# BRITISH CHEMICAL ABSTRACTS B. - APPLIED CHEMISTRY

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# L-GENERAL; PLANT; MACHINERY.

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Air-screening method for determining the graincomposition of powders. H. W. GONELL (Z. Ver. Deut. Ing., 1928, 72, 945-950; Chem. Zentr., 1928, ii, 1128).-An air-screening device is described.

A. A. ELDRIDGE.

Dephlegmation. K. V. KOSTRIN and N. M. AKOPOV (Neft. Choz., 1927, 13, 311-316).—A description is given of the work performed by the following apparatus: a distilling flask carries a vertical tube ( $30 \times 300$ mm.) insulated with asbestos (50 mm.) connected by a U-tube to the top of a reflux condenser jacketed with steam. The lower orifice of this condenser is connected by a T-piece to a Liebig condenser or to a gas-trap which can be connected either with a second Liebig condenser or directly to the still. CHEMICAL ABSTRACTS.

Manometer for determination of gases in vapours. D. F. OTHMER (Ind. Eng. Chem. [Anal.], 1929, 1, 46-47).-In measurements with steam it is often important to detect admixture with non-condensible gases. For such work, and particularly for use in boilers, a manometer has been applied, of which one arm dips below water level, and the other terminates in a bulb surrounded by steam. This bulb contains air sufficient to give a pressure of about 400 mm. (Abs.), sealed by water, and mercury is used as the indicating liquid. When no air is present in the steam, the bulb functions as an air thermometer and gives its maximum deflection, but the addition of air to the steam tends to equalise the pressure on both sides of the manometer.

R. H. GRIFFITHS. Apparatus for gasometric determinations in technical analysis. J. HÜBSCHER (Süddeut. Apoth-Ztg., 1928, 68, 447-448; Chem. Zentr., 1928, ii, 1466).

# PATENTS.

Furnaces. A. W. BENNIS (B.P. 302,471, 19.11.27).-The space beneath a firegrate is divided into at least three compartments with separate air supplies, hot air is supplied to the middle compartment(s), cool or medium air to the front, and cool air to the back. The air supplies may be controlled merely by dampers, or separate fans may be used for forced draught. Two air heaters may be used in tandem, the air to the front passing through one only, and to the middle through both. B. M. VENABLES.

Furnace and method of treating bodies therein. W. G. BERGMAN, Assr. to D. P. OGDEN and E. F. PLUMB (U.S.P. 1,701,223, 5.2.29. Appl., 18.4.24).-A firing chamber is arranged between the inlet and outlet ends of a tunnel-like chamber through which a helicoidal flow of gases is maintained from the outlet end to the inlet end. The escape of flowing gases from the chamber to the open air is regulated at points arranged along the chamber between the firing chamber and the inlet.

cating motion by means of an internal de-

J. S. G. THOMAS. Regulation of combustion air in furnaces or the like. Askania-Werke A.-G. (B.P. 287,132, 21.2.28. Ger., 16.3.27).-The fuel is supplied by a number of independently adjustable mechanical devices, each of which operates a measuring fan or pump which draws from a common main, so that the total amount of air or fluid withdrawn will be proportional to the total fuel burnt. In the main is a constriction, the pressure drop across which is caused to regulate the supply of com-B. M. VENABLES. bustion air.

Retort. S. B. MATHEWSON and W. K. JAMISON, Assrs. to trustees of LECAR CARBON Co. (U.S.P. 1,700,684, 29.1.29. Appl., 15.2.24).-The apparatus comprises an oven chamber, a furnace communicating with the upper part thereof, flues connecting the chamber to a second chamber below the floor of the first, and an escape stack for the heated gases passing from the lower chamber. A system of piping in the upper part of the oven chamber communicating with a conduit leading outside the chamber can be detachably connected to a number of A. B. MANNING. containers.

Rotary drum for drying machines. K. FRASER, and W. J. FRASER & Co., LTD. (B.P. 302,541, 10.5.28) .--A cylindrical drum is divided by radial partitions into a number of segmental compartments, other shorter radial partitions are provided, and both sets carry lifter blades. Lengthwise the drum may be divided into zones, the partitions in one zone being staggered with reference to the next. A heating jacket may be provided and the heating fluid from the jacket may pass into the interior of the drum in direct contact with the material. B. M. VENABLES.

Drying of paper or other material. H. W. Cowan (B.P. 279,816, 6.10.27. Can., 29.10.26) .- The drying rolls, or other machine from which the moisture evolved normally passes into the atmosphere, is surrounded by a cover, to which heated air is supplied and from which air is exhausted in greater amount, so that some air is withdrawn from the room. Provision may be made for recirculating some of the air withdrawn, with or without condensation of moisture and re-heating. B. M. VENABLES.

Grinding and mixing apparatus. D. ANDERSON & SON, LTD., and R. O. CHILD (B.P. 302,561, 20.7.28) .--Two co-axial propellers have their blades of opposite inclination and are rotated at different speeds. They are submerged in a vessel of the material to be treated and work in conjunction with fixed breaker bars of various B. M. VENABLES. shapes.

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CL. I.-GENERAL; PLANT; MACHINERY.

Apparatus for grinding and crushing material. J. K. BLUM (B.P. 288,527, 10.4.28. U.S., 8.4.27).— The apparatus comprises an annular hammer member co-operating with a stationary casing and anvil member. The annulus is given a combined rotating and reciprocating motion by means of an internal element rotating eccentrically, which may be a toothed pinion meshing with teeth on the interior of the annulus. The space between the annulus and the casing diverges in all directions from the point where impact takes place.

B. M. VENABLES. Mills for grinding, crushing, or pulverising material. A. SONSTHAGEN and G. M. POVERUD (B.P. 303,227, 15.10.27).—During the progress of grinding the material (e.g., cocoa) is well exposed to the air so as to permit the escape of gases. The grinding surfaces may consist of a number of suitably spaced bars, so that the material is alternately ground between bars and deflected in a direction inclined to the general rotation. B. M. VENABLES.

Combined drying and pulverising apparatus. SIMON CARVES, LTD., Assees. of Soc. ANON. POUR L'UTILISATION DES COMBUSTIBLES (B.P. 297,119, 12.9.28. Fr., 16.9.27).—The rotating shell of a grinding machine is surrounded by a fixed casing forming an annular space through which a current of hot gas and the material to be ground are passed before entering the grinding chamber. The annular space may be divided into two by a cylindrical envelope which rotates with the grinding machine, the material and hot gas then being caused to make two longitudinal passes before entering the grinding chamber. Movement of the material is effected by inclined blades, and means are provided to cause the hot gas to be evenly distributed over the circumference.

B. M. VENABLES. Multi-stage apparatus for mixing, stirring, emulsifying, etc. A. L. MOND. From I. G. FARBEN-IND. A.-G. (B.P. 301,701, 12.5.28).—The liquid passes through slots in the wall of the axial inlet conduit, then through moving paddle rings and fixed guides alternately in a radial direction. B. M. VENABLES.

Apparatus for use in the treatment of solids with liquids. W. H. CAMPBELL (B.P. 304,387, 25.10.27).—The pulverulent solid is charged on to a number of diaphragms when in a moderately inclined position, and is subjected to leaching or other treatment while the diaphragms are horizontal; it is discharged by inclining the diaphragms very steeply, preferably one at a time. During charging, the lowest one or two diaphragms are reserved empty until the end; they then receive the material which was retained in the feed passages. B. M. VENABLES.

Apparatus for the production and dispersion of solids in liquids. W. H. WHATMOUGH (B.P. 304,178, 14.10.27).—Amorphous aggregates are dispersed in a liquid by means of an apparatus comprising a fluid-tight casing in which a beater is rotated at high speed with large clearances and is preferably eccentric to the casing. The beater may be constructed of a number of thin sheets of metal with fine teeth, or of a number of layers of wire gauze from the edge of which wire strands project, or of alternate layers of each.

B. M. VENABLES.

Mixing solids suspended in liquid. DUNCAN STEWART & Co., LTD. From P. HIRSCHFELDER (B.P. 303,339, 11.7.28).—A number of segmental compartments are arranged round a central discharge chamber, an annular feed launder being formed in the wall between the two. A pulp containing several grades of solids is supplied to one compartment, say no. 1, where the coarser settles and the fine material overflows to no. 2, and so on for several compartments. The feed is then charged to no. 2, so that coarser material is deposited on top of the finer. When discharge is required the contents of the compartment are agitated by compressed air and run out through the central discharge chamber.

B. M. VENABLES.

Apparatus for pneumatic separation or grading of solid materials. WOODALL-DUCKHAM (1920), LTD., and A. McD. DUCKHAM (B.P. 302,400, 8.10.27).—The air from a grinding apparatus is cleaned before admission to the fan in an apparatus designed on the principle of sudden expansion followed by gradual contraction. B. M. VENABLES.

Separation of solid materials. H. KOLITSCH (B.P. 275,970, 2.8.27. Austr., 14.8.26).—In the "handpicking" process of separation of materials, especially raw mining products, the controlling factor is differing behaviour to ordinary unaltered light. In this invention ordinary unaltered light is excluded, but all other forms of radiation are claimed to be able to control a separation of individual pieces of the mixture, through alreadyknown devices such as thermo-elements or selenium cells. The radiation may come from an outside source, or may be produced by the minerals themselves, *e.g.*, by heating. B. M. VENABLES.

Separators [for solids and liquids]. F. H. LUENSE (B.P. 289,847, 28.2.28. U.S., 4.5.27).—The pulp is thrown from the rims of a number of rotating bowls upon the inner surface of a cylindrical screen, through which the liquid passes and upon which the solids are retained. The solids falling down the screen are lifted and thrown upon it again by means of a rotating, inverted cone at the bottom. The rotating parts are driven by a motor through a vertical shaft, and the whole rotating assembly and frame of the motor is supported only by a swivel at the top; the frame of the motor is prevented from turning by a resilient device. B. M. VENABLES.

Linings for grinding, refining, and mixing machines. S. BRAMLEY-MOORE (B.P. 298,899, 11,10.28, U.S., 15.10.27).—A method of construction is described by means of which the interior lining of a mill may be formed from round, easily-renewable rods.

B. M. VENABLES.

Filter presses. F. B. LEHMANN (J. M. LEHMANN) (B.P. 297,354, 5.12.27. Ger., 19.9.27).—A form of filter press for very high pressures embodying the use of coiled metallic strip as the filter medium is described.

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Prevention of caking of crystals, powders, etc. A. C. Scorr (B.P. 305,721, 11.11.27).—The crystals, e.g., of sodium nitrate for use as a fertiliser, are mixed intimately with about 1—5% of a finely-divided insoluble silicate, e.g., asbestos or its mixtures with talc or mica. L. A. COLES. Absorbing and recovering the vapours of volatile liquids. RUTH-ALDO CO., INC., Assees. of H. L. BARTHELEMY (B.P. 282,792, 28.12.27. Fr., 28.12.26).— The vapours are absorbed in a wash liquid which readily foams; e.g., acetone vapour is removed from air by means of a solution of ammonium oleate and glycerin in soft water. B. M. VENABLES.

Apparatus for cooling and hardening fluid substances. S. FYHN (B.P. 302,398, 17.9.27).—The substance is spread in a thin layer on the interior wall of a vertical cylinder, which is surrounded by an annular jacket. Cooling is effected by the evaporation of a liquid within the jacket, the cooling liquid being caused to flow in a thin layer on the opposite side of the same wall, and pockets or ribs are provided to promote evaporation. B. M. VENABLES.

Evaporating ovens. CHEM. WERKE VORM. H. &. E. ALBERT (B.P. 283,495, 23.12.27. Ger., 11.1.27).—An evaporator, of the type where the liquid passes through a tube rotating in an oven, is provided with another oven and tube above the first through which the waste gases pass and in which the liquid is preheated.

B. M. VENABLES. Vacuum distillation. C. R. BURCH, F. E. BANCROFT, and METROPOLITAN-VICKERS ELECTRICAL CO., LTD. (B.P. 303,078-9, [A] 21.9.27 and 12.3.28, [B] 21.9.27).-Materials such as mineral oils are subjected to distillation in vacuo at the lowest possible temperature consistent with commercial speed of operation, whereby cracking and the formation of azeotropes are reduced. The material is distilled from a number of shallow pools each provided with a cooled condensing surface a short distance away, so arranged that practically all the molecules leaving the pools will strike the condensing surface. The distance between the pools and their condensers may be of the order of 1 cm., and is in any case less than the mean free path of the molecules of residual gas (other than the distillates) at the pressure employed, which may be of the order of 1 dyne/cm.<sup>2</sup> B. M. VENABLES.

Carrying out exothermic catalytic gas reactions and apparatus therefor. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,192, 23.9.27).-The catalyst is contained in a number of tubes of a bundle, the incoming gas is passed first outside the tubes and then through them, and the resistance to flow through the catalyst must be less than the resistance to flow outside the tubes. The tubes are, therefore, very closely spaced, and if round tubes are used the spaces between may be partly filled with inert material such as iron rods, but it is simpler to use square, hexagonal, or other prismatic tubes. When such tubes are used, the tube walls may touch everywhere, in which case some of them will be left empty for the first (heat-interchange) flow and others filled with catalyst for the return (reaction) flow. In a convenient construction the tubes are held in a tube plate at one end only, and are tapered at that end to allow plenty of space for distribution of incoming B. M. VENABLES. gas.

Removal of solid particles from flue gases. M. PRÉTOT and F. ULLMANN (B.P. 281,651, 28.11.27. Fr., 4.12.26).—The washing liquid such as soda solution is sprayed by compressed air upon turbine-like blades of rotors which are of frusto-conical shape, each inverted with respect to its neighbour. B. M. VENABLES.

Method for obtaining a perfect high vacuum. S. LOEWE (B.P. 280,560, 10.11.27. Ger., 11.11.26).— After evacuation, but prior to sealing-off, a valve-like device within the evacuated bulb is moved to obstruct the stem leading to the pump, so that the gases evolved from the glass when heated for sealing almost entirely move in the direction of the pump. The valve-like device may be of mica, or of glass with an iron or metal tail which is manipulated by a magnet.

B. M. VENABLES.

Manufacture of friction material for braking and coupling purposes. KIRCHBACH'SCHE WERKE KIRCH-BACH & Co., and M. K. KIRCHBACH (B.P. 284,269, 26.1.28. Ger., 26.1.27).—A mixture of fibrous material and phenolic condensation product is subjected to a pressure in a mould exceeding 3000 atm.; the heat of compression causes pre-hardening sufficient to prevent warping in the subsequent drying and final hardening by heat.

B. M. VENABLES.

Mixing machine. W. F. DEHUFF, Assr. to GLEN MIXER Co., INC. (Re-issue 17,215, 12.2.29, of U.S.P. 1,656,665, 17.1.28).—See B., 1928, 175.

Absorption refrigerating apparatus. Electro-Lux, Ltd., Assees. of Platen-Munters Refrigerating System Aktiebolag (B.P. 281,710, 3.12.27. Ger., 4.12.26. Addn. to B.P. 270,319).

Absorption refrigerating systems. Electrolux, Ltd., Assees. of A. Lenning (B.P. 283,474, 9.1.28. U.S., 8.1.27).

Processes of refrigeration. M. HIRSCH (B.P. 290,305, 12.5.28. Ger., 12.5.27).

Sorting of granular materials. K. C. APPLEYARD, P. W. BEWICKE, (SIR) J. F. LAYCOCK, M. R. PORTALL, and W. E. MANNERS (B.P. 305,608, 5.11.27).

Joints of pipes or other vessels adapted to withstand fluid pressure. VICKERS, LTD., and D. M. ANDERSON (B.P. 305,609, 7.11.27).

Coating of materials (B.P. 304,812). Separation of gaseous mixtures (B.P. 288,216).—See II.

# II.—FUEL; GAS; TAR; MINERAL OILS.

Yorkshire, Nottinghamshire, and Derbyshire coalfield. South Yorkshire area. Parkgate seam. ANON. (Dept. Sci. Ind. Res., Fuel Res., Survey Paper No. 13, 1929, 64 pp.).—The Parkgate seam has been examined throughout the South Yorkshire area by means of detailed analyses of pillars of coal cut from the entire thickness of the seam, and taken at 16 points well distributed over the area. The seam consists of three main divisions, "tops," "middle coal" or "hards," and "bottoms." The "tops" and "bottoms" consist of bright coal of the normal bituminous type, and the "hards " mainly of durain with some bright coal in the middle. A portion of the seam adjacent to the roof and floor is sometimes of inferior quality and is not mined. Of the dirt bands associated with the seam the middle dirt band occurs with the most regularity, and shows a

gradual increase in thickness from the centre of the area northwards to beyond Barnsley. The seam exhibits no marked gradation of properties across the South Yorkshire area, but slight differences are noticeable between sections taken in the north and south respectively. The moisture contents of the former are comparatively low, whilst those of sections taken from the east of Sheffield are comparatively high. The percentage of ash, in general, is very low. The volatile matter is comparatively high. The average sulphur content is less than 2%; it is nowhere under 1%, and in one sample reaches 3.17%. The phosphorus content is rather high (up to 0.03%) in certain sections. The calorific value of the seam is high, averaging 14,430 B.Th.U./lb. The m.p. of the ash is also comparatively high, varying from 1320° to 1400°. The cokes obtained in the Gray-King assay were highly swollen in all but three samples. The ash and volatile matter in the "tops" and "bottoms" are generally higher than in the "hards," which contain considerably less sulphur but generally more phosphorus than the rest of the seam. The banded constituents, vitrain, clarain, durain, and fusain, have been separated and analysed in each section. The suitability of the coal for gas making, steam raising, and the production of coke for metallurgical purposes is briefly discussed. A. B. MANNING.

Brown coals and bituminous coals of Hessen, and their utilisation. A. SANDER (Brennstoff-Chem., 1929, 10, 21-26).-The brown coals of Hessen, which are obtained from both underground and surface workings and in deposits up to 60 ft. thick, have a high water content (of the order of 60%) and an ash content varying from 2.5 to 14.9%. On low-temperature carbonisation they yield considerable quantities of tar (about 17-26%, on the dry material), which can be satisfactorily "berginised." Two low-temperature carbonisation plants, of the Kohlenveredlung A.-G. type and having a daily throughput of 600 and 1000 tons of brown coal, respectively, have recently been erected in Hessen, the semi-coke being used for generating power or for domestic purposes. This area also yields coals with a lower bitumen content, which are used directly for firing, and a lignite which is low in ash and gives a fairly robust coke on high-temperature carbonisation.

W. T. K. BRAUNHOLTZ.

Classification of coal. S. W. PARR (Univ. Illinois Eng. Exp. Sta. Bull., 1928, No. 180, 59 pp.).—A discussion of various classifications and the fundamental factors involved. The following formulæ (where Ais the ash and S the sulphur) are put forward: pure coal substance ("unit coal")=1.00 — (1.08A + 0.55S); unit B.Th.U. = (indicated B.Th.U. -5000S)/[1.00— (1.08A + 0.55S)]; "unit volatile matter" = determined volatile matter — (0.08 + 0.4S)/(1.08A + 0.55S). CHEMICAL ABSTRACTS.

Swelling of coals. II. G. LAMBRIS (Brennstoff-Chem., 1929, 10, 44-50; cf. B., 1928, 916).—If coal is heated rapidly in the usual crucible tests an outer crust of coke is quickly formed which prevents further swelling. This has led to the erroneous supposition that greater swelling is obtained with more gradual heating. If the coal (1 g.) is spread on a layer of kieselguhr in a crucible

placed in a vertical tube furnace at 900° free swelling takes place and a residue of coke up to 17 cm. high is obtained. The swelling power determined by this method is about three times that found by the Bochum method, using coal of the same degree of fineness, and is still greater if the coal is more finely powdered. The swelling power is also several times greater with rapid heating than with gradual heating, but bears no relation to the coke yield. The volume of the coke is determined by immersing the piece in a mixture of paraffin wax (2 pts.) and liquid paraffin (3 pts.) at 60°, removing, wiping with cotton wool, and, when cold, measuring the displacement in water. The volumes found in duplicate determinations of swelling power agree, on an average, to within 8%. W. T. K. BRAUNHOLTZ.

Use of the Berthelot-Mahler calorimeter. V. KOSTJEJEV (Arh. Hemiju, 1929, 3, 27-29).—Powdered fuels such as coke, coal, and charcoal may, when not compressible into tablets, be burned in starch capsules, the heat of combustion of which is about 3600 g.-cal./g. A supply of these capsules may be kept in hermetically closed jars. R. TRUSZKOWSKI.

Origin and decomposition of carbon disulphide in gas making. II. Carbon-sulphur complex. W. J. HUFF and J. C. HOLTZ (Proc. Amer. Gas Assoc., 1927, 1431-1435; cf. B., 1928, 77).-When a gas oil rich in sulphur was cracked at 650°, carbon disulphide could not be detected in 2 litres of gas; at 730° it was detected, and at 980° considerable quantities were found. Possibly carbon surfaces, when exposed (at temperatures employed in gas manufacture) to gases containing sulphur, at first absorb sulphur, thereby assisting the decomposition of sulphur compounds and decreasing the exposed carbon surface; saturation is approached, and finally carbon disulphide is evolved. The decrease of the tendency of heated refractory surfaces to decompose sulphur compounds is due to the formation of a carbon-sulphur complex, as well as to the presence of carbon. Coke heated in hydrogen behaves like clean pumice, no carbon disulphide being formed at 730° except on continuous use. With higher cracking temperatures deposition of carbon and dissociation of sulphur compounds increase. Conditions favouring the fixation of high local concentration of sulphur, such as sudden heating with decreased diffusion, reduction of exposed surfaces, and the rapid removal of carbon disulphide when formed, favour the production of carbon disulphide. Any process giving the gas extended contact with heated surfaces sufficiently low in, or free from, sulphur will decrease the concentration of carbon disulphide in the gas or eliminate it. CHEMICAL ABSTRACTS.

Removal of tar fog from gases. A. FISCHER (Chem. Fabr., 1929, 101—102).—In laboratory research on the carbonisation of lignite it is desirable to have a tar-fog separator which is complete in its action, causes no back-pressure, and does not involve the risk of interruption of an experiment. To fulfil these conditions a small Cottrell-Möller apparatus was designed. It consisted of a  $50 \times 5$  cm. steel tube chromium-plated, internally flanged at each end to large Jena flasks, and having an axial wire. It is driven from the ordinary lighting circuit, an oil transformer giving a direct P.D. of 10,000 volts between the wire and the tube, the latter being earthed. The throughput of gas may vary between 10 and 1500 litres/hr. without the tar separation being disturbed. C. IRWIN.

"Gasol "from carbonisation gas, and use of the gas for cutting and welding. H. SCHOLTZ (Chem.-Ztg., 1929, 53, 159).—The mixture of ethane, ethylene, and higher homologues known as "gasol" was used for welding two sheets of metal 3 mm. thick and 250 mm. long. With a gasol pressure of 0.048-0.05 atm. and oxygen pressure 0.9 - 1.0 atm. the operation took 6 min. and was accomplished without extinction of the flame or heating of the burner. In other experiments lower pressures and a mixture with 20% of acetylene were successfully used, the weld in the latter case surviving a 180° bending test. Operating costs with the mixture were lower than those with acetylene or gasol alone, but if the original carbonisation gas is used the economy of its employment depends on the thickness of material R. H. GRIFFITH. and on the type of burner.

Pyrogenic decomposition of wood tar in presence of hydrogen and under pressure. V. N. IPATIEV and A. D. PETROV (Ber., 1929, 62, 401-407).-The behaviour of wood tar (Kahlbaum) and of a specimen richer in pitch when heated at about 450° in hydrogen under pressure and in the presence of the oxides of iron and aluminium has been investigated. The total yield of liquid products of pyrolysis is greater than that obtained by distillation with phosphoric acid, and the proportion of hydrocarbons and fractions of low b.p. is higher. The amount of unsaturated hydrocarbons so produced is markedly lower than that obtained by cracking in the absence of hydrogen. Short heating with hydrogen decomposes phenolic ethers, unsaturated cyclic ketones, and acids with predominating formation of aliphatic hydrocarbons and phenols, the latter being produced by decomposition of the ethers and ketones. Protracted treatment with hydrogen reduces the phenols and saturated cyclic ketones to hydrocarbons of the aromatic and polymethylene series. The most favourable conditions for the formation of liquid products consist in the use of an excess of hydrogen in the presence of catalysts at 440-460°. The fractions of high b.p. from the products of pyrolysis are free from the unpleasant odour of the original wood bar; they do not darken when preserved, and may be used as solvents, extractives, or lubricants. In preliminary experiments it is shown that cyclohexanone at 420-440° gives much cyclohexane and small amounts of condensed products. Homoisophorone at 460-480° yields phenols and hydrocarbons, the proportion of the latter increasing as the experiment is prolonged. Pyrocatechol diethyl ether affords benzene and phenol at 430°, whereas palmitic acid at 460° yields aliphatic hydrocarbons.

H. WREN.

Influence of dusty filling materials on the plastic and elastic properties of bituminous substances. H. SUIDA and A. SCHMÖLZER (Petroleum, 1929, 25, 251—254).—For characterising the binding materials used in road construction, the sp. gr., softening point, penetration, and ductility were measured; the filling materials were limestone, quartz, granite, brick,

and asbestos in particles less than 0.065 mm. in diam., which gave a surface of about 2000 cm.<sup>2</sup>/g. Mixtures containing 5-70% of filler were prepared and tested for ductility, hardness, and ability to bend. The bending test employed a prismatic piece, 15 cm. long and of 4 cm.<sup>2</sup> cross-section, which was rested on two supports 11 cm. apart and observed under a load of 500 g. In the ball-hardness test a loaded steel ball, 1.8 cm. in diam., was used. It was found necessary to choose suitable temperatures, loads, and times for the different series of binding materials in order to reveal their behaviour most strikingly, so that no direct comparison of all the results could be made. It appeared, however, that increasing amounts of filler altered plastic and elastic properties continuously, granite and quartz giving very similar results. The effects of different fillings on the results of all the tests are discussed for petroleum, coaltar, and asphalt pitches; in coal-tar pitch there are components capable of reaction with substances like chalk, so that its behaviour is different from that of other pitches where such compounds are not present. Particular properties observed with asbestos and brick dust are ascribed to the physical nature of the surfaces R. H. GRIFFITH. involved.

Rapid determination of sp. gr. of semi-solid bituminous substances. S. E. BERKENBLIT (Ind. Eng. Chem. [Anal.], 1929, 1, 39—40).—A method is described for measuring the sp. gr. of substances such as asphalt, which soften at about 50—80°. A test piece, obtained from a cylindrical brass mould of standard dimensions, is washed and dried before weighing, and then immersed in water in a special apparatus where the volume of water displaced can be read accurately and rapidly by adjustment of level in a movable burette. The measurements are all carried out at 25°. R. H. GRIFFITH.

Paraffin content of road asphalts. H. BURSTYN (Petroleum, 1929, 25, 257–260).—Asphalts which contain paraffins are not generally suitable for road construction except when these substances are not crystalline. In Polish and Russian petroleum pitches the content of paraffin is generally high, and two preparations from Galicia, known as Galbit and Molfalt, are found to contain 0.46 and 0.22%, respectively, of paraffin when analysed by Marcusson's method; they are also of good quality practically. R. H. GRIFFITH.

Improved fractionation for cracking processes. D. B. KEYES (Chem. & Met. Eng., 1929, 36, 78).— Vapours from a cracking still, or from the top of a stripping column in which crude tar is "reduced" by means of a countercurrent of steam, are led into the bottom of an ordinary fractionating column, indirectly heating liquid accumulating at that point before entering the side of the column through a valve where the pressure is reduced to 50—100 lb. Cold feed is heated by passing through a coil acting as a reflux condenser at the top of the column, and is then introduced into the column near its middle, where it is stripped of its volatile products. Re-cycle stock passes out of the bottom, whilst gasoline vapours from the top of the column are condensed and pass to a gas separator in accordance with usual practice. H. S. GARLICK.

British: Chemical Abstracts-B

Process for obtaining lubricating oils and vaseline from petroleum residues. M. MORGEN-STERN (Motorenbetrieb, 1929, 2, 3-4).—The process consists in warming the petroleum residues by means of low-pressure steam, whereby practically quantitative separation into oil and asphalt is accomplished.

Knock ratings of pure hydrocarbons. A. W. NASH and D. A. HOWES (Nature, 1929, 123, 276-277). Since unsaturated hydrocarbons differ widely in their reactivities towards oxidising agents, it was anticipated that they would possess widely different anti-knock values; comparative experiments with cyclohexane, benzene, cyclohexene, toluene,  $\Delta^{\beta}$ -pentene, trimethylethylene, diamylene, diisobutylene, and "ethyl fluid" show that this is the case. cycloHexene has anti-knock properties equivalent to those of benzene, whilst the others, particularly diamylene and diisobutylene, are far more effective. The advantages of the two lastnamed compounds (cf. B.P. 253,131; B., 1927, 835) are considered. Of the olefines examined, those which are the more stable towards bromine, sulphuric acid, potassium permanganate, and potassium dichromate are the more effective in suppressing knocking.

A. A. ELDRIDGE.

W. S. NORRIS.

X-Ray quantitative analysis of lead tetraethyl gasoline. R. H. ABORN and R. H. BROWN (Ind. Eng. Chem. [Anal.], 1929, 1, 26—27).—The passage of X-rays through solutions of lead tetraethyl in gasoline is measured by means of an ionisation chamber and galvanometer circuit, a Coolidge tube working at 35 kv. and 20 milliamp. being a suitable source of radiation. A Bragg-type ionisation chamber is used, and the liquid under examination is contained in a brass vessel with aluminium windows. It is found that the deflection produced in the galvanometer is directly proportional to the concentration of lead tetraethyl, but in order to obtain accurate measurements careful control of current and of the type of radiation is necessary.

## R. H. GRIFFITH.

Production of power alcohol from waste vegetable materials. A. C. THAYSEN and L. D. GALLOWAY (Ann. Appl. Biol., 1928, 15, 392-407).—A discussion of the fermentation of grass, straw, husks, etc. by *Bacillus acetoethylicus*. The yield (gals./ton) of alcohol and acetone is : *Hyparrhenia glauca* 18, corncobs 22.5, esun grass 17, *Phormium tenax* 10, rice husks 14, rice straw 19, wheat straw 16, *Andropogon gayanus* 20, *A. tectorum* 22, sorghum stems 17, papyrus (air-dry) 16.5, sisal-hemp residue 4.8, sugar-beet residues 24.7. CHEMICAL ABSTRACTS.

Transformations of fatty acids during geological epochs. I. G. L. STADNIKOV and A. WEIZMANN. II. G. L. STADNIKOV and Z. VOZSCHINSKAJA (Brennstoff-Chem., 1929, 10, 61-63, 81-82).—See B., 1929, 158.

See also A., Mar., 271, Ignition temperature of combustible gaseous mixtures (PRETTRE and LAF-FITTE). 294, Pityrol (SHOYAMA; OGATA; HIDAKA).

Crude petroleum and seeds. MURPHY.-See XVI.

## PATENTS.

Method and apparatus for washing coke, panash, coal, etc. R. NORGATE (B.P. 306,170, 14.11.27).—

Water is injected as a spiral or helical swirl into a washing chamber from a stationary injector.

Coke oven. C. OTTO (U.S.P. 1,698,272, 8.1.29. Appl., 9.4.27. Ger., 3.4.26).—Each vertical heating flue of a coke oven is provided with a removable burner nozzle, which can be taken out through an opening in the top of the oven in alinement with the flue. The opening can be closed by a removable cover brick and a loosely fitting filler brick which is packed in with insulating material. Both bricks are provided with sight holes in alinement with the flue.

A. B. MANNING.

**Coke oven.** W. M. PERSON (U.S.P. 1,698,651, 8.1.29. Appl., 26.4.23).—A coke oven with horizontal flues and with a pair of regenerators directly below the coking chamber is provided with independent passages connecting the ends of the flues with one regenerator, the passages from the top of the regenerator to the level of the flues being substantially vertical. The other regenerator is similarly connected with the other ends of the flues. A. B. MANNING.

Intermittent carbonising plant. N. J. BOWATER, A. H. LYMN, and CHAMBER OVENS, LTD. (B.P. 304,859, 7.11.27).—The smoke and fume emitted when the chambers of an intermittent carbonising plant are charged are removed through a flue or pipe which is attached to the charging mechanism and is so arranged that its lower end is situated closely over an aperture in the chamber additional to those through which the chamber is charged, whilst its upper end is situated under one of a number of orifices in a manifold from which the smoke can be exhausted by natural draught or by means of a fan. The orifices in the manifold are provided with valves so that those not in use may be kept closed. A. B. MANNING.

Gasification [of coal]. P. D'H. DRESSLER, ASST. to AMER. DRESSLER TUNNEL KILNS, INC. (U.S.P. 1,698,493, 8.1.29. Appl., 10.4.20. Renewed 9.5.25).—Coal is distilled in a continuous tunnel kiln, heat being transmitted to the coal by circulating the gases through a space which is partially separated from the main passageway, and in which they are heated. A decarbonising liquid is mixed with the gases passing through the separated space. A. B. MANNING.

Coating or impregnation of granular, pulverulent, or powdered materials with liquid compositions, and manufacture of briquettes etc. Η. NIELSEN and B. LAING (B.P. 304,812, 26.10.27).-The granular or powdered material is allowed to fall through a chamber countercurrent to an atomised stream of pitch or other impregnating composition. The material is first passed through a dryer or preheater in which its temperature is raised, or lowered, if the initial temperature is too high, to that most suitable for effective coating. Steam and/or hot gases are admitted to the lower part of the chamber, the temperature of which is so regulated that any surplus of the coating material is revolatilised. The coated material is passed continuously to a mixing device and thence to the briquetting apparatus. A. B. MANNING.

Gas-producing and oil-recovery plant. Combustion chambers. F. L. DUFFIELD (B.P. 305,253 and 305,270, 1.11.27).—(A) Pulverised fuel is first heated with air at  $200-250^{\circ}$  in order to destroy its tendency to become viscid, is then carbonised, the volatile products being collected, and is finally gasified. The first two processes are carried out in an annular space surrounding a dome-shaped chamber in the lower part of which lie the combustion and gasification chambers. The coal passes down a perforated spiral trough in the annular space, which is divided into two compartments. In

processes are carried out in an annular space surrounding a dome-shaped chamber in the lower part of which lie the combustion and gasification chambers. The coal passes down a perforated spiral trough in the annular space, which is divided into two compartments. In the upper compartment heated air is blown through the coal as it descends, whilst in the lower the coal is carbonised in a current of hot producer gas. Part of the carbonised product is fed, together with air, as primary fuel to the combustion chamber; the remainder, with gas as the carrying medium, is supplied as secondary fuel to the gasification chamber. The hot gases produced pass from the top of the dome-shaped chamber through the tubes of a steam generator and thence downwards through a heat-exchanger in which the gas used for the carbonisation process, the air for combustion, and the air for the oxidation of the fuel are preheated. (B) A primary supply of powdered fuel and air is introduced into a combustion chamber with a velocity of 150-300 ft./sec., and secondary air is introduced, with the same velocity, at two different levels. The zone of combustion within the chamber is regulated by suitably apportioning the secondary air supply between the inlets at the two levels. A. B. MANNING.

Means for producing gas. G. L. REICHHELM, Assr. to GASIFIER Co. (U.S.P. 1,699,543, 22.1.29. Appl., 7.2.20).—Finely-divided carbonaceous fuel is supplied to a vertical, cylindrical gas producer into which air under pressure is blown tangentially. Mechanical means are provided for synchronously controlling the supplies of fuel and air. A. B. MANNING.

Separation by intense cooling of gaseous mixtures containing acetylene. GES. F. LINDE'S EIS-MASCHINEN A.-G. (B.P. 288,216, 4.4.28. Ger., 4.4.27).— Acetylene, the presence of which causes difficulties in the separation of gaseous mixtures by intense cooling, is removed quantitatively therefrom by passage of the gas over heated catalysts, *e.g.*, partially dehydrated hydroxides of aluminium and iron, the acetylene being thereby decomposed, hydrogenated, or polymerised. The apparatus comprises a reaction chamber containing the catalyst, and periodically reversible regenerators for bringing about heat interchange between the inflowing and outflowing gases. A. B. MANNING.

Separating from water tarry substances dissolved therein. P. FRANKE (B.P. 279,465, 19.10.27. Ger., 21.10.26).—Tarry substances, e.g., phenols and pyridine, are separated from water by treatment with a tar-oil fraction of boiling range  $240-320^{\circ}$  and  $d \cdot 04-1 \cdot 05$ , from which the substances are subsequently recovered by successive extractions with alkali and dilute acid, or by distillation under vacuum. In carrying out the process the tar oil and water may be caused to flow through one another in countercurrent, or the water may be passed over a layer of the oil below which is maintained a layer of alkali lye, the oil layer being kept in motion by means of a stirrer without, however, disturbing the relative positions of the layers. A portion of the oil is withdrawn from time to time, washed with dilute acid, and returned; the lye is similarly withdrawn and replaced by fresh lye as required.

A. B. MANNING.

Manufacture of valuable products from montan wax. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 305,552, 5.9.27).—Yellow or white products. similar to carnauba wax but easier to hydrolyse than either carnauba or crude montan wax, are prepared from the latter (1 pt.) by the action of chromic acid and at least 5 pts. of a mineral acid (sulphuric or phosphoric) in 30—70% aqueous solution, at 100—130°. Bleaching and hydrolysis of the wax esters are accomplished simultaneously, the duration of the process and the quantity of chromic acid being so chosen that carbonisation is avoided. W. S. NORRIS.

Manufacture of liquid and other hydrocarbons and derivatives thereof by the destructive hydrogenation of carbonaceous materials. I. G. FARB-ENIND. A.-G. (B.P. 293,719, 8.8.27. Ger., 7.8.26. Cf. B.P. 275,663; B., 1929, 45).—The process is carried out by treatment with a large excess of hydrogen or gases containing hydrogen in a circulatory system under a pressure of at least 50 atm. and in the presence of a catalyst consisting of cobalt or a compound thereof with up to 10% of metals of the 6th group of the periodic system or compounds thereof, e.g., cobalt with up to 10% of chromium oxide. Iron or other materials giving rise to the formation of methane and separation of carbon should be excluded from contact with the hot reacting H. S. GARLICK. materials.

Manufacture of low-boiling oils and cyanides. J. C. CLANCY (B.P. 304,421, 21.11.27).-High-boiling hydrocarbon oils are brought into intimate contact with a bath of molten alkali cyanide, and distilled under cracking temperatures and pressures. Desulphurisation occurs with formation of thiocyanate. Any moisture reacts with the molten cyanide to form ammonia which in part decomposes and tends to hydrogenate the unsaturated hydrocarbons formed during the process. The evolved hydrocarbon vapours and gases are suitably collected. A portion of the cyanide destroyed during the reaction is regenerated by leading a current of ammonia into contact with the molten reagent during the distilling operation. The fusion residues are discharged from the still while still molten and treated to reconstitute the cyanide by adding sodium carbonate and subjecting the mixture to the action of a nitrogenous gas. The distillation process may be modified by running a molten mixture of commercial sodium cyanide and thiocyanate into the top of a column still filled with catalytic packing units comprising solid masses of iron, nickel, or copper, while introducing the preheated oil or vapours thereof at an intermediate part of the still. H. S. GARLICK.

Treatment of hydrocarbons with liquid sulphur dioxide. ALLGEM. GES. F. CHEM. IND. M.B.H. (B.P. 281,337, 28.11.27. Ger., 26.11.26).—In the treatment of hydrocarbons with liquid sulphur dioxide the latter is recovered from its mixtures with hydrocarbons by evaporation in stages, the low pressures necessary for complete evaporation in intermediate and final British Chemical Abstracts-B. 274

on to the surface of gently flowing water, and, after stratification, the aqueous naphthenate solution and the oil layer are withdrawn separately, the oil layer being allowed to settle again in a second tank. L. A. COLES.

Mineral oil derivative and its manufacture. G. ALLEMAN, ASST. to SUN OIL CO. (U.S.P. 1,694,463, 11.12.28. Appl., 6.2.24).—Alkali precipitate (soda sludge) obtained in the refining of lubricating oil is treated with water to give, e.g., a total water content of 58%, and the mixture is heated to a steam pressure of at least 4 atm. and cooled under pressure. The mineral oil is separated and the soap and resin solutions are concentrated, the soap and resin being then filtered off from the sodium sulphate or other salts.

R. BRIGHTMAN.

Apparatus for manufacture of carbon. A. LEDERER (U.S.P. 1,700,942, 5.2.29. Appl., 14.8.25. Austr., 24.4.25).—See B.P. 251,244; B., 1927, 244.

Coal washing apparatus. P. Wolf (B.P. 306,169, 14.11.27. Addn. to B.P. 206,151; B., 1925, 162).

Preparing charges containing [atomised] liquid fuel for use in internal-combustion engines. MARION STEAM SHOVEL CO., Assees. of S. P. and H. A. COWARDIN (B.P. 282,342, 2.8.27. U.S., 15.12.26).

Scavenging the residues of combustion deposited on the sliding surfaces of internal-combustion engines operated by pulverulent fuel. I. G. FARBENIND. A.-G. (B.P. 274,905, 25.7.27. Ger., 26.7.26).

Gas burners. C. H. KEMPTON and W. BISSELL (B.P. 305,786, 7.1.28).

Vacuum distillation (B.P. 303,078—9).—See I. Carbon dioxide in flue gases (U.S.P. 1,701,181).— See XI.

## III.—ORGANIC INTERMEDIATES.

Analysis of acetic anhydride in the presence of strong acids by thermometric titration. T. SOMIYA (Proc. Imp. Acad. Tokyo, 1929, 5, 34-37; cf. B., 1927, 439).-The effect of strong acids in retarding the velocity of acetylation of aniline can be eliminated by addition of pyridine, and a sharp end-point obtained; under these conditions the acetylation velocities of aniline hydrochloride and aniline sulphate in acetylene tetrachloride are also sufficiently great to afford fairly satisfactory end-points. The amount of pyridine to be added is very important, and should be less than that of acetic acid in the solution to be titrated. Standardisation of solutions, and analysis of samples of aniline, aniline hydrochloride, and aniline sulphate are described. in detail. C. W. SHOPPEE.

Manufacture of  $\beta$ -naphthol. A. I. ZAKHAROV (J. Chem. Ind. Moscow, 1928, 5, 720—721).—In presence of 1—2% of sodium carbonate the thermal bends of the fusion curve of sodium naphthalene-2-sulphonate and alkali are flattened. The fused mass does not thicken, and overheating and charring are absent. When the last portions of the sulphonate are added, the acid or even the neutral sulphonate tends to decompose on account of the decrease of total alkalinity; the presence of sodium carbonate preserves the salt from thermal

stages being obtained by a jet apparatus operated by the sulphur dioxide vapour from the first-stage evaporators, which are heated to give the required increase of pressure. The reduction in pressure obtained by means of the jet is also employed to remove sulphur dioxide vapour from the cooler, the liquid sulphur dioxide in which is thus brought down to the temperature required for treatment of the oil. W. S. NORRIS.

Transformation of gaseous hydrocarbons. P. H. HULL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 304,914, 21.1.28).—The hydrocarbons are passed through a low-tension arc maintained between poles of tungsten, tungsten carbide, molybdenum, osmium, or tantalum. The electrodes preferably diverge in the direction of the stream of gas, and may consist of rods fastened by clips to a water-cooled tube. The process may be utilised, for example, in the production of acetylene from methane. A. B. MANNING.

Manufacture of lubricating oils. L. MELLERSH-JACKSON. From SUN OIL Co. (B.P. 305,846, 7.3.28).— The light fractions of crude mineral oil are distilled off under limited temperature and pressure in the absence of oxygen, the residue is treated with aqueous soda, the water and certain oil fractions are removed under reduced pressure, and the residue, distributed over an extended surface, is brought into heat exchange with mercury vapour, while limiting the temperature of the mercury vapour and the pressure on the oil to minimise cracking. The condensed lubricating oil distillate is then freed from malodorous volatile constituents by being distributed over an extended surface while being subjected to a high vacuum. W. S. NORRIS.

Manufacture of lubricating and insulating oils. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 305,553, 5.10.27).—Substances such as coal, tars, mineral oil, etc., if solid are extracted by means of a solvent or subjected to destructive hydrogenation, and if liquid are destructively hydrogenated or cracked, yielding liquid products; the latter, specifically those of the middle-oil type, are exposed in the coherent liquid phase to the action of high-tension electric currents, preferably of high frequency, yielding lubricating or insulating oils. Thus brown coal is hydrogenated at 450° and 200 atm., and, of the liquid products, a fraction of b.p. substantially up to 300° (viscosity  $2 \cdot 7^{\circ}$  [Engler] at 50°) is subjected to a current of about 10,000 volts and 500 periods. After separating the viscous products by distilling off the more volatile constituents, the latter may be treated again.

W. S. NORRIS.

Purification of petrol. W. M. KNOWLING and M. KOSTEVITCH (B.P. 287,141, 14.3.28. Fr., 16.3.27).— Tar, water, sulphur compounds, etc. are removed by treatment with a mixture of activated charcoal and activated silica gel, the latter being either of the widepore or the capillary-pore species, or a mixture of the two. A. B. MANNING.

Recovery of oils and naphthenic acids from still residues obtained in the purification of mineral oils. C. ARNOLD. From HUMBLE OIL & REFINING Co. (B.P. 304,926, 6.2.28).—The hot residues are discharged decomposition before it has been attacked by the sodium hydroxide. The fused mass contains 93-96% of  $\beta$ -naphthol. Excess of carbonate causes frothing. CHEMICAL ABSTRACTS.

See also A., Mar., 265, Equilibrium between methyl alcohol, carbon monoxide, and hydrogen (Smith and BRANTING). 273, Chlorine carrier (FIERZ-DAVID). 289, Oxidation of methane with nitrogen peroxide (FROLICH and others). Intermediate products of combustion of methane (RIESENFELD and GURIAN). 307, Pyrogenic dissociation of condensed ring systems (ORLOV). Fluoranthene (VON BRAUN and ANTON). 308, Preparation of perylene (MARSCHALK). 310, Halogen derivatives of  $\beta$ -naphthol unsubstituted in position 1 (MARSCHALK). 316, Use of zirconium tetrachloride in organic synthesis (KRISHNA-MURTI). 318, Derivatives of 2-methylanthraquinone and anthraflavene (Ruggli and MERZ). 319, Naturally occurring anthraquinone derivatives (MITTER and BISWAS). Reduction products of hydroxyanthraquinones (HARDACRE and PERKIN). 329, Derivatives of quinoneacridone (SCHARVIN and GALPERIN).

Naphthalene in gas. FULWEILER and others; POWELL; CUNDALL. Power alcohol. THAYSEN and GALLOWAY.—See II. Polyhydric alcohol-polybasic acid reaction. KIENLE and HOVEY.—See XIII. Rectification of alcohol. HOUGHLAND.—See XVIII. Liquor cresoli saponatus. FEIST.—See XX.

## PATENTS.

Catalytic oxidation of organic compounds. SEL-DEN Co., Assees. of A. O. JAEGER (B.P. 291,419, 11.11.27. U.S., 3.6.27).-Oxidation catalysts give improved and more easily controlled results when mixed with "stabilisers," viz., sulphates, phosphates, halides, chlorates, nitrates, cyanides, arsenates, antimonates, bismuthates, borates, carbonates, or other innocuous salts of alkali metals, alkaline-earth metals, or other metals which form oxides not reducible by hydrogen. To these may be added "stabiliser promoters" which enhance their effect; there are usually mild catalysts, and include zeolites and other base-exchanging diluents, particu-larly those which are highly porous. Examples are : (1) ammoniacal silver vanadate on kieselguhr heated to expel water and ammonia, impregnated with aqueous potassium (or potassium hydrogen) sulphate, and dried at 80-100° to give a catalyst for formaldehyde from methyl alcohol, maleic acid from benzene, etc.; (2) potassium vanadomolybdate, ferric pyrovanadate, and potassium hydroxide on kieselguhr, dried at 400° and treated with burner gases at 400-500° to convert the alkali into sulphate, forming a catalyst for anthraquinone from anthracene etc.; (3) kieselguhr and silica gel, impregnated with potassium molybdate, sodium tungstate, lithium tantalate, and potassium silicate, calcined and sulphated with burner gas, to give a catalyst for chlorobenzaldehyde from chlorotoluene. Seventeen other examples are given. C. HOLLINS.

Oxidation of volatile organic compounds [acetaldehyde to acetic acid]. IMPERIAL CHEM. INDUS-TRIES, LTD., S. W. ROWELL, and H. S. HIRST (B.P. 304,350, 18.10.27, 2.1. and 12.7.28).—A continuous process for the air-oxidation of acetaldehyde in presence of a catalyst (e.g., manganese acetate and a trace of cobalt acetate) is described in which acetic acid containing the dissolved catalyst flows down a tower, meeting a countercurrent of air and acetaldehyde vapour. Acetic acid is withdrawn at the base of the tower and separated by freezing or distillation into pure acid (equal in amount to new acid produced) and catalyst acid, the latter being returned to the tower. The temperature is regulated by withdrawing liquor from a plate near the middle of the tower, cooling it, and returning it a little higher up the tower; the amount so circulated per hour may be, say, ten times the amount of acid fed per hour to the tower. C. HOLLINS.

Manufacture of aliphatic [acetic] anhydrides. BRIT. CELANESE, LTD., H. DREYFUS, and C. I. HANEY (B.P. 304,156, 15.7.27).—Charring is avoided by mixing the acid vapour with an inert gas (nitrogen, hydrogen, or steam) preheated to a temperature above that of anhydride formation; *e.g.*, acetic acid vapour at 200— 300° is mixed with steam at 800—1000°, the mixture being kept at 500—800°. C. HOLLINS.

Manufacture of aliphatic [acetic] anhydrides. H. DREYFUS (B.P. 305,147, 30.7.27).—The (acetic) acid vapour is passed with hydrogen at 500—700° through a copper tube which has previously been heated to the required temperature in hydrogen. Nickel, cobalt, iron, or alloys of these or other metals, which in the form of acetates are reduced to the metallic state by hydrogen at the temperature of reaction, may be used in place of copper. C. HOLLINS.

Manufacture of esters. C. ARNOLD. From STAN-DARD OIL DEVELOPMENT CO. (B.P. 305,308, 21.11.27).--Esterification is performed in presence of a solvent in which the solubility of the ester is greater than that of its components or water; *e.g.*, acetates of *sec.*-butyl alcohol and *sec.*-amyl alcohol are made in presence of a petroleum fraction of initial b.p. 300° and d 1.247. C. HOLLINS.

Manufacture of isopropyl alcohol. G. O. CURME, JUN., and E. W. REID, ASSTS. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,695,249, 11.12.28, Appl., 19.7.19). Propylene is absorbed in sulphuric acid or phosphoric acid at -10° to 0°, the temperature rising to 15-20° at the finish, and the product, containing, e.g., 80% of isopropyl sulphate, is hydrolysed by emulsification with water below 40°. R. BRIGHTMAN.

Manufacture of hydrocarbon derivatives and of unsaturated hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 305,603, 6.10.27).—The gases (up to pentane) from hydrogenation of coal are passed with steam or hydrogen sulphide over suitable catalysts at 700—750° to give olefines (propylene and butylene), acetic and other acids, alcohols, aldehydes, and in the case of hydrogen sulphide thiophen and other sulphur compounds. Catalysts used are manganese dioxide, alumina, molybdena, chromium oxide, and metal sulphides. C. HOLLINS.

Manufacture of organic bases. I. G. FARBENIND. A.-G. (B.P. 283,163, 5.1.28. Ger., 5.1.27).—For the production of pyridine bases from acetylene and ammonia without formation of acetonitrile, a catalyst comprising an inorganic salt (zinc chloride, ferric chloride, cadmium chloride) capable of combining with ammonia to give complex salts is used. A typical product, with zinc chloride at  $280-290^{\circ}$ , gives fractions b.p.  $60-75^{\circ}$ (29%),  $75-110^{\circ}$  (6%),  $110-125^{\circ}$  (26%),  $125-160^{\circ}$ (24%),  $200^{\circ}$  (15%). C. HOLLINS.

Manufacture of hexahydroaniline [cyclohexylamine]. A. CARPMAEL. From I. G. FARBENIND, A.-G. (B.P. 305,507, 5.11.27).—An 88% yield of cyclohexylamine is obtained in 30 min. by heating aniline at 250—  $300^{\circ}$  in hydrogen at 100 atm. in presence of 2% of cobalt oxide, prepared, e.g., by heating cobalt oxalate at 200— $400^{\circ}$ . C. HOLLINS.

Manufacture of aromatic [hydr]oxyaldehydes. I. G. FARBENIND. A. G. (B.P. 294,889, 30.7.28. Ger., 30.7.27).—Hydroxylated benzylamines are boiled with an isatin (especially a salt of isatin-5-sulphonic acid) in aqueous alkaline solution (cf. Traube, A., 1911, i, 960). The preparation of salicylaldehyde from o-hydroxybenzylamine, vanillin from vanillylamine, and 5-aldehydo-o-cresotic acid from 5- $\omega$ -aminomethyl-o-cresotic acid is described. C. HOLLINS.

Manufacture of substituted thioglycollic acids. I. G. FARBENIND. A.-G. (B.P. 287,858, 26.3.28. Ger., 24.3.27. Addn. to B.P. 281,290; B., 1928, 921).— The process of the prior patent is applied to the production of thioglycollic acids from halogenated benzenesulphonic acids, *e.g.*, 2-chloro-*p*-xylene-5-sulphonic acid, 3-chlorotoluene-6-sulphonic acid, 1:2:3-trichlorobenzene-4- or -5-sulphonic acid; in each case the sulphonic group is converted into a sulphonyl chloride group, which is reduced to thiol and condensed with chloroacetic acid. C. HOLLINS.

Manufacture of halogenated organic compounds. COMP. NAT. MAT. COL. & MANUF. DE PROD. CHIM. DU NORD RÉUNIES, ETABL. KUHLMANN (B.P. 294,462, 11.11.27. Fr., 23.7.27).—Aromatic amines are halogenated in the form of their arylsulphonyl derivatives. o-Toluenesulphon-o-toluidide, m.p. 134°, chlorinated with chlorate and hydrochloric acid, gives a product, m.p. 154°, from which on hydrolysis 5-chloro-o-toluidine is obtained. 2-Nitrotoluene-4-sulphon-p-toluidide, m.p. 131°, with chlorine in tetrachloroethane at 50°, gives the 3-chloro-ptoluidide, m.p. 152°; toluene-p-sulphonanilide yields the 2:4-dichloroanilide; the  $\beta$ -naphthylamide similarly affords a toluene-p-sulphon-1-chloro- $\beta$ -naphthylamide, m.p. 105°; from these the bases are obtained by hydrolysis. C. HOLLINS.

Manufacture of 2:3-aminonaphthol and derivatives thereof. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 304,439, 15.12.27).—An O-arylsulphonate of 2:3-hydroxynaphthamide is treated in alcohol with alkaline hypochlorite or hypobromite and then hydrolysed with hot caustic alkali. 2:3-*Hydroxynaphth*amide O-p-toluenesulphonate, m.p. 216°, obtained from the amide and toluenesulphonyl chloride, gives 2:3aminonaphthol O-p-toluenesulphonate, m.p. 144—145°, which may be hydrolysed to 2:3-aminonaphthol; the benzenesulphonate, m.p. 170°, behaves similarly. The O-p-toluenesulphonate, m.p. 184—185°, of 6-bromo2:3-hydroxynaphthamide (m.p. 295°) yields the O-p-toluenesulphonate, m.p. 105°, of 6-bromo-2:3-aminonaphthol (m.p. 222°). C. HOLLINS.

Manufacture of acenaphthene derivatives. G. T. MORGAN and H. A. HARRISON (B.P. 305,754, 8.12.27).— On nitration of acenaphthene with nitric acid in acetic anhydride at  $-5^{\circ}$  to  $+16^{\circ}$  3-nitroacenaphthene, m.p.  $151^{\circ}$ , crystallises on cooling; the 5-nitro-compound may be obtained by diluting the mother-liquor. Reduction gives 3-aminoacenaphthene, m.p.  $81^{\circ}$  (cf. 1-amino-, m.p.  $136^{\circ}$ , 4-amino-, m.p.  $87^{\circ}$ , 5-amino-, m.p.  $108^{\circ}$ ). The 3- and 5-isomerides may be separated by means of petroleum, b.p.  $80-100^{\circ}$ , from the reduced product of ordinary nitration. C. HOLLINS.

Manufacture of [hydr]oxydiaryl ketones. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 305,763, 16.12.27).—A hydroxyaromatic acid chloride is condensed with an aromatic hydrocarbon etc. in presence of aluminium chloride. 2:3-Hydroxynapthoyl chloride with benzene gives phenyl 3-hydroxy- $\beta$ -naphthyl ketone, m.p. 161—162°; with toluene, p-tolyl 3-hydroxy- $\beta$ -naphthyl ketone, m.p. 152—153°; with anisole, p-anisyl 3-hydroxy- $\beta$ -naphthyl ketone, m.p. 152—153°; with anisole there is obtained 2-hydroxy-4'-methoxy-3:5-dimethylbenzophenone [p-anisyl 4-hydroxy-m-5-xylyl ketone], m.p. 105—106°. C. HOLLINS.

Manufacture of naphthol ether carboxyamides alkoxynaphthamides] and aminonaphthol ethers. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 304,441, 19.12.27).—Hydroxynaphthamides yield on alkylation O-ethers, which by Hofmann's reaction are converted into aminonaphthol ethers. 2:3-Methoxynaphthamide m.p. 170°, and 2:3-aminonaphthol methyl ether, m.p. 107°; 2-carboxymethyl-3-naphthamide and 3-amino- $\beta$ -naphthylglycollic acid; 1-ethoxy-2-naphthamide, m.p. 154°, and 2-amino-1-naphthol ethyl ether, m.p. 48—49°; 2:6-hydroxynaphthamide, m.p. 209° (benzyl ether, m.p. 198°; methyl ether, m.p. 224°), and the benzyl (m.p. 176°) and methyl (m.p. 156—157°) ethers of 2:6-aminonaphthol are described. C. HOLLINS.

Manufacture of salts of acid sulphuric esters of nitro-9:10-dihydroxyanthracenes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 304,436, 9.12.27).-A nitroanthraquinone is treated with finely-divided copper or zinc in dry pyridine (or other tertiary base) in presence of chlorosulphonic acid or esters or of sulphur trioxide. The nitro-group is not reduced, the product being the water-soluble pyridine salt of the disulphuric ester of the nitroleucoanthraquinone. Products from 1- and 2nitroanthraquinones, 1-nitro-2-methylanthraquinone, and 1:5-dinitroanthraquinone are described. Treatment with acid oxidants (nitric or nitrous acid, chromic acid) regenerates the starting-material; with nonoxidising acids mixtures of nitro- and amino-anthraquinones are formed. C. HOLLINS.

Manufacture of new solid diazo-azo compounds. O. Y. IMRAY, From I. G. FARBENIND, A.-G. (B.P. 305,498, 28.10.27).—A *p*-aminoazo compound of the type, arylamine  $\rightarrow o$ -aminophenol ether, containing no sulphonic or carboxylic group, is diazotised and treated with an alkali halide, or with sulphuric acid and/or a sulphate, or with a heavy-metal salt with or without sodium chloride, or with an arylsulphonate. The diazo compound separates or is isolated by evaporation. Anhydrous or partly anhydrous salts may be added to the solid diazo compound. Twenty examples are given including: *p*-nitroaniline  $\rightarrow$  cresidine, 2:5-dimethoxyaniline, 4-ethoxy-*m*-toluidine, *o*-aminophenyl benzyl ether; *o*-chloroaniline  $\rightarrow 2:5$ -dimethoxyaniline, 2-ethoxy-*a*-naphthylamine, cresidine, *o*-anisidine.

C. HOLLINS.

Production of 1-diazoanthraquinone-2-carboxylic acids. L. EIFFLAENDER, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,700,790, 5.2.29. Appl., 23.11.26. Ger., 28.11.25).—See B.P. 262,119; B., 1928, 45.

Naphthenic acids from mineral oils (B.P. 304,926).—See II.

# IV.—DYESTUFFS.

# PATENTS.

Manufacture of vat dyes. I. G. FARBENIND. A.-G. (B.P. 279,489, 21.10.27. Ger., 23.10.26).—4:4'-Dimethylthioindigo, obtained by the usual methods from 3-chloro- or 3-nitro-o-toluidine by way of 3-chloroor 3-nitro-o-tolunitrile, shows excellent fastness to light. C. HOLLINS.

Manufacture of vat dyes. I. G. FARBENIND. A.-G. (B.P. 280,866, 28.10.27. Ger., 18.11.26).—1-(3-Benzanthronylamino)anthraquinones carrying an N-alkyl group are converted into vat dyes by alkaline condensing agents; e.g., 1-(3-benzanthronylmethylamino)anthraquinone with alcoholic potassium hydroxide at 98° yields a blue-green vat dye. C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 305,082, 26.10.27).—1:1'- or 2:1'-Dianthraquinonylamines carrying at least one acridone ring (NH in 4- or 4'-position) and also an aroylamino-group are converted into vat dyes (carbazole derivatives ?) by treatment with sulphuric acid alone at  $30-40^{\circ}$  or with sulphuric acid at  $15-20^{\circ}$  followed by oxidation, e.g., with nitrous acid. The dianthraquinonylamine from 4-aminoanthraquinone-1:2-acridone and 5-chloro-1benzamidoanthraquinone affords in this way an olive dye; the isomeride from 4-chloro-1-benzamidoanthraquinone gives a greyish-green dye. C. HOLLINS.

Preparation of yellow monoazo dyes fast to light. H. WAGNER and B. VOSSEN, ASSTS. to GRASSELLI DYE-STUFF CORP. (U.S.P. 1,701,248, 5.2.29. Appl., 13.8.27. Ger., 19.8.26).—See B.P. 276,353; B., 1928, 183.

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

Chemical composition of wool, with special reference to the protein of wool fibre (keratin). H. R. MARSTON (Council for Sci. & Ind. Res., Australia, Bull. No. 38, 1928, 36 pp.).—Analyses of wools from various localities in Australia indicate that after careful purification the composition of the wool keratin from these sources is constant within the limits of experimental error. The cystine content of the fibre (determined by the Folin-Looney method after hydrolysis

with 20% sulphuric acid) was found to be 13.1%, whilst the tyrosine and tryptophan contents were 4.8 and 1.8%, respectively. For the determination of humin, amide, basic, and non-basic nitrogen a modification of Van Slyke's method was used. The preliminary acid hydrolysis introduces very serious errors unless rigidly controlled, since the l-cystine formed undergoes progressive racemisation, resulting, on treating the hydrolysate with phosphotungstic acid, in the formation of a racemised cystine phosphotungstate which is very soluble in comparison with the *l*-cystine compound. Further, in the determination of amide nitrogen by distilling the hydrolysate with lime in vacuo approximately one half of the cystine initially present is decomposed. The arginine content of wool keratin (corrected, for ammonia derived from cystine) was found to be 10.2%, and the histidine and lysine contents (corrected for the abnormal yield of amino-nitrogen by cystine) to be 6.9 and 2.8%, respectively. Amide nitrogen amounted to 1.2%. The isoelectric point of wool keratin was found to be  $p_{\rm H}$  3.4. In the preparation of the wool for analysis, the samples were dehydrated in absolute alcohol, extracted for 48 hrs. with absolute ether, washed in 95% alcohol, then in 15 changes of tap water, and dried at 40° for 24 hrs. The wool was then combed, equilibrated in 0.01N-hydrochloric acid for 12 hrs., repeatedly washed in distilled water, and dried at 100°. Complete desiccation was then obtained by three successive treatments with absolute alcohol followed by drying for 96 hrs. at 100°. D. J. NORMAN.

Rigidity of wool and its change with adsorption of water vapour. J. B. SPEAKMAN (Trans. Faraday Soc., 1929, 25, 92—103).—The rigidity of dry Cotswold wool fibres has been studied as a function of the fibre diameter. The influence of adsorbed water on the rigidity of wool fibres has been determined under adsorption and desorption conditions, and expressions have been found relating the rigidity to the amount of water adsorbed and to the relative humidity. The reduction in relative rigidity with changing humidity can be expressed by a formula of the adsorption isotherm type and is probably caused by adsorption of water on particular groups in the wool molecule, but the simplicity of the rule is masked by the fact that water can be adsorbed in two other ways in addition.

## O. J. WALKER.

Value of the ignition method for determining the cotton content of asbestos goods. H. Sommer (Gummi-Ztg., 1929, 43, 1107-1108) .- On account of the variation in the loss in weight on ignition suffered by various grades of asbestos or even by one grade at different temperatures, it is not possible to obtain trustworthy indications as to the proportion of any cotton present by an ignition method. Similarly, the hygroscopic moisture and the loss on treatment with 80% sulphuric acid fail to give accurate indications. The only satisfactory method is by means of cuprammonium solution. A shredded sample (0.5-1 g.) is weighed after exposure for several hours in air of 65% R.H., the moisture content of the material being determined in a separate sample by drying for 2-3 hrs. at 110°; any fat or starch should be removed previously. The sample is treated in a closed flask overnight, with

frequent shaking, with 50 c.c. of fresh cuprammonium solution (approx. 20 g. Cu per litre). After filtration through a Gooch crucible the residual asbestos is washed successively with cuprammonium solution and water and then dried at 110° until constant. The proportion of cotton is derived by difference. D. F. Twiss.

Cellulose ethers [alkylated celluloses]. E. BERL and H. SCHUPP (Cellulosechem., 1929, 10, 41-59).-A detailed account of the methylation and ethylation of various celluloses is given. Methylation of cellulose (linters) with methyl sulphate and 15% sodium hydroxide solution at the ordinary temperature (cf. Denham and Woodhouse, B., 1913, 974; 1914, 1084; 1917, 607, and Heuber and von Neuenstein, A., 1923, i, 17) affords first of all a monomethylcellulose, partly soluble in water, completely so in Schweizer's reagent, and insoluble in the usual organic solvents. Further methylation of this produces methylated celluloses with gradually increasing methoxyl content, until trimethylcellulose results (25-28 methylations). This derivative is insoluble in water and Schweizer's reagent. The solubility in water of the methylated celluloses diminishes gradually after the second methylation, finally becoming insoluble. In two of the cases examined the methoxyl content of the water-soluble portion and of the original material is the same. The solubility of the methylated products in Schweizer's reagent diminishes appreciably with increasing methoxyl content; two hydroxyl groups must be present in the cellulose molecule to give a completely soluble product. Methylation of alkalisoluble cellulose (by previous treatment with 75% sulphuric acid) with methyl sulphate under a variety of conditions gives derivatives containing 19.17-30.71% of methoxyl, according to the amount of methyl sulphate used. Methylation proceeds at the ordinary temperature, but analogous ethylation requires an initial temperature of 50° for reaction to commence. Monoethylcellulose is soluble in water and insoluble in alcohol. whilst diethylcellulose is soluble in alcohol and insoluble in water. The relative viscosity of aqueous solutions of the former increases considerably with increase of concentration, whilst an alcoholic solution of the latter also gives an increase, not nearly so marked as with the former. Ethylation of cellulose with ethyl chloride and sodium hydroxide (cf. G.P. 322,586 of 1912) gives products having varying compositions and properties, according to temperature of reaction, time of heating, presence of water, and amount of ethyl chloride used. Thus, at 100-120°, under otherwise identical conditions, the ethylated derivative is almost completely soluble in alcohol, whilst at 60-80° the product is only partially so. Further ethylation of alcohol-soluble and -insoluble products increases the ethoxyl content, does not affect the solubility in alcohol, and renders them soluble in chloroform. The viscosities of chloroform solutions of these substances vary considerably. Ethylation with 16 mols. of ethyl chloride at 115° gives a product (2.5 ethoxyl groups per mol.) partly soluble in chloroform, dichloroethylene, and benzene, insoluble in all other solvents. Further ethylation at 100° affords triethylcellulose, also obtained by direct ethylation of cellulose at 115° with 48 mols. of ethyl chloride. This derivative is soluble in chloroform, dichloroethylene,

benzene, and acetic acid. Further ethylation of a water-soluble ethylcellulose (ethoxyl content  $18 \cdot 2\%$ ) with ethyl chloride and sodium hydroxide at 80° increases the ethoxyl content to  $38 \cdot 85\%$ ; the new derivative is then insoluble in water, but soluble in alcohol. Dimethylcellulose can be ethylated with ethyl chloride and sodium hydroxide at 90—100°. It is not possible to ethylate cellulose with ethyl chloride in presence of pyridine or magnesium hydroxide. Hot sodium hydroxide solution does not hydrolyse dimethyl- or diethyl-cellulose.

H. BURTON.

Standard method for determining the viscosity of cellulose in cuprammonium hydroxide. Com-MITTEE OF DIVISION OF CELLULOSE CHEMISTRY OF THE AMERICAN CHEMICAL SOCIETY (Ind. Eng. Chem. [Anal.], 1929, 1, 49-51).-Details of the required apparatus, of the experimental procedure, and of the preparation and standardisation of the cuprammonium solution for use in determining the viscosity of 2.5% solutions of cellulose are given. For ordinary solutions the falling-sphere method is used, the time being taken for glass beads of in. diam. to fall a vertical distance of 15 cm. through the solution maintained at 25°. When the viscosity is very low a special pipette is used of such dimensions that the shearing force applied in it is approximately the same as that due to the falling glass bead, thus making it possible to obtain the same apparent viscosity on a single solution by both methods. This pipette is calibrated by timing the outflow of an oil of known viscosity. The procedure gives satisfactory results with a wide variety of celluloses, and if satisfactory measurements cannot be made with cellulose solutions of the above concentration other concentrations may be used and the viscosity corresponding with a 2.5% solution calculated by the application of a suitable formula such as Joyner's. B. P. RIDGE.

Determination of a-cellulose. SUB-COMMITTEE 2 OF DIVISION OF CELLULOSE CHEMISTRY OF THE AMERICAN CHEMICAL SOCIETY (Ind. Eng. Chem. [Anal.], 1929, 1, 52—54).—The  $\alpha$ -cellulose content of two grades of cotton linters and three grades of sulphite pulp has been determined by four variations of the same method and by workers in eight different laboratories. The following results of the study of different factors affecting the determinations are given. (1) Dilution of the sodium hydroxide-cellulose solution with an equal volume of water and stirring immediately before filtering does not precipitate  $\beta$ -cellulose and facilitates filtration. (2) The yield of  $\alpha$ -cellulose is not affected by washing with water at temperatures above 20°. (3) Cellulose is more soluble in sodium hydroxide solution alone than in a similar solution containing dissolved cellulose. (4) Mercerisation in air and in nitrogen give the same yields of  $\alpha$ -cellulose. In the method which is recommended to the Cellulose Division of the American Chemical Society as a tentative standard method the cellulose is mercerised with 17.5% sodium hydroxide for 45 min., the alkali being added in several portions, an equal volume of water is added, the  $\alpha$ -cellulose filtered off through a Gooch crucible, washed with water, 10% acetic acid, and water, and dried to constant weight at 105°. Close agreement is shown between the results from the different laboratories. B. P. RIDGE.

Determination of a- and β-cellulose. R. BERGQVIST (Papier-Fabr., 1929, 27, 119-123).-The influence of various factors in the determination of a- and B-cellulose, such as the conditions of filtering, the ratio of sodium hydroxide solution to cellulose material, the amount of water used for dilution after the mercerisation, etc., has been investigated, and the method of carrying out each stage of the determination is discussed. The following points are emphasised : (1) The temperature of the experiments should be controlled by use of a thermostat at 20°, or a correction should be applied depending on the temperature employed. The wash-water used should also be at 20°. (2) After addition of the diluting water, filtration and washing must be done as quickly as possible ; a suitable filtering medium is a piece of coarse linen fabric placed in a Buchner funnel. (3) For dilution of the sodium hydroxide solution after mercerisation of the cellulose the same quantity of water (300 c.c.) should always be used. (4) For the kneading operation 5 min. should suffice, and, if care be taken in working, reproducible results are easily obtained within the limits for  $\alpha$ -cellulose of  $\pm 0.1\%$ . B. P. RIDGE.

Determination of cellulose and amount of chlorine consumed in its isolation. M. W. BRAY (Ind. Eng. Chem. [Anal.], 1929, 1, 40-43).-The solvent-extracted sawdust or wood pulp is chlorinated in a Jena glass crucible with a fritted glass bottom by passing chlorine under slight pressure from a gas burette up through the crucible and over into a Hempel gas pipette. The volumes of gas passed over and that remaining in the apparatus are measured, the contents of the crucible are washed with water (titration of the washings giving the amount of hydrochloric acid formed), treated with sodium sulphite solution, and again washed. These treatments are repeated until the lignin is completely removed and the fibres are uniformly white, when the weight of dry material remaining is determined. The time taken for the first chlorination is 3-4 min., and for each subsequent chlorination 1-2 min., but the removal of lignin and other foreign substances is as complete as when, by the older processes,  $\frac{1}{2}$ -1 hr. is taken. The method has the advantages that the chlorination is more uniform and complete than when it is carried out by suction or in beakers, the time is shortened without impairing efficiency, the total chlorine used together with that forming hydrochloric acid may be measured at the various stages of the process, and there is less degradation of the cellulose as shown by the  $\alpha$ -cellulose content. B. P. RIDGE.

Recovery of the soda lyes from the industrial delignification of Tripoli esparto. C. PADOVANI and E. SALMOIRAGHI (Annali Chim. Appl., 1929, 19, 23-45).—In the autoclave treatment of these lyes the best clarification is obtained at 350°, the pressure exceeding 200 atm. Under this treatment the lyes undergo a species of "cracking," about 80% of the original organic matter being converted into tar of high calorific value. No methyl alcohol is formed, but acetic acid is obtained in amount approximating to that originally present. In general, treatment of the lyes subsequent to chlorination is uneconomical unless they are subjected to concentration. A systematic study was made of the influence on the results of the maximum temperature reached and of the velocity of heating. T. H. POPE.

Can the mechanical wood-pulp content of paper be determined to a fraction of a per cent? KORN (Papier-Fabr., 1929, 27, 142-143).-The mechanical wood pulp present may be determined either microscopically or chemically. The former method needs considerable dexterity and experience, and results accurate only to  $\pm 5\%$  are obtained under the best conditions. In chemical methods the complete separation of the wood pulp from the rest of the paper is very difficult, so that indirect methods with their inherent drawbacks are used. Halse's method depends on the determination of the lignin content of the paper and calculation from this of the wood-pulp content by means of a formula. The latter depends on an average value for the lignin content of sulphite-cellulose of 3%, but for the same paper the amounts of lignin found may vary within wide limits. A further complication is introduced by variations in the moisture content of the material examined. It is concluded that the determination of the wood-pulp content to within 1% is impossible. B. P. RIDGE.

Rosin sizing [of paper]. S. R. H. EDGE (Proc. Tech. Sect. Papermakers' Assoc., 1928, 9, 56-68).— See B., 1928, 564.

See also A., Mar., 245, Röntgen diagrams of cellulose (HERZOG and JANCKE). Structure of cellulose (MARK and MEYER). 299, Allylcellulose (SAKU-RADA). Action of fatty acids on cellulose (MALM and CLARKE). 300, Cellulose xanthamides (NAKA-SHIMA). 342, Relation between cystine yield and total sulphur in wool (RIMINGTON).

## PATENTS.

Process for hydrophilising flax in the raw or retted state to obtