail end of each impeller chamber into the next elevator chamber, and means are provided to create a difference of pulp level between the inlet and outlet of the elevator chambers.

B. M. VENABLES.

Continuously-operating ore or ore-shale converting retort furnace. A. M. BEAM (U.S.P. 1,627,538, 3.5.27. Appl., 26.2.24).—In a roasting furnace a retort and flue consist of two channel members, the web of one channel fitting with the side flanges of the second to provide a longitudinal passage. A plate fitting on the flanges of the first channel forms a second similar passage.

C. A. KING.

Electric furnace for the manufacture of aluminium. E. R. LAUBER (E.P. 256,193, 22.6.26. Conv., 30.7.25).—The bottom of a furnace for the production of aluminium by electrolysis consists of material, *e.g.*, alumina, corundum, magnesia, which is not attacked by molten aluminium. The cathode is formed by a layer of molten aluminium to which the current is led by means of graphite blocks. C. A. KING.

Methods and apparatus for fusing metals by the electric arc process for welding, cutting, etc. BRITISH THOMSON-HOUSTON CO., LTD., Assees. of I. LANGMUIR and P. P. ALEXANDER (E.P. 257,955, 6.9.26. Conv., 5.9.25).—In the process of welding with a fusible electrode, the molten weld is flooded with a mixture of nitrogen and hydrogen, the hydrogen being in such proportion (about 6%) as to prevent nitrogenisation of the metal. C. A. KING.

Manufacture of very thin wires. G. L. HERTZ, Assr. to N. V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,627,971, 10.5.27. Appl., 22.4.25. Conv., 18.6.24).— See E.P. 235,893; B., 1926, 245.

**Open-hearth furnace.** E. BOSSHARDT (U.S.P. 1,627,425, 3.5.27. Appl., 30.6.24. Conv., 25.10.23).— See E.P. 223,870; B., 1925, 456.

Sealing metal to glass. G. B. Jonas, Assr. to N. V. Philips' Gloeilampenfabr. (U.S.P. 1,627,780,

10.5.27. Appl., 2.4.26. Conv., 11.5.25).—See E.P. 251,951; B., 1927, 80.

Ore washing installations (E.P. 254,693).—See I. Separation of minerals etc. (E.P. 268,043).—See I. Furnace (U.S.P. 1,624,043).—See I.

Beryllium filament (U.S.P. 1,622,604).-See XI.

#### XI.—ELECTROTECHNICS.

Silver accumulator. F. JIRSA and K. SCHNEIDER (Z. Elektrochem., 1927, 33, 129-134).-A secondary cell in which the positive electrode consisted of silver peroxide, yielding silver on discharge; in an alkaline electrolyte was patented by Jungner in 1899, and was claimed to have several advantages. With a view of finding reasons for its lack of success in practice, a model accumulator of the type Ag|NaOH solution|Fe has now been examined. Positive plates consist of iron or nickel gauze on which has been pressed the active mass of spongy silver. Negative plates are prepared by smearing iron gauze with a paste of powdered iron and gum arabic. On charging, the silver is converted first into the oxide and then into the peroxide, the open circuit E.M.F. corresponding with these two stages being 1.17 and 1.47 volts. Using plates with 0.063 g. of silver per cm.2 of surface, and charging and discharging at 0.11 amp./dm.2, the capacity was 269 watt-hours per kg. of active material and in a charge-discharge cycle the current efficiency was 95.5% and the energy efficiency 68.4%. Increasing the amount of active material per unit surface or using thicker plates diminished the capacity at given current density. In successive charges the E.M.F. decreases and the capacity falls notably. This is attributed to silver peroxide, which tends to form a colloidal suspension, being carried by cataphoresis to the negative plate, and there reduced to silver. This action does not occur if the electrolyte is kept free from carbonate, and it can be prevented completely, even when much carbonate is present, by the addition of potassium sulphate. H. J. T. ELLINGHAM.

Electro-deposition of tin. FRAINE.—See X.

Electrolytic corrosion tests. BLUM and RAWDON.— See X. Burnard

## PATENTS.

Method and apparatus for electroplating. BRITISH THOMSON-HOUSTON CO., LTD., Assees. of C. E. JONES (E.P. 259,983, 15.10.26. Conv., 15.10.25).—In an electroplating process the anode consists of scrap metal held in a "cage" of perforated metal, *e.g.*, expanded sheet, which is itself in electrical connexion with the metal container. C. A. KING.

Apparatus for the electrolysis of water. L. CASALE (E.P. 268,998, 1.6.26).—The active faces of the electrodes are made of wire nets or perforated metal sheet, and one or more diaphragms, formed of asbestos cloth kept taut by a rod, are inserted between adjacent active faces of opposite polarity. These are carried by supports, each having two diverging extensions, for conveying into the respective gas-receiving bells the gases evolved between the diaphragm and the active face of the electrode. J. S. G. THOMAS. Decreasing the destruction of graphite and carbon electrodes used in the electrolysis of chlorides. KÖNIGSBERGER ZELLSTOFF-FABR. & CHEM.-WERKE KOHOLYT A.-G., and E. SCHLUMBERGER (G.P. 437,532, 30.7.24).—Electrolyte is caused to flow into the bath, wholly or in part, through pores, channels, etc. in the electrodes, the channels being of such form that the length of path travelled by the electrolyte in the electrodes is throughout the same. J. S. G. THOMAS.

Apparatus for ozonising air and converting it into nitric oxide. A. NEGLE (E.P. 269,046, 30.8.26).— A combustion device consisting of several arc-producers contained in a closed compartment is arranged above an ozoniser having the form of a surface condenser arranged in a separate air-tight closed casing. Both devices are connected to a source of high-tension, highfrequency multiphase current producing a strong electric field in the ozoniser and a large superficial homogeneous arc field in the combustion chamber.

J. S. G. THOMAS.

Cement for joining graphite plates used as electrodes. F. WIECZOREK (G.P. 437,552, 13.8.25).— Powdered grog, clay, sand, graphite, and water-glass are mixed to form a paste. The cement so produced is suitable for making electrodes used in the carbide industry, and lasts as long, at the high temperature of working, as the cemented graphite plates. J. S. G. THOMAS.

Induction furnace. M. UNGER, ASST. to GENERAL ELECTRIC CO. (U.S.P. 1,626,437 and 1,626,485, 26.4.27. Appl., [A], 31.12.25. [B], 16.2.21).—(A) A crucible forming a reservoir for the charge has a looped channel communicating therewith at its periphery and a primary winding for inducing heating currents in the liquid charge in the channel which has a greater cross-sectional dimension in a direction at right angles to its axis in order to permit increased circulation of the charge by electromagnetic repulsion. (B) A furnace body, forming a main chamber for containing a pool, has a number of openlooped channels arranged side by side on a common winding axis communicating at their ends with the chamber and with each other at their bends.

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

Getter [for incandescence lamp filaments] and its application. D. MACRAE and H. K. RICHARDSON, Assrs. to WESTINGHOUSE LAMP Co. (U.S.P. 1,623,351, 5.4.27. Appl., 8.6.22).—Incandescence lamp filaments are passed through an aqueous suspension of a getter having a refractory constituent, *e.g.*, thorium oxide, and a fusible constituent, *e.g.*, cryolite, into a tube furnace, where they are heated to fuse the cryolite and produce adherence of the getter. After being sealed into the lamp, each filament is heated to volatilise the getter.

T. S. WHEELER.

Beryllium filament. A. NYMAN, ASST. to DUBILIER CONDENSER CORP. (U.S.P. 1,622,604, 29.3.27. Appl., 7.11.24. Renewed 4.3.26).—Beryllium is claimed as the principal ingredient of the filaments of thermionic valves. T. S. WHEELER.

Removing residual gases from the metal casings of electric discharge tubes. SIEMENS-SCHUCKERT-WERKE G.M.B.H. (E.P. 257,620, 27.8.26. Conv., 28.8.25). —The metal casing is surrounded by a gas or liquid, e.g., liquid or gaseous sodium or potassium, which absorbs residual gases more easily than does the metal of the casing. A temperature gradient being established in the metal jacket, residual gases diffuse from the inside to the outside of the casing. Preferably, the pressure in the medium surrounding the casing is so low that the average length of free path of gases coming in contact with the casing is so great that only molecular flow controls the flow of gas, so that penetration of gas into the discharge vessel is prevented. J. S. G. THOMAS.

Electric fuse. SIEMENS & HALSKE A.-G. (G.P. 437,053, 7.10.23).—A fuse is constructed of a metal with a high conductivity, such as copper, coated electrolytically with chromium or cobalt. L. A. Coles.

Thermo-element for measuring high temperatures. F. KRUPP A.-G., Assees. of F. STÄBLEIN (G.P. 438,019, 24.6.24).—An element capable of prolonged use at 1300° has one arm constructed of a heat-resisting chromium-nickel alloy, containing, e.g., 84—88% Ni and 12—16% Cr, and the other of a chromium-nickeliron alloy with an iron content of at least 20%, containing, e.g., 60% Ni, 25—30% Fe, and 10—15% Cr.

L. A. Coles.

Production of dry cells. Twin Dry Cell Battery Co., Assees. of C. P. DEIBEL and W. G. WAITT (E.P. 244,494, 15.12.25. Conv., 15.12.24).

[Suspension for plates in] secondary electric batteries. "HART" ACCUMULATOR CO., LTD., and C. R. HARDY (E.P. 269,380, 28.7.26).

**X-Ray tube.** G. HOLST and A. BOUWERS, Assrs. to N. V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,626,465, 26.4.27. Appl., 6.11.23. Conv., 6.12.22).

Electric furnace for aluminium (E.P. 256,193).— See X.

Insulating materials (G.P. 437,115).—See XIII.

## XII.—FATS; OILS; WAXES.

Determination of the higher saturated fatty acids insoluble in water in fats and fatty acid mixtures. S. H. BERTRAM (Chem. Weekblad, 1927, 24, 266-269).-An exhaustive review of the literature is given, and the sources of error in the various methods are described. Accurate results may be obtained by oxidising the saponified material with alkaline permanganate, removing the fatty acids after acidification by means of light petroleum, transforming these into the magnesium salts, those of the higher fatty acids being precipitated whilst those of all other acids remain in solution, treating the precipitate after washing with dilute sulphuric acid, and extracting the higher insoluble fatty acids with light petroleum. Results are given for mixtures made up from specially purified acids, for oleic and other unsaturated acids from various sources and after various "purifications," and for a large number of common oils and fats. S. I. LEVY.

Chemical mechanism of linseed oil drying. W. L. EVANS, P. E. MARLING, and S. E. LOWER (Ind. Eng. Chem., 1927, 19, 640-641).—The relative effect on both the acid value and the iodine number of drying linseed oil films to which had been added previously equimolecular amounts of lead acetate, manganous acetate, or cobalt acetate was studied by the general procedure previously described (B., 1927, 82). 0.01 mol. of the crystalline acetates was incorporated in 200 g. of linseed oil of iodine value 176, a temperature of 271° for 20 min. being sufficient to complete the incorporation, and this value was chosen as unity. Results were obtained at concentrations 0.05, 0.10, 0.20, and 0.40 of the chosen unit. From the results plotted graphically, it was found that a general inverse relationship of the iodine value and the acid value exists in a drying linseed oil film, and the concentration of the drier bears a definite relationship, of an exponential character, to the decreasing iodine value. Furthermore, the general difference in behaviour of the three driers expressing both acid values and iodine values, respectively, as functions of time, is in accord with the well-known relative difference in the drying speed of oils containing these driers. The general results obtained are in agreement with the view that during drying peroxide formation occurs at the ethenoid linkages of the unsaturated acids of the glyceride with subsequent splitting of these peroxides into aldehydes which in turn are oxidised to acids. H. M. LANGTON.

Tung oil from Australian grown trees of Aleurites Fordii (Hemsley). A. R. PENFOLD (Tech. Museum, Sydney, 1926, Bull. No. 12. 9 pp.).—The yield and analytical constants of oils obtained from tung oil trees grown in New South Wales are given. Although the quantity of fruit obtained is rather small, the quality of the oil is at least equivalent to Chinese oil.

S. S. WOOLF.

**Cockchafer oil.** E. KOPP (Seifensieder-Ztg., 1926, 53, 800).—The raw material, dried by gentle heat, yields on extraction  $14 \cdot 2 - 16 \cdot 9\%$  of a thick reddish-brown oil of characteristic, not unpleasant smell, and having  $d^{15}$  0.925, acid value  $42 \cdot 5$ , saponif. value  $157 \cdot 5$ , ester value  $115 \cdot 0$ , Reichert-Meissl value  $1 \cdot 8$ , iodine value  $73 \cdot 7$ ,  $n_D^{20}$  1.4678. The isolated fatty acids are grey-green and soft, and have m.p. 31°, f.p. 27°. The dried residue from which the oil has been removed contains  $66 \cdot 9\%$  of protein, and can be used as cattle food. S. S. WOOLF.

Bromine-iodine values of fats. VAUBEL.— See XIX.

#### PATENTS.

Stabilising emulsions of wool oils and water. I. G. FARBENIND. A.-G., Assees. of FARBWERKE VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 246,867, 29.1.26. Conv., 29.1.25).—In the manufacture of the emulsions of oil and soap solution used for wool-softening (e.g., "softening oil," "spinning oil," "spike oil"), an aromatic, hydroaromatic, or aliphatic-aromatic sulphonic acid of high mol. wt. or a salt thereof is used as protective colloid. These stabilisers have higher protective power than mucilaginous substances, do not cause the formation of hardening crusts on drying, and are readily removed in the subsequent "scouring" process (cf. E.P. 169,962; B., 1923, 22 A).

S. S. WOOLF.

Emulsification of oils and fats and products made therefrom. R. DOUGLAS and H. G. LOESCH,

Assrs. to DOUGLAS PECTIN CORP. (U.S.P. 1,625,641, 19.4.27. Appl., 4.9.23).—A solution of pectin is used to stabilise oil or fat emulsions. S. S. WOOLF.

Manufacture of fatty acids from hydrocarbons. W. C. H. PATAKY and F. J. NELLENSTEIJN (U.S.P. 1,627,452, 3.5.27. Appl., 24.8.25. Conv., 27.8.24).— See E.P. 239,178; B., 1925, 929.

Detersive compound. J. F. MOSELEY (U.S.P. 1,627,446, 3.5.27. Appl., 1.9.21. Conv., 16.10.19).— See E.P. 162,691; B., 1921, 478 A.

Manufacture of perfumed medicated transparent soaps. P. VILLAIN (U.S.P. 1,626,708, 3.5.27. Appl., 31.10.24. Conv., 7.11.23).—See E.P. 228,282; B., 1925, 251.

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

Painting cement and plaster : problems involved in, and the results of, super-accelerated alkaliwater tests. H. A. GARDNER (U.S. Paint Manufrs. Assoc., Feb., 1927, Circ. No. 299, 65-124).-An account is given of a series of tests designed to simulate the action on paint of the strongly alkaline surfaces of damp, freshly-laid surfaces of cement and plaster. Each of 8 types of paint was applied over each of 16 types of primer, on fresh cement and plaster slabs, which were subsequently placed in water tanks to about two thirds of their thickness, the water seeping through the panels from the underside and meeting the painted surface charged with alkali. Full details of the composition of the coatings applied and of the behaviour of the panels after 1, 8, and 12 days' immersion and after air-drying and heating are tabulated, and photographs of typical panels reproduced. Further exposure tests on similar panels in warm, moist air and in cold air, and an examination of the water-resisting powers of the primers used are also described. The results are discussed, and the importance of suitable priming is stressed. General suggestions for treatment of cement, stucco, and plaster surfaces, exterior and interior, are put forward.

S. S. WOOLF.

Commercial flat white wall paints (lithopone type). E. F. HICKSON (U.S. Paint Manufrs. Assoc., Mar., 1927, Circ. No. 305, 204—213).—The following data are tabulated for 27 flat white wall paints of the lithopone type :—chemical analysis, consistency, brushing and levelling qualities, reflection and gloss, fastness to light, elasticity (Kauri reduction tests and various stoving and bending tests), behaviour on heating painted panels for 5 hrs. at 105°, keeping qualities, and durability (roof exposure for 3 months). The results are discussed with reference to proposed changes in Federal Specification No. 21 (U.S. Government Specification for flat interior lithopone paint). A relation between elasticity tests and exposures is indicated. S. S. WOOLF.

Covering power [of pigments] and degree of dispersion. F. HEBLER (Kolloid-Z., 1927, 41, 365— 371).—The conclusion reached by Kühn (B., 1926, 287) that the covering power of a pigment is not affected by the size of the particles, but only by their number, has been further investigated. A number of homo-disperse systems of graphite were prepared by subjecting a suspension of graphite to sedimentation, the coarser sediments being obtained by fractional elutriation, the finer by Perrin's method of fractional centrifuging (Koll.-Chem. Beih., 1909, 1, 251). The particle radius was determined by sedimentation analysis, using Stokes' formula, and also directly from the experimental results, using Svedberg's formula (A., 1924, ii, 96). Good agreement was obtained by the two methods. The permeability to light of the graphite suspensions was now measured on the optical bench, and it was found that for both reflected and transmitted light the absorption of light increases with increasing number of particles and with decreasing particle size (*i.e.*, increasing degree of dispersion). Kühn's results are thus not confirmed. L. L. BIRCUMSHAW.

Chemical mechanism of linseed oil drying. Evans, Marling, and Lower.—See XII.

#### PATENTS.

**Production of printing inks.** M. LUDWIGSEN (E.P. 268,966, 13.4.26).—Waste from cellulose manufacture, *e.g.*, sulphite lye or terpenes or these two substances containing small proportions of resin, is evaporated to a solid content of 40—60%, and glycerin or a glyceride (rendered viscous by the addition of oily matter, *e.g.*, wheel grease, petroleum jelly) is added. Linseed oil factice may replace part of the glycerin etc. Colouring matter is finally incorporated.

S. S. WOOLF.

Oxidised pine oil for pigments. J. K. SPEICHER, Assr. to HERCULES POWDER CO. (U.S.P. 1,626,362, 26.4.27. Appl., 20.7.26).—The pigment is ground in admixture with oxidised pine oil. H. ROYAL-DAWSON.

**Production of oil-varnish substitutes.** G. PETROV (G.P. 437,612, 26.3.24).—Phenols are condensed with formaldehyde in the presence of acidic or basic catalysts, and the products, after cooling to 40—50°, are mixed with naphthenic acids obtained by oxidation of naphtha or other mineral oils. The resulting semi-fluid substances are dissolved in alcohol and used as insulating varnishes, binding media for moulding compositions, etc.

#### S. S. WOOLF.

Preparation of condensation products of carbamide and formaldehyde. I. G. FARBENIND. A.-G., Assees. of W. GEISEL (G.P. 437,646, 18.11.24. Addn. to G.P. 416,252; B., 1926, 99).—During the curing process applied to the syrupy condensation products of carbamide or its derivatives with formaldehyde or its polymerides, hardening is accelerated and the tendency to clouding and even depositing is counteracted if the mass be subjected to the action of ultra-violet light.

#### S. S. WOOLF.

Treatment of condensation products of carbamide or its derivatives with formaldehyde. F. POLLAK (E.P. 266,389, 30.9.25. Conv., 1.10.24).— Hardened final condensation products of carbamide or its derivatives and formaldehyde are reduced to a fine powder mechanically or by boiling with mineral acid (of 10% strength) and cooling. The powder so obtained is softened by treatment with a small quantity of any of the solvents mentioned in E.P. 240,840 (B., 1926, 22), or of the soluble initial condensation products of the above-mentioned substances, and may then be moulded under the combined action of pressure and heat.

S. S. WOOLF.

Manufacture of resinous insulating and impregnating materials. SIEMENS-SCHUCKERTWERKE G.M.B.H. (G.P. 437,115, 2.4.25).—Small amounts of bases which do not affect the physical properties of these materials are added, in order to convert the water-soluble resinic acids into insoluble salts. The amount of base added is much less than the equivalent of the resinic acids.

J. S. G. THOMAS. **Purification of abietic acid.** G. H. DUPONT (U.S.P. 1,628,535, 10.5.27. Appl., 23.4.25. Conv., 28.4.24):— See E.P. 244,980 ; B., 1926, 202.

**Process of colouring materials.** J. P. TRICKEY, Assr. to QUAKER OATS CO. (U.S.P. 1,627,939, 10.5.27. Appl., 2.1.23. Renewed 23.3.27).—See E.P. 234,862; B., 1926, 642.

Production of resinous substances. CHEM. FABR. DR. K. ALBERT, G.M.B.H., Assees. of A. AMANN and E. FONROBERT (U.S.P. 1,623,901, 5.4.27. Appl., 4.2.24. Conv., 18.8.17).—See E.P. 259,030; B., 1926, 988.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

#### PATENTS.

**Rubber compositions.** F. C. DYCHE-TEAGUE (E.P. 268,853, 10.12.25).—The mixture of carbon and catalyst formed, *e.g.*, by the decomposition of carbon monoxide in the presence of finely-divided nickel or iron, is used as a compounding ingredient for rubber. The incorporation of 36 pts. with rubber (100 pts.), zinc oxide (10 pts.), sulphur (4 pts.), and diphenylguanidine (1 pt.), after vulcanisation, yields a product superior to one otherwise similar but containing gas black, in its greater toughness and decreased deadness. The mixture of carbon and catalyst is also more compact, more easily introduced into rubber, and expedites vulcanisation. D. F. TWISS.

Manufacture of rubber products. A. R. F. VAN DER MARK and H. KREMER (Swiss P. 116,630, 8.7.25. Conv., 11.7.24).—Unvulcanised coagulated latex is shaped into hollow or sac-like form and the walls are stretched until the desired thickness is attained; the product is then dried and can be vulcanised. Colours may be introduced into the latex before coagulation.

D. F. Twiss.

Vulcanisation of rubber. C. J. T. CRONSHAW and W. J. S. NAUNTON, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,627,636, 10.5.27. Appl., 28.6.26. Conv., 9.9.24).—See E.P. 244,159; B., 1926, 138.

Manufacture of a deterioration retarder for rubber. S. M. CADWELL, ASSR. to NAUGATUCK CHEMI-CAL CO. (U.S.P. 1,626,784, 3.5.27. Appl., 23.9.24).— See E.P. 240,407; B., 1925, 1000.

## XV.-LEATHER; GLUE.

Specific ion effects in the behaviour of tanning agents towards collagen treated with neutral salts. K. H. GUSTAVSON (Fourth Colloid Symposium Monograph, 1926, 79—101).—There is at present no sharp division between primary, secondary, and residual valencies in protein reactivity. Vegetable tannage is largely a function, under like conditions, of the state of hide powder as to secondary valency and specific surface forces. The one-bath chrome tanning process is essentially a primary valency reaction. There are three factors involved in the specific ion effects shown by very basic fresh chromium sulphate liquors with collagen treated with neutral salts: (1) a primary valency reaction between positively-charged chromium complexes or micelles and protein carboxyl groups, (2) formation of molecular compounds between complex chromium anions and basic protein groups, (3) adsorptive processes. CHEMICAL ABSTRACTS.

Testing glue. MEESS (Betriebsführung, 1926, 5, 120– 125; Chem. Zentr., 1927, I, 677).—Rudeloff's method (cf. B., 1918, 743 A) gives reliable results. The tenacity does not decrease regularly with increasing dilution of the glue; with increases from 100 to 150% and from 200 to 300% of water, the change in tenacity is only small, but it decreases rapidly as the water is increased from 150 to 200%. The humidity of the air has but little effect upon the tenacity, which also is only affected by prolonging the drying period from 24 hrs. to 48 hrs. when the weaker glue is used. Heating the glue at 40°, but not at higher temperatures, is advantageous with thick glue. L. A. COLES.

Preservation of gelatin emulsions. T. SABALITscнка and E. Böнм (Chem.-Ztg., 1927, 51, 301-302).-Gelatin solutions were treated with various preservatives in increasing amounts up to 0.1%, and observations made of the time required for mould to appear, for the mould to form a complete covering, and for the complete breaking down and liquefaction of the jelly. Boric acid and sodium benzoate showed a lower bactericidal action than benzoic and salicylic acids, but even in the case of the latter the formation of mould was only delayed and not entirely prevented. By the addition of 0.1%of methyl p-hydroxybenzoate the gelatin solutions could be preserved for periods varying from 40 to 71 days without the appearance of mould, or of such turbidity or coloration as occurred in the case of the other preservatives. F. R. ENNOS.

## PATENTS.

Manufacture of shaped articles of casein. A. BARTELS and O. MIECH (U.S.P. 1,623,085, 5.4.27. Appl., 24.3.26. Conv., 15.5.23).—Unhardened casein material, roughly moulded to the desired shape, is partially hardened by immersion in hexamethylenetetramine solution, dried till the moisture content is reduced to about 17%, rapidly softened by the action of heat, finally formed by pressing, and completely hardened and dried. T. S. WHEELER.

**Dyeing of leather.** M. C. LAMB (U.S.P. 1,628,160, 10.5.27. Appl., 9.6.25. Conv., 27.4.25).—See E.P. 255,555; B., 1926, 839.

Manufacture of basic chromium salts (E.P. 251,267).—See VII.

#### XVI.—AGRICULTURE.

Manuring in drills. H. BURK (Z. Pflanz. Düng., 1927, B6, 145-162).—In numerous field trials with sugar beet, superphosphate gave better results when drilled in with the seed than when sown broadcast. In parallel experiments with Rhenania phosphate, basic slag, and dicalcium phosphate the reverse proved to be the case. A. G. POLLARD.

Determination of the manurial requirement of soils for phosphate by means of the citrate method. O. LEMMERMANN, L. FRESENIUS, and LESCH (Z. Pflanz. Düng., 1927, B6, 163-178).-The customary determination of citric-soluble phosphate in soils is supplemented by a consideration of the "relative solubility," viz., the percentage of total phosphate which is soluble in 1% citric acid solution. Total phosphate is extracted with aqua regia and in highly organic soils preliminary treatment with nitric acid is recommended. The carbonate content of the soil is determined by means of Schiebler's apparatus, using citric acid. In preparing the citric acid extract of soils, sufficient acid is used to neutralise carbonates and give in addition a 1% solution of free acid. Soil and citric acid solution are shaken together for 6 hrs. followed by a further 2 hrs. on the subsequent day. Soils having more than 0.025% of citric-soluble phosphate do not usually need phosphate when the "relative solubility" is greater than 25%. Where the citric-soluble phosphate is less than 0.020%, a phosphate requirement is indicated. Between these two ranges the phosphate requirement is ill-defined and varies with the "relative solubility" figure. Detailed comparisons between the above method, pot experiments, field experiments, and the Neubauer method are recorded A. G. POLLARD. and discussed.

Laboratory and field experiments on the use of 3:5-dinitro-o-cresol and its sodium salt for winter spraying. C. T. GIMINGHAM and F. TATTERSFIELD (J. Agric. Sci., 1927, 17, 162—180).—Observations are recorded on the use of solutions of 3:5-dinitro-o-cresol and its sodium salt as sprays against the eggs of certain insects. Both substances are effective and, in addition, have a cleansing action on the bark of trees. The concentration used in the spraying experiments on trees was a 0.25% solution of 3:5-dinitro-o-cresol, or its equivalent in the case of the sodium salt.

G. W. ROBINSON.

Relationship between the chemical constitution of organic compounds and their toxicity to insects. F. TATTERSFIELD (J. Agric. Sci., 1927, 17, 181-208).-There is a rough correlation between both the mol. wt. and volatilities of organic compounds and their toxicity as contact insecticides, but the direct connexion is probably with some other property, such as adsorption. An account is given of the chemical compounds found in certain highly toxic tropical leguminous plants. The toxicity of a number of organic compounds to Aphis rumicis and to the eggs of Selenia tetralunaria is examined in relation to their chemical constitution. Substitution of certain radicals in the benzene ring affects toxicity, but the number substituted and their relative position also have an effect. 3:5-Dinitro-o-cresol has a powerful ovicidal effect. Toxicity increases in the fatty acids up to undecoic acid, after which it declines. Their physical properties only partially explain their toxic action.

G. W. ROBINSON.

Soil biodynamics. I. Microbiological characteristics of southern soils. S. KOSTICHEV, A. SHELOU-MOVA, and O. SHUL'GINA (Bull. BUR. Agric. Microbiol. [Russia], 1926, 1, 5—45).—In these soils there are two simultaneous processes : (a) the nitrogen-fixing organisms are active, while other groups of micro-organisms are decomposing the native rock and supplying mineral substances ; (b) mineralisation of organic residues increases the nitrogen content and supplies mineral constituents for the plant. In certain Crimean soils the nitrogen balance is maintained without the addition of nitrogen. CHEMICAL ABSTRACTS.

Soil biodynamics. II. Microbiological characteristics of the soils in the podsol region. 1. Fixation of atmospheric nitrogen. M. KORSAKOVA and V. BILINKINA (Bull. Bur. Agric. Microbiol. [Russia], 1926, 1, 47–72).—Cultivated soils maintain the highest number of *Azotobacter* and *Clostridium*.

#### CHEMICAL ABSTRACTS.

Soil biodynamics. III. Microbiological characteristics of the soils in the podsol region. 2. Energy of soil microbiological activities. M. KORSAKOVA and G. LOPATINA (Bull. Bur. Agric. Microbiol. [Russia], 1926, 1, 73-84).—No correlation was found between the energy of utilisation of mannitol and the presence of nitrogen-fixing organisms.

## CHEMICAL ABSTRACTS.

Availability of phosphates in calcareous or alkaline soils. J. F. BREAZEALE and P. S. BURGESS (Arizona Agric. Exp. Sta. Tech. Bull., 1926, 10, 209-237). -Neutral or acid soils in humid regions (especially in abundance of organic matter), but not calcareous or alkaline soils in semi-arid regions, may respond to applications of insoluble phosphates. Plants are able to absorb phosphorus readily from solutions containing as little as 0.1 in 106 parts. The presence of dissolved carbon dioxide increases the solubility and availability of the phosphorus. Tricalcium phosphate alone is of little value as a source of phosphorus. The slow reaction of iron and aluminium hydroxides with calcium phosphate is accelerated by carbonic acid alone, but retarded by carbonic acid and calcium carbonate. In a neutral soil containing no carbonic acid, in a black alkali soil, or in a soil containing excess of carbonic acid and calcium carbonate, the plant will be more favoured than the iron and aluminium hydroxides in the competition for the phosphorus in applied calcium phosphate. CHEMICAL ABSTRACTS.

Determination of the colloidal material in soils. G. J. BOUYOUCOS (Science, 1926, 64, 362).—The colloidal content of a soil can be determined in 15 min. by dispersing 50 g. of soil in a mortar, placing it in a tall cylinder with a total volume of 1050 c.c. of water, shaking vigorously for 2 min., and determining the density of the liquid by means of a hydrometer. The percentage of the material remaining in suspension at the end of 15 min. is equal to the percentage of the colloids as determined by the heat of wetting method. Only 3 of 31 soils gave abnormal results. The rate of settling can also be determined by the hydrometer method. A. A. ELDRIDGE. Rapid determination of the moisture content of soils. G. J. BOUYOUCOS (Science, 1926, 64, 651-652). —The soil is vigorously shaken with absolute ethyl alcohol, the mixture immediately filtered, and the change in density of the alcohol is determined by means of a hydrometer. The method is almost as absolute as the oven method. A. A. ELDRIDGE.

#### PATENTS.

Obtaining a culture mixture for fertiliser production. C. A. BAUMGARTEN-CRUSIUS (U.S.P. 1,626,579, Appl., 12.2.26. Conv., 23.1.23. Cf. E.P. 26.4.27. 258,149; B., 1926, 991).-The process comprises subjecting a small mass of decomposing soil, horse manure, and the like to a temperature of 60° for about 20 min. to destroy undesired organisms, then dividing the mass into two portions, sterilising one, dividing the other into two parts, and keeping these at a temperature favourable to the growth of bacteria, one part being under aerobic and the other under anaerobic conditions. The culture is then made by lixiviating the two parts, and adding the liquid to the sterilised portion of soil. This is then used for inoculating a mass of decomposable material consisting of soil, horse manure, beech leaves, and peat.

C. T. GIMINGHAM. **Production of fertiliser.** A. G. STILLWELL (U.S.P. 1,622,684, 29.3.27. Appl., 21.10.24).—Waste-water from fish- and meat-canning factories is freed from oil, evaporated to d 1.24—1.41, and the syrup thus obtained is mixed with calcium hydrogen phosphate and dried at 120°. T. S. WHEELER.

Combating injurious fungi. I. G. FARBENIND. A.-G. (G.P. 436,923, 13.2.23).—The poisonous action of thallium compounds on all types of fungi is much greater than that of other known inorganic poisons, such as mercuric chloride or cyanide. To prevent the growth of such organisms on wood, therefore, the latter may be impregnated with a solution of thallium sulphate containing 0.2 pt. per 1000, or a solution of another thallium salt, *e.g.*, the sulphite, oxalate, or sodium thallium thiosulphate, etc., of equivalent thallium content. A. B. MANNING.

## XVII.—SUGARS; STARCHES; GUMS.

Determination of starch in confectionery. GROSS-FELD.—See XIX.

## PATENTS.

Preparation of [pure] tricalcium saccharate. C. STEFFEN (E.P. 261,693, 4.1.26. Conv., 17.11.25).— Tricalcium saccharate is washed under high pressure in a press, and mashed with 7—8 times as much water or lime water. Lime is added until 0.4-0.5% of sugar remains in solution. The tricalcium saccharate suspension thus obtained is used for washing fresh tricalcium saccharate obtained by the precipitation of sugar from a molasses or impure sugar solution with lime powder. The washing is effected in the filter press. Tricalcium saccharate of at least 99% purity is obtained.

B. FULLMAN.

Manufacture of lactose. S. C. MEREDITH and N. N. T. NYBORG (U.S.P. 1,626,857, 3.5.27. Appl., 12.12.22).—Alkali is added to whey in excess of that required to neutralise the acid content, the whey is then heated gradually to coagulate and precipitate the albumin, and the alkali content is gradually decreased as the temperature increases. W. G. CAREY.

## XVIII.—FERMENTATION INDUSTRIES.

Clarification of wine. O. REICHARD (Z. Unters. Lebensm., 1927, 53, 163-168).-The clarification of wine by means of potassium ferrocyanide is described. When the treatment is correctly performed, by adding the exact amount of ferrocyanide, the whole of the salt occurs in the dregs, which normally are removed by filtration or by settling. The wine then contains only a trace of iron in solution, and when centrifuged gives a slight deposit of Prussian blue. If the wine is "overclarified," by adding too much of the "clearing salt," then a greenish opalescence is produced in the wine, Prussian blue is deposited on centrifuging, and free ferrocyanide and hydrocyanic acid are present in solution. This latter can be determined by making the wine strongly alkaline, warming it at 60°, and driving the hydrocyanic acid into silver nitrate solution by a stream of carbon dioxide. The correction of an excessive addition of ferrocyanide by adulteration with a wine rich in iron ("back-clarifying") is illicit, as the effects vary with the time which elapses between the "over-clarify-ing" and its correction. If carried out immediately, no ill-effects follow, but if the "over-clarified" wine is kept, the excess ferrocyanide decomposes and, on addition of the iron compound or of the wine containing iron, is precipitated leaving free hydrocyanic acid in solution.

#### H. J. DOWDEN.

Methyl alcohol contained in spirits prepared from grape and fruit residues. G. REIF (Z. Unters. Lebensm., 1927, 53, 168-171).-Methyl alcohol occurs in brandy from the decomposition, during fermentation, of the methyl ester of pectic acid present in the fruit skins. The author has examined the methyl alcohol content of numerous samples of grape and fruit brandies. In the manufacture of the fruit brandy the skins, stalks, etc. of apples, pears, and plums, after expression of the juice, are placed in oak casks and allowed to ferment, without addition of sugar or yeast. After fermentation, the liquid is distilled with addition of wood charcoal to absorb fusel oil etc. The refined spirit contains between 40 and 50% by vol. of alcohol. The method of manufacture has no influence on the methyl alcohol content, but, in general, that of spirit produced from the marc of grapes is lower than that of spirit from fruit. For grape brandy, the methyl alcohol forms 0.3-0.7% by vol. of the total liquid or 0.6-1.4% by vol. of the total alcohol, whilst for brandy prepared from fruits, the values are 0.4-0.9%, calculated on the total liquid, or 0.8-1.8% by vol. of the total alcohol.

#### H. J. DOWDEN.

Preservative values of green and of kiln-dried hops. A. C. CHAPMAN and C. W. McHugo (J. Inst. Brew., 1927, 33, 294).—The preservative properties of green hops during the ripening period do not increase, but, in a few cases, even diminish slightly. During kilning the reduction of preservative power is considerable. C. RANKEN.

#### PATENTS.

Recovery of glycerin from fermented molasses mash. [A], S. K. VARNES, [B], J. W. LAWRIE, ASSTS. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,626,986 and 1,627,040, 3.5.27. Appl., [A], 14.6.22. [B], 6.6.22).— (A) Unclarified concentrated slop is divided into minute portions and "exploded" into mist in contact with a hot gaseous carrier chemically inert at the temperatures necessary for the rapid vaporisation of the glycerin, which is subsequently condensed. (B) The unclarified concentrated slop is heated and atomised under a pressure of at least 30 lb./sq. in. (approx.) in contact with a gaseous carrier at a temperature at which the glycerin is vaporised. The evolved glycerin vapours are subsequently condensed. C. RANKEN.

Brewing pan heated by internal steam tubes. J. SCHAEFER (G.P. 437,481, 3.11.23).—The pan is fitted with a number of arched tubes lying in a common plane perpendicular to the axis of the stirrer, having their ends attached to the inner wall of the pan and connected up in a continuous network by curved tubes attached to the outer wall, these tubes being provided with means for drawing off condensed water. L. A. COLES.

Separating solids from liquids (E.P. 268,051).— See I.

Drying apparatus (E.P. 268,237).-See I.

**Preparations of blood and meat juice** (F.P. 609,540).—See XIX.

Extraction of cream of tartar (F.P. 579,472).— See XX.

#### XIX.—FOODS.

Bromine-iodine values of butter and other edible fats. W. VAUBEL (Z. Unters. Lebensm., 1927, 53, 151-154).-The author has shown (cf. B., 1926, 887) that bromine values of fat can be classified as primary and secondary. A tertiary value is now developed, this being the absorption of bromine by the fat or oil after contact with bromide-bromate solution for 2 days. For linseed oil, these three values were 127, 172, and 240 respectively. Butter-fat values varied between 36 and 49 for the primary, 51-70 for the secondary, and 70-100 for the tertiary. The saponified fats, however, showed distinctly lower values in all three cases. Olive oil has higher values for the saponified oil than for unsaponified. The iodine values of tallow show a decrease when the fat is saponified, whilst lard shows an increase. For a series of margarine samples, the values for the saponified fat, were invariably higher than those for the unsaponified, and it is suggested that this fact affords a means of distinguishing between butter and margarine.

#### H. J. DOWDEN.

Relation between the fat content of milk and that of the resultant cheese. S. Gov (Z. Unters. Lebensm., 1927, 53, 160—163).—It is shown that the food regulation, which requires in cheese a minimum fat content of 40% in the dry material, is not too stringent. Formerly no fat limit was stipulated, but "whole" milk had to be used. Figures have been collected over a period of 1 year, for over 100 samples of cheese from CL. XIX.-Foods.

39 different factories, showing the fat content of the original milk (determined at the factory) compared with that of the cheese (determined by the author). The lowest milk-fat figure of  $2 \cdot 4\%$ , which is extremely low for a mixed milk, gave a cheese of fat content  $42 \cdot 28\%$ . Normal "whole" milk, of fat content  $2 \cdot 7\%$  and over, produced cheese of  $47 \cdot 8\%$  fat content. The whole series shows that normal milk will always give cheese which will satisfy the requirements of the food regulations. H. J. DOWDEN.

Polarimetric determination of starch in confectionery products. J. GROSSFELD (Z. Unters. Lebensm., 1927, 53, 156-160).-The author shows that the method previously described (cf. A., 1917, ii, 223) is particularly suitable for the determination of starch in such materials as peach and apricot kernels used for the manufacture of marzipan. The method of heating at 3½ atm. pressure and converting the starch into dextrose by boiling with 25% hydrochloric acid involves considerable errors owing to the conversion of hemicelluloses into sugars. In the polarimetric method, to compensate for the 3% error introduced by insoluble matter and fat, the conversion factor in the formula is reduced from 5.444 to 5.28, and to compensate for sugar 0.18 is deducted from the difference in polarimeter readings. The formula then becomes :--Starch =  $(B - A - 0.18) \times 5.28\%$ . H. J. DOWDEN.

Determination of the vegetable constituents of marmalade. W. REIDEMEISTER (Z. Unters. Lebensm., 1927, 53, 81—100).—10 g. of the material under investigation are macerated with 200 c.c. of warm water, and, after settling, the supernatant liquid and diluted pulp are decanted, and examined under the microscope. Pips, skins, and hairs of a variety of fruits and berries are encountered, and even mites, which indicate that dried fruit has been used in the preparation of the preserve. The sediment is examined macroscopically. The author describes and illustrates photographically the characteristic structure, dimensions, and behaviour towards stains, of the epidermis, seeds, sacs, styles, etc. of a large number of fruits, berries, and vegetables, which are commonly found as adulterants.

## H. J. DOWDEN.

Storage of oranges. E. BOTTINI (Annali Chim. Appl., 1927, 17, 129-163).-Oranges keep best when stored in crates or small barrels in presence of ammonium carbonate or trioxymethylene or, preferably, the two together. Fruit stored in this way is unaltered in composition, but the superficial parts undergo appreciable hardening, owing to hydrolysis of the soluble pectin by the ammonia and the consequent formation of insoluble pectic substances (pectic acid), which intensify the connexion between the cells. The retardation of the evaporation of water from the fruit, thus effected, results in a drier atmosphere, and hence acts unfavourably on the development of micro-organisms. Moreover, methyl alcohol is liberated and undergoes oxidation to aldehyde, and then to formic acid, which also have preservative influence. T. H. POPE.

Determination of amino-acids in foodstuffs. J. TILLMANS and J. KIESGEN (Z. Unters. Lebensm., 1927, 53, 126-131).—The first method consists in titrating

with caustic soda to  $p_{\rm H}$  11.8 using tropæolin O as an indicator, and as a colour standard a buffer solution of pure caustic soda containing 0.00668 c.c. of N-caustic soda per c.c. and made normal in sodium chloride. The second method is based on the observations of Willstätter and Waldschmidt-Leitz (cf. A., 1922, ii, 169), that amino-acids can be determined alkalimetrically in strong alcoholic solutions. 5-10 g. of the material are dissolved in 250 c.c. of hot water, 0.3 g. of barium chloride is added, approximately neutralised, and then 10 c.c. of 2N-caustic soda solution are added. Ammonia is removed by aspirating air for  $2\frac{1}{2}$ —3 hrs., after which the solution is approximately neutralised with hydrochloric acid and diluted to 100 c.c. with 2N-sodium chloride solution, first treating with animal charcoal if the solution is dark coloured. As a comparison, determinations were made by Grünhut's formaldehyde method (cf. B., 1919, 788 A). For the tropæolin titration Grünhut's colorimeter is used (loc. cit.). Into one cylinder are introduced 30 c.c. of the solution, about 1.8 g. of sodium chloride to make it about normal, and 0.5 c.c. of a 0.1% aqueous solution of the indicator. Into the comparison cylinder are poured 30 c.c. of the buffer solution and 0.5 c.c. of the indicator. The amino-acid solution is titrated with 0.1N-caustic soda solution to the same tint as the buffer. The free alkali required to produce  $p_{\rm H}$  11.8 is titrated with 0.1N-hydrochloric acid solution using neutral red as indicator, and a buffer solution of  $p_{\rm H}$  7 as colour standard. The volume of free alkali is deducted from the amino-acid titration. For the alcoholic titration, 5 c.c. of acid and sodium chloride solution are mixed with 50 c.c. of 96% alcohol previously neutralised with 0.1N-caustic soda solution, and, after adding 0.5 c.c. of 4% alcoholic thymolphthalein solution, the mixture is titrated with 0.1N-caustic soda solution. A further 5 c.c. of the amino-acid solution are titrated to neutrality with either caustic soda or hydrochloric acid, using neutral red as indicator, the titer being added to, or subtracted from, the first titration according as acid or alkali is required to produce neutrality. The results obtained by all three methods were found to be in close H. J. DOWDEN. agreement.

Formaldehyde titration as a means of distinguishing artificial from natural foodstuffs. J. TILLMANS and J. KIESGEN (Z. Unters. Lebensm., 1927, 53, 131-137).-A solution of the substance under investigation is treated with caustic soda solution using phenolphthalein as indicator, until the solution is rosecoloured. To a 40% solution of formaldehyde containing a little phenolphthalein caustic soda solution is also added until the red colour appears. On mixing the two red solutions, the colour remains unchanged if an artificial material is present, or only one or two drops of caustic soda solution are required to restore the colour. When natural foodstuffs are under investigation, however, the colour at once vanishes on mixing the two red liquids and a more or less considerable volume of 0.1Ncaustic soda solution is required before the colour is restored. In the investigation of lemon juice, 10 c.c. of juice required over 100 c.c. of alkali to produce a coloration, and after addition of the formaldehyde solution a further 2 c.c. were required to restore the colour. A sample of natural juice which had been kept for 10 years required 83 c.c. of alkali to give the red tint and  $6\cdot15$  c.c. to restore it. Honey 20 g. dissolved in 100 c.c. of water required 1—3 c.c. of alkali to restore the red colour after the addition of the formaldehyde solution. One sample which gave a figure less than  $1\cdot0$  c.c. was concluded to be a mixture of pure and artificial honey. Numerous samples of vinegar were examined. For the natural product, the volume of  $0\cdot1N$ -caustic soda required for the restoration of the colour varied from 2 to 8 c.c., whilst for artificially prepared vinegars the colour remained unchanged. H. J. DOWDEN.

Cockchafer oil. KOPP.-See XII.

#### PATENTS.

Cooling of sterilised milk or other liquid in bottles. T. D. BOTTERILL (E.P. 268,907, 26.1.26).— The bottles, contained in crates, are passed by means of an endless conveyor, through tanks having successively lower temperatures. F. R. ENNOS.

**Preparation of milk chocolate.** GRUYÈRE USINES LAITIÈRES S.A. (F.P. 611,731, 25.2.26).—Milk, the fat content of which has been artificially increased, is pasteurised and evaporated *in vacuo* at a moderate temperature. The solid product, containing very little water, is powdered, mixed with the ordinary chocolate constituents, and worked into blocks etc.

F. R. ENNOS.

**Process for making cheese.** M. G. J. FOUASSIER (E.P. 268,889, 11.1.26).—A homogeneous cheese that is not "cooked" before ripening is prepared by crumbling the drained curds and turning them over from time to time during the ripening process. The ripened cheese is then melted and pasteurised or even sterilised by heating at 75—100° in the presence of 2% of sodium tartrate, precautions being subsequently taken to preserve it in an aseptic condition.

F. R. ENNOS.

Preparing fish foods. C. BIRDSEVE (E.P. 257,222, 8.3.26. Conv., 24.8.25).—The flesh of fresh fish, reduced to fragments, is placed in moulds, with or without the addition of a small amount of salt to express the protoplasm from the cells. While cold, the fish is subjected to pressure or stirring; it finally solidifies by the aid of the retained gluey content.

F. R. ENNOS.

Preparations of blood and meat juice. L. A. PARET (F.P. 609,540, 20.1.26. Conv., 22.1.25).—Fresh blood or raw meat juice, after treatment with aromatic substances, is allowed to ferment and the liquid, containing all the natural vitamins, is filtered, mixed with salt, and evaporated at a low temperature. The powdered product is used as a foodstuff by mixing with cooked flour, chocolate, etc., whilst the filtered liquid forms a drink. F. R. ENNOS.

Process for obtaining decomposition products from materials containing protein. E. JENA (E.P. 247,944, 10.2.26. Conv., 17.2.25).—Natural animalcovering substances, *e.g.*, hair, wool, horn, feathers, etc., consisting chiefly of keratin, are cleaned and freed from fat by dilute sodium carbonate solution, dried, and ground. The powder is hydrolysed by heating at 110° with alkali under pressure to a glue-like mass, from which an acid protein substance is precipitated by addition of acid. The filtrate on treatment with excess of alkali yields an alkaline protein substance. The products are purified by re-precipitation, and washed with water under pressure. F. R. ENNOS.

Removing sulphur dioxide and compounds from food products. L. ROSENSTEIN (U.S.P. 1,623,070, 5.4.27. Appl., 18.3.26).—The fruit is treated, either by dipping or spraying, with an aqueous solution of hydrogen peroxide containing sodium bicarbonate and allowed to dry, the adhering solution oxidising the sulphur dioxide to sulphuric acid. The strength of the hydrogen peroxide solution is adjusted according to the amount of sulphur compound to be removed.

F. R. ENNOS.

Manufacture of condensed milk. J. R. HATMAKER (U.S.P. 1,626,818, 3.5.27. Appl., 6.4.26. Conv., 5.11.25). —See E.P. 254,941; B., 1926, 766.

Preservation of food for men or animals. T. SABA-LITSCHKA (U.S.P. 1,627,342, 3.5.27. Appl., 8.4.25. Conv., 17.4.24).—See F.P. 596,843; B., 1926, 383.

Fertiliser (U.S.P. 1,622,684).—See XVI.

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

Australian synthetic menthol. A. R. PENFOLD (Chim. et Ind. [Compt. rend., 5th Congrès Chim. Ind., 1925], 1927. 2 pp.).—A review of the preparation and properties of synthetic menthol made by the electrolytic reduction of piperitone, with a note on the relative toxicities of synthetic and natural menthols (cf. B., 1922, 78 A; 1924, 926). E. H. SHARPLES.

[Determination of] mercury in mercuric salicylate. I. GRIFFITH and P. P. RAMANUSKAS (Amer. J. Pharm., 1927, 99, 242—244).—The proposed method consists in dissolving  $2 \cdot 5$  g. of the sample in 25 c.c. of nitric acid, heating the solution, and cautiously adding 20 c.c. of sulphuric acid. When the reaction subsides, the liquid is boiled until nearly colourless, a further 10 c.c. of nitric acid are added, and the boiling is continued until the yellow colour disappears. The cooled solution is diluted to 250 c.c., and mercury determined either volumetrically by adding ferric ammonium sulphate and titrating with 0.1N-potassium thiocyanate solution to a permanent yellowish-red colour, or gravimetrically by precipitation as mercuric sulphide. W. J. POWELL.

Italian petit-grain oils. M. A. ALBRICCI (Parf. moderne, 1926, 10, 252–253; Chem. Zentr., 1927, I, 655).—Oils from the leaves and young twigs of bergamot, lemon, mandarin, and bitter-orange trees grown in Calabria had the following characteristics. Bergamot:  $d^{15}$  0.8991,  $\alpha_D - 5^{\circ}$ , saponif. value 199.73, acetyl value 213.69, acid (as acetic acid) 0.12%, esters (as linalyl acetate) 69.9%, free alcohols (as linalol) 3.8%, citral 2.10%, constituents containing oxygen 75.8%, nonvolatile residue on the water-bath 4.18%, solubility in 70% alcohol 1 in 3 vol., yield of oil 0.34%. Lemon:  $d^{15}0.8749, \alpha_{\rm D}+23.31^{\circ}$ , saponif. value 28.87, acetyl value 130.10, citral 26.90%, esters (linalyl acetate) 10.12%, non-volatile residue 7.53%, solubility in alcohol at  $85^{\circ}$ 1 in 8 vol., yield of oil 0.143%. Mandarin:  $d^{15}0.9775$ ,  $\alpha_{\rm D}+12^{\circ}$ , saponif. value 179.20, methyl anthranilate 58.95%. Bitter orange:  $d^{15}0.8971, \alpha_{\rm D} - 6.12^{\circ}$ , saponif. value 214.6, esters (linalyl acetate) 75.10%, citral 0.49%, non-volatile residue 1.99%, solubility in alcohol at  $80^{\circ}$ 1 in 1.3 vol., yield of oil 0.204%.

#### E. H. SHARPLES.

**Pulegone oil.** P. LIOTTA (Riv. Ital. delle essenze e profumi, 1926, 8, 103; Chem. Zentr., 1927, I, 655).— Oils from the fresh and dried plants had:  $d^{15}$  0.9394 and 0.9389,  $\alpha^{20} + 26^{\circ}$  6' and  $+ 26^{\circ}$  5', acid values 0.30 and 0.31, esters (as menthyl acetate) 0.90% and 0.95%, free alcohols (as menthol) 9.25% and 9.12%, saponif. values 237 and 242, ketones 68.75% and 70% and both were soluble in 2 vols. of 70% alcohol.

## E. H. SHARPLES.

Acetone oils. H. SUIDA and H. POLL (Z. angew. Chem., 1927, 40, 505-507).-Yellow acetone oil, b.p. 60-250°, was purified by washing with 5-10% hydrochloric acid and subsequent distillation over lime, thereby undergoing an 8% loss. The resulting mixture was fractionated under reduced pressure, the fractions being collected at intervals of 3°. By formation of the bisulphite additive products, the methyl ketones were separated from the higher ketones. Further fractionation or investigation of the oxidation products enabled the constituents of those fractions of b.p. 60-152° to be identified as : acetone, methyl ethyl ketone,  $\gamma$ -methylbutan- $\beta$ -one, pentan -  $\beta$  - one,  $\delta$ -methylpentan -  $\beta$  - one,  $\gamma$ -methylpentan- $\beta$ -one, hexan- $\gamma$ -one, hexan- $\beta$ -one, mesityl oxide,  $\beta$ -methylhexan - $\gamma$ -one,  $\gamma$ - ethylpentan -  $\beta$ -one,  $\gamma$ -methyl- $\Delta\gamma$ -hexen- $\epsilon$ -one, and heptan- $\beta$ -one. Distillation of synthetic mixtures of the calcium salts of butyric and acetic acids showed that mixed ketones as well as the symmetrical compounds result. The bearing of these facts on the origin of the unsymmetrical ketones in the wood-distillate is discussed. No evidence for the existence of cyclic ketones in the heavy acetone oil was obtained (cf. Pringsheim and Bondi, A., 1925, i, 1072). L. M. CLARK.

Essential oils of Eriostemon Coxii (Mueller) and Phebalium Dentatum (Smith). A. R. PENFOLD (J. Proc. Roy. Soc. N.S.W., 1926, 60, 331-344).-The leaves and terminal branchlets were steam-distilled in each case and the pale yellow, mobile oils, having an odour resembling that of the passion fruit (Passiflora edulis) had the following characteristics. Eriostemon Coxii:  $d_{15}^{15}$ 0.8794 - 0.8810,  $\alpha_{\rm D}^{20} + 20.75^{\circ}$  to  $+ 22.6^{\circ}$ ,  $n_{\rm D}^{20} 1.4600 - 1.4600$ 1.4637, ester value 94.1-116.6, ester value after acetylation 120.0-133.5, solubility in 80% alcohol by wt. 1 in  $3\frac{1}{2}$  -  $8\frac{1}{2}$  vol. The average yield of oil was 0.55%, and the principal constituents were d-x-pinene, an olefinic terpene (ocimene), butyl isovalerate, amyl isovalerate, linalol (?), geraniol, citronellol, and darwinol, both free and combined as isovalerates and caproates, sesquiterpene (cadinene), with small quantities of sesquiterpene alcohol, phenolic substances, and a paraffin of m.p. 64-66°. Phebalium Dentatum: d15 0.8704-

0.8776,  $\alpha_{20}^{20} + 18.5^{\circ}$  to  $+ 20.25^{\circ}$ ,  $n_{20}^{20} \cdot 1.4626$ — 1.4666, ester value 64.4—90.3, ester value after acetylation 114.1—129.2, solubility in 80% alcohol 1 in 6— 7 vol. The average yield of oil was 0.21% and the principal constituents were *d*- $\alpha$ -pinene, an unidentified terpene, butyl and amyl butyrates and *iso*valerates, geraniol and citronellol free and as butyrate, caproate, and formate, with small quantities of citral, sesquiterpene, sesquiterpene alcohol, phenolic substances, and a paraffin of m.p. 65— $66^{\circ}$ . E. H. SHARPLES.

Germicidal values of some Australian essential oils and their pure constituents, together with those for some essential oil components and synthetic substances. IV. A. R. PENFOLD and R. GRANT (J. Proc. Roy. Soc. N.S.W., 1926, 60, 167-170; cf. B., 1926, 804).-Rideal-Walker coefficients and the physical constants of Australian and Indian sandalwood oils, oil from Zieria macrophylla, zierone, isomenthol, phloroacetophenone dimethyl ether, hydrocinnamaldehyde, hydroxycitronellal, and a range of aliphatic aldehydes and alcohols of the C8-C12 series are given. The aldehydes and alcohols have relatively high coefficients, i.e., 1% suspensions in  $7\frac{1}{2}$ % rosin soap solution and in alcohol gave the following respective values;  $C_8$ -aldehyde, 16 and 22;  $C_8$ -alcohol, 25 and 26. These values decreased with increase in the number of carbon atoms. The dispersion varied according to the medium used, some being more highly dispersed in alcohol than in soap solution, and vice versa. The germicidal effect of the alcohol itself is negligible with those substances having coefficients over 4. E. H. SHARPLES.

Use of 3 : 5-dinitro-o-cresol for spraying. Giming-HAM and TATTERSFIELD.—See XVI.

Toxicity of organic compounds. TATTERSFIELD. —See XVI.

#### PATENTS.

Manufacture of aromatic derivatives of formamide. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 269,302, 13.3.26).—Arylformamides are produced by the action of carbon monoxide (or gases rich in it) on primary or secondary aromatic amines, at high pressures and temperatures of 70—200°, in the presence of catalysts such as formic acid, formates, formic esters, formamide or its derivatives, alkali metals or their hydroxides or alkoxides, etc. *E.g.*, formanilide is formed by the action of carbon monoxide at 200 atm. pressure on aniline, at 180—200°, in the presence of sodium and methyl alcohol. B. FULLMAN.

Manufacture of alkyl formates. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 268,890, 11.1.26).— Gas mixtures containing 40-45% of carbon monoxide (e.g., water-gas), preferably freed from moisture, carbon dioxide, iron carbonyl, and sulphur compounds, are passed under pressure into an aliphatic alcohol at a suitable temperature. The residual gas, containing only 20% or less of carbon monoxide, is suitable for further reactions, e.g., production of methyl alcohol, catalytic hydrogenations, ammonia synthesis, the remainder of the monoxide being removed in the usual manner if desired. Water-gas, for example, is washed with water and alkali and passed through anhydrous methyl alcohol at 200 atm. pressure to remove moisture, and then through pressure vessels at  $70^{\circ}$  containing methyl alcohol and a little sodium methoxide, to give methyl formate. The gas may be distributed through the alcohol by means of a perforated flat spiral pipe or a perforated false bottom. Alternative forms of apparatus are described. The vessels may be tin-lined or enamelled, but copper, iron, and nickel are unsuitable. *n*-Butyl formate is similarly prepared. The matter of E.P. 252,848, and 254,819 (B., 1926, 646, 721) is disclaimed. C. HOLLINS.

Conversion of alkaline-earth formates [into oxalates]. O. P. C. BREDT, ASST. to TROJAN POWDER CO. (U.S.P. 1,622,991, 29.3.27. Appl., 7.4.21. Renewed 12.1.24).—Calcium formate is treated with sodium hydroxide solution, and the sodium formate obtained is converted by heat into sodium oxalate, which is transformed into calcium oxalate by treatment with calcium hydroxide from the first stage of the process.

#### T. S. WHEELER.

Preparation of solid, water-soluble, basic aluminium acetate and formate. CHEM. FABR. GRÜNAU LANDSHOFF & MEYER A.-G., and E. FRANKE (G.P. 437,637, 16.10.21).—Aqueous solutions of aluminium acetate or formate are evaporated in the form of finelydivided sprays. Even at 130° completely water-soluble products are obtained. C. HOLLINS.

Preparation of benzoic acid and benzoates. G. BLANC (F.P. 586,383, 7.8.24).-Benzaldehyde is added gradually to a dry, well-stirred mixture of a catalyst (silver, platinum, nickel, copper, nickel oxide, cobalt oxide, or salts of these) and a carrier (sand, kieselguhr, powdered carbon, or anhydrous salts, such as sodium sulphate, chloride, or carbonate), air or oxygen being passed in at the same time. The temperature rises rapidly and is maintained at 100°. The powdery product is dissolved in water, and a little permanganate is added so long as decolorisation occurs. The mixture, neutralised with sodium carbonate, gives a quantitative yield of sodium benzoate on evaporation. In the example given, nickel carbonate and anhydrous sodium carbonate are used. C. HOLLINS.

Decolorisation and purification of lactic acid. H. GOUTHIÈRE (F.P. 582,402, 6.9.23).—Nitric acid (40%) is slowly added to crude lactic acid at 85—90°, the temperature not being allowed to exceed 105°. Powdered charcoal is added to complete the decolorisation and to remove the last traces of nitric acid. The lactic acid and charcoal are filtered off through a press and separated by dissolving in water. C. HOLLINS.

Extraction of citric acid. F. TALLADA (F.P. 570,557, 5.9.23. Conv., 13.12.22).—The juice of citrons, acid oranges, green mulberries, and other acid fruit is boiled for a short time with zinc or a zinc salt. The precipitated zinc citrate is dissolved in very dilute citric acid and electrolysed, giving zinc, oxygen, and a more concentrated citric acid solution. The latter is electrolysed again with more zinc citrate until a saturated citric acid solution is obtained, which is then evaporated. The

zinc citrate may alternatively be dissolved in very dilute mineral acid before electrolysis. C. HOLLINS.

Purification of salicylic acid. DEUTSCHE GASGLÜH-LICHT-AUER-GES. M.B.H. (G.P. 437,924, 29.11.24).—On addition of an aqueous solution (e.g., 5%) of a soluble salt of thorium, zirconium, or the cerite- or yttria-earth metals to crude sodium salicylate solution (containing, e.g., 10% of salicylic acid), the impurities are precipitated with the first portions of the rare-earth salicylate. The bulk of the salicylic acid is obtained pure by acidifying the filtrate. The rare earth is dissolved out of the impure precipitate with the calculated quantity of dilute acid, and used again. C. HOLLINS.

Production of alkyl- and aralkyl-resorcinols. H. HIRZEL, Assee. of W. SCHILT (E.P. 256,225, 24.7.26. Conv., 30.7.25).-An aldehyde and a ketone, one of which contains a 2:4-dihydroxyphenyl group, are condensed together in presence of 10% sodium hydroxide, and the resulting unsaturated ketone is reduced. The hydroxyl groups may be protected by alkylation or acylation. The reaction is particularly useful for the synthesis of 2:4-dihydroxy-1-hexylbenzene, m.p. 65-66°, b.p. 185-188°/12 mm., from 2:4-diethoxyacetophenone by condensation with n-butaldehyde or crotaldehyde, followed by reduction with hydrogen and nickel to 2:4-diethoxyphenyl amyl ketone, further reduction by Clemmensen's method to 2:4-diethoxy-1-hexylbenzene, and de-ethyla-C. HOLLINS. tion with hydrobromic-acetic acid.

Extraction of cream of tartar. V. POPOFF (F.P. 579,472, 8.3.24).—The grape residues (marc of grapes) are treated, one part with alkali (preferably sodium carbonate), the rest with acid (preferably sulphuric acid). To the acid portion potassium chloride is added, and the acid and alkaline portions are stirred vigorously together. Cream of tartar, 95—97% pure, separates in good yield. C. HOLLINS.

Preparation of monomethylarylamines. E. MERCK CHEM. FABR., Assees. of H. MAEDER and W. KRAUSS (G.P. 437,975, 13.9.24).-N-Methylenearylamines, or their polymerisation products, are reduced with the calculated quantity of hydrogen in presence of Thus Na metal catalyst at ordinary temperature. methylene-p-aminophenol is reduced in alcoholic solution with hydrogen and palladised carbon, and the acidified filtrate is evaporated under reduced pressure to give a Methylaniline is salt of N-methyl-p-aminophenol. obtained in almost quantitative yield by catalytic reduction of anhydroformaldehyde-aniline in alcoholic C. HOLLINS. solution.

**Preparation** of  $\beta\beta'$ -dihalogeno-ethers. W. SCHOELLER (G.P. 437,159, 8.7.23).—Olefines are shaken, preferably under pressure, with a concentrated aqueous suspension of mercuric sulphate; the resulting dimercurated products are converted into mercurihalogen compounds, which are finally treated with halogen in a non-hydrolysing solvent. An aqueous suspension of mercuric sulphate, shaken vigorously with ethylene under pressure in a vessel previously evacuated, absorbs the gas very rapidly, and on addition of aqueous potassium iodide to the filtered liquid  $\beta\beta'$ -bisiodomercuridiethyl ether is obtained in 95% yield, with a little  $\beta$ -hydroxy- $\beta'$ iodomercuridiethyl ether. By the action of iodine in warm ethyl acetate the product is converted into  $\beta\beta'$ -di-iododiethyl ether, b.p. 93—94°/1 mm. The dichloro- and dibromo-derivatives are similarly prepared. Propylene or higher olefines may also be used. C. HOLLINS.

Manufacture of guaiacol. I. GUBELMANN, H. J. WEILAND, and O. STALLMANN, ASSTS. to NEWPORT Co. (U.S.P. 1,623,949, 5.4.27. Appl., 30.9.25).—A solution of diazotised o-anisidine in aqueous sulphuric acid is added, with normal sodium phosphate solution to boiling copper sulphate solution. Guaiacol is obtained in 95% yield. T. S. WHEELER.

Preparation of thebaine derivatives. C. H. BOEHRINGER SOHN, CHEM. FABR., Assees. of SCHÖPF (G.P. 437,451, 12.7.24).-Thebaine, dihydrothebaine, or their salts are treated in presence of alcohols with nitrosating reagents (amyl nitrite, methyl nitrite, nitrosyl chloride, nitrososulphonic acid, etc.). To thebaine hydrochloride, suspended in a mixture of absolute alcohol, chloroform, and amyl nitrite at 0°, 10% alcoholic hydrogen chloride is added dropwise. After dilution with water and extraction with ether, the aqueous layer is separated and made alkaline with 2N-sodium hydroxide. A sodium salt crystallises out at 0°, and is decomposed with carbon dioxide, giving a new oxime, C22H28O5N2, m.p. 230-234°. From the filtrate from the sodium salt, ether extracts an alkali-insoluble by-product, and treatment with carbon dioxide then precipitates an isomeric oxime, m.p. 238-240° (decomp.). Ethyl nitrite or nitrosyl chloride gives the same two oximes, the formation of which seems to proceed according to the equation:  $C_{19}H_{21}O_3N + NOCI + 2EtOH =$  $C_{22}H_{28}O_5N_2 + H_2O + MeCl.$  If methyl alcohol be substituted for ethyl alcohol, there is obtained an oxime, C20H24O5N2, m.p. 240-242° (decomp.). Dihydrothebaine, m.p. 160-161°, gives with chloroform, absolute methyl alcohol, amyl nitrite, and methyl-alcoholic hydrogen chloride, a resinous oxime (hydrobromide, m.p. 240-245°; methiodide, m.p. 235-240°). The products have therapeutic application. C. HOLLINS.

Preparation of derivatives of 1-phenyl-2:3dimethyl-5-pyrazolone. I. G. FARBENIND. A.-G., Assees. of K. Böttcher and F. Stolz (G.P. 436,519, 16.5.23. Addn. to G.P. 423,028; B., 1926, 611).—Deriva-CMe=—C·NRR' tives of the type (I) 1 are obtained NMe·NPh·CO

by the action of alkylating, hydroxyalkylating, or aralkylating agents upon compounds (I) in which R' = H, R being alkyl or aralkyl. The methylation of 4-benzylamino- and the benzylation of 4-methylaminoderivatives are excluded. Compounds (I) are described in which  $R = CH_2Ph$ , R' = Et, m.p.  $62-64^\circ$ ; R=Me, R' = allyl, m.p.  $53-54^\circ$ ;  $R = CH_2Ph$ , R' = allyl, m.p.  $58-60^\circ$ ;  $R = R' = CH_2Ph$ , R' = allyl, m.p.  $58-60^\circ$ ;  $R = R' = CH_2Ph$ , m.p.  $92-93^\circ$ ; R = Me,  $R' = CH_2 \cdot CH_2OH$ , m.p.  $85-87^\circ$  (using ethylenebromohydrin);  $R = CH_2Ph$ ,  $R' = CH_2 \cdot CH_2 \cdot OH$ , base oily, hydrochloride, m.p.  $178^\circ$  (using ethylene oxide); R = benzyl,  $R' = CH_2 \cdot CH(OH) \cdot CH_2 \cdot OH$ , base oily, hydrochloride, m.p.  $128-130^\circ$  (using glycide); R = methyl,  $R' = CO_2Et$ , m.p.  $119-120^\circ$  (using ethyl chloroformate). The products are antipyretic, and are less toxic than the 4-benzylmethylamino-compound.

C HOLLINS.

Making therapeutic materials. E. SILTEN (U.S.P. 1,626,562, 26.4.27. Appl., 5.12.24).—Derivatives of 2-arylquinoline-4-carboxylate anhydrides are dissolved in a solution of hexamethylenetetramine in proportions such that the product contains more than 1 mol. of hexamethylenetetramine per mol. of anhydride. Products for intravenous injection are made from a solution of a hexamethylenetetramine derivative (a) of 2-arylquinoline-4-carboxylic acid containing 6 mols. of amine per mol. of acid, and (b) of phenylcinchophenic acid, molar ratio as above, and having a ratio water : hexamethylenetetramine = 73:27 by wt. B. FULLMAN.

Acylamidohydroxyphenylarsenious oxides. I. G. FARBENIND. A.-G., Assees. of L. BENDA (U.S.P. 1,622,271, 29.3.27. Appl., 19.6.25. Conv., 3.7.24.—5-Chloro-3acetamido-4-hydroxyphenylarsinic acid is readily reduced, by treatment with sulphur dioxide in presence of concentrated hydrochloric acid to which sodium iodide has been added, to 5-chloro-3-acetamido-4-hydroxyphenylarsenious oxide, which is of therapeutic value. T. S. WHEELER.

Manufacture of a diuretic product. P. CACGIA (U.S.P. 1,617,701, 15.2.27. Appl., 7.8.25).—A mixture of secretin and amylase in dilute hydrochloric acid is treated with a small quantity of toluene or sodium fluoride, and heated at 40° for 1 day. The filtered solution is of use as a hypodermic injection.

T. S. WHEELER.

Preparation of cucurbocitrin. I. S. BARKSDALE (U.S.P. 1,626,321, 26.4.27. Appl., 8.7.26).—Powdered water-melon seeds are treated with slightly alkaline water at 60°. The liquid is concentrated, treated successively with basic lead acetate and hydrogen sulphide, and filtered. The filtrate is treated with ether and dry alcohol in excess, the resultant precipitate being collected and dried. B. FULLMAN.

**Production of crystallised perfumes.** G. JAKOVA-MERTURI (F.P. 610,673, 5.2.26).—Tartaric acid and either magnesium, potassium, or sodium chloride or a mixture of the three salts are added to the perfume alone or in alcoholic solution, and then a second alcoholic solution of the perfume containing an alkali carbonate is added. *E.g.*, sodium bicarbonate is added to both solutions, and to the mixture is added a concentrated solution of sodium or potassium silicate.

E. H. SHARPLES.

Simultaneous synthesis of methyl alcohol and liquid hydrocarbons. G. PATART (E.P. 247,932, 28.1.26. Conv., 23.2.25).—See F.P. 594,121; B., 1927, 346.

Synthetic production of higher alcohols. Simultaneous manufacture of methyl alcohol and liquid hydrocarbons by synthesis. G. PATART (E.P. 247,177-8, 28.1.26. Conv., 7.2.25).—See F.P. 593,648—9; B., 1927, 347.

Reduction of halogenated hydrocarbons. A. F. S. BELLONE, ASST. to Soc. CHIM. DES USINES DU RHÔNE (U.S.P. 1,627,881, 10.5.27. Appl., 28.1.24. Conv., 21.11.23).—See E.P. 225,174; B., 1925, 613.

Preparing a reaction product of acetaldehyde and aniline. S. M. CADWELL, ASST. to NAUGATUCK Снемісаь Со. (U.S.P. 1,627,230, 3.5.27. Appl., 26.5.23). —See E.P. 216,478; В., 1924, 848.

Manufacture of metal iodide-alkali iodide compounds of therapeutic value. H. TRUTTWIN (U.S.P. 1,627,359, 3.5.27. Appl., 28.6.23. Conv., 3.7.22).—See G.P. 397,828; B., 1924, 967.

Production of a chloride capable of reaction from pinene or purified turpentine oil. F. HöнN (Е.Р. 269,624, 11.1.26).—See G.P. 426,865 ; B., 1927, 378.

Mixtures for generating carbon dioxide (G.P. 437,113).—See VII.

Preparation of [pure] tricalcium saccharate (E.P. 261,693).—See XVII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Law of blackening of the photographic plate at low densities. E. A. BAKER (Proc. Roy. Soc. Edinburgh, 1926, 47, 34-51).-The results given in the previous paper (B., 1925, 569) are extended for isochromatic and blue-sensitive plates, and correspond with those found for panchromatic plates. When the density is low p depends only on the exposure time. The value of p was not affected by wave-length as was found in the case of panchromatic plates. A theory of the deviations from the reciprocity law is discussed. In this connexion it is stated that it need only be considered that the deposit formed on full development is a measure of the action which has taken place. For the purposes of the theory, quanta are treated as molecules, obeying the law of mass action, and it is shown that two quanta are concerned in the photographic action, the two being absorbed within a short interval of time, giving the effect of two distinct absorptions when the exposure is short, and of a simultaneous absorption when the exposure is long. The two quanta must be taken up by different components of the absorber, and the maximum wave-lengths effective in the two stages must be close to the maximum for any photographic action. Either component may absorb the first quantum. The following formula is developed :  $y = n (1 - e^{-2\hbar^2 It/k})$ , where y is the number of molecules of the latent image, n is the number of double absorbers, and h and k are constants. It is considered that a single molecule of latent image is sufficient to make a grain developable. The formula is compared with results of Slade and Higson (B., 1921, 27 A). The assumption of one double absorber in a grain gives good agreement with the observed values, whilst the assumption of a large number in each grain is untenable. The fact that in the case of short exposures to intense light p rises above unity is ascribed to reversal, the theory of which is examined. It is assumed that reversal involves the absorption of four quanta. To explain certain discrepancies between theory and the observed results in the case of process plates, it is assumed that the two components of the double absorber are not exactly similar, and an equation is evolved which

gives good results in the case of process plates. A similar formula can be developed on the assumption that there are two different kinds of absorber present, having similar components. W. CLARK.

Comparative study of a glycine and a metolquinol developer. L. LOBEL and J. LEFÈVRE (Bull. Soc. Franç. Phot., 1927, 14, 5-9).—Measurements show that the stated difference between the results of development in glycine and in metol-quinol is only an apparent difference, the two developers giving identical results as far as the final result is concerned. A glycine developer in 15 and 20 min. gave characteristic curves identical with those obtained with a metol-quinol formula in 2 and 4 min. By suitable dilution of the metol-quinol solution, the two developers gave practically identical results in equal development times. Identity of action was attained if a little potassium bromide was added to the metol-quinol to depress fog. W. CLARK.

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Colour photography or kinematography. L. DUFAY, and Soc. ANON. COMP. D'EXPLOIT. DES PROC. DE PHOT. EN COULEURS L. DUFAY (E.P. 262,386, 28.7.26. Conv., 4.12.25).—A selecting screen for plates or films for colour photography consists of two contiguous and separable multi-colour screen layers of different relative intensities of coloration, arranged so that similar coloured parts of each layer coincide exactly. The screen layers consist of a thin sheet of celluloid coated with a layer of transparent gelatin, which is dyed with dyestuffs having an affinity for both the gelatin and the celluloid, so that the celluloid is dyed to any desired intensity after the gelatin has been dyed throughout its thickness to any other desired intensity. The other side of the celluloid is coated with emulsion. The face of the celluloid is prepared to receive the gelatin coating by treating with ether, ethyl acetate, or acetone, so as to regulate the intensity of coloration of the celluloid. A substance such as baryta may be introduced into the emulsion so as to render it reflective. After the photograph has been taken, the gelatin selecting screen is washed off.

W. CLARK.

Development of photographic plates, films, and the like. SILVER SPRINGS BLEACHING & DYEING Co., LTD., and A. J. HALL (E.P. 268,155, 22.4.26).—2:4-Diaminodiphenylamine is used as a developer for photographic plates etc. either alone or in conjunction with other developers such as metol and amidol, the usual methods of application being employed.

A. J. HALL.

Production of multi-coloured bromoil transfer impression prints. F. SCHMID (E.P. 257,940, 2.9.26. Conv., 2.9.25).—A print is made in black from a bromoil matrix in the known way, and is then painted over with a water colour or tempora colour, dried, and then coated with a transparent resin varnish. The varnish is dried and a further print is then made, or else a number of prints alternating with coatings of varnish. A suitable varnish consists of an alcoholic solution of shellac, sandarac, and the lac known as "Zapon." W. CLARK. Reversal process for direct positives. Ica A.-G. (G.P. 437,598, 26.2.24).—The developed silver image is treated with a mixture of lead nitrate and potassium ferricyanide, and the second exposure is given before the second development. The process can be used for paper, glass, or film images, and also for line and half-tone exposures. W. CLARK.

Process for obtaining direct positives by reversal. G. E. ZELGER, Assr. to PATHÉ CINÉMA, ANC. ÉTABL. PATHÉ FRÈRES (U.S.P. 1,628,445, 10.5.27. Appl., 14.4.23. Conv., 16.2.23).—See E.P. 211,431; B., 1924, 579.

Process of preparing sensitive layer on a suitable base. M. P. SCHMIDT and W. KRIEGER, Assrs. to KALLE & Co., A.-G. (U.S.P. 1,628,279, 10.5.27. Appl., 19.6.25. Conv., 28.5.24).—See E.P. 234,818; B., 1925, 692.

## XXIII.—SANITATION; WATER PURIFICATION.

Waters with a solvent action on iron and manganese, the rôle of carbonic acid in metallic corrosion, and the determination of free carbonic acid in water. H. LÜHRIG (Gas- u. Wasserfach, 1927, 70, 381-389) .- The mechanism of the corrosive action of natural waters on metals is discussed and illustrated by examples from the author's practice. All waters exert a solvent action on iron or manganese in virtue of their hydrogen-ion content. In general, the free carbonic acid, determined by titration or by its solvent action on marble, gives a practical measure of the corrosive action of a water on metals, but exceptional cases may arise. Hard waters containing calcium bicarbonate can exert a solvent action on iron owing to the ease with which the bicarbonate decomposes giving free carbonic acid, although here the deposition of a layer of calcium carbonate on the metal may act as a protective layer against further attack. Water samples containing free carbonic acid readily lose carbon dioxide unless they are kept in bottles completely filled. In determining free carbonic acid by the action of the water on marble the usual time of contact can be very considerably shortened by shaking and then filtering before determining the hardness. A convenient and rapid method of determining free carbonic acid is based on its removal by the passage of a current of carbon dioxide-free air through the water, and titrating before and after. The method fails for waters with a high temporary hardness, but the corrosive action of such waters is of little importance. If necessary, however, they could be diluted with carbonic acid-free water before making the determination.

## A. B. MANNING.

Treatment of water. J. H. HARRINGTON, Assr. to G. W. ROBB (U.S.P. 1,623,342, 5.4.27. Appl., 2.2.24).— An apparatus for determining the chlorine content of treated water consists of three adjacent troughs, through which the water is passed in parallel, and in one of which it is treated with potassium iodide and starch. The colour so produced is compared with a

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maximum and minimum depth of colour formed in the other troughs by means of blue glass plates.

## T. S. WHEELER.

Distilling apparatus. Water still. E. V. OLIVER (U.S.P. 1,625,222—3, 19.4.27. Appl., [A], 3.10.21. Renewed 6.10.24. [B], 4.12.23).—(A) A still comprises a casing which contains a well at the bottom, and a condenser and means for collecting condensate. A reservoir communicates with the well, and has a supply pipe brought in from above with a float valve; it has an open top, but has a lid of hinged sections meeting round the supply pipe with a fluid-tight flanged joint. (B) A condenser comprises vertical chambers alternately open at the top for cooling fluid and at the bottom for condensate. The cooling fluid is supplied by a many-branched pipe to the bottoms of the cooling fluids chambers. B. M. VENABLES.

Automatic [base exchange] water softener. C. P. EISENHAUER, Assr. to DURO Co. (U.S.P. 1,625,198, 19.4.27. Appl., 12.10.25).—A water softener comprising containers for softening material and for regenerating material is arranged so that the amount of water flowing through makes or breaks an electrical circuit which controls a needle valve that allows water pressure to reach hydraulically-operated valves which effect the change from softening to regeneration and back again. B. M. VENABLES.

Water-softening apparatus. K. W. BARTLETT (U.S.P. 1,624,382, 12.4.27. Appl., 7.6.20).—The water passes through a heater to a settling tank of two chambers connected at their lower ends, from the second of which it passes through a valve to a filter, and through another valve to the service pipe. A connexion, with pump and valve, is also provided from the filter back to the first settling chamber, to which chamber the reagent is also added. B. M. VENABLES.

Apparatus for distributing water to be purified at different depths in the clarifier. BAMAG-MEGUIN A.-G. (G.P. 437,043, 17.3.25).—The apparatus conveying the water into the clarifier consists of two perpendicular cylinders, one fitting tightly inside the other, provided with sets of openings so arranged that by rotating one of the cylinders those at a desired depth are brought opposite to one another, thus admitting the water at that depth. L. A. COLES.

Apparatus for de-aerating water. J. SIM (Can.P. 261,617, 22.5.25).—The apparatus comprises a series of compartments each provided with an inlet and outlet, the last compartment being fitted with means for removing air and water vapour. Each inlet is provided with a valve controlled by a float inside the compartment it serves. L. A. COLES.

Water purifier in which the water is heated with steam and then filtered. ATLAS-WERKE A.-G. (G.P. 436,882, 11.7.25).—Continuous operation of the plant is rendered possible without the provision of an intermediate storage tank, by using water which has already been purified for injecting the fresh water into the apparatus. L. A. COLES.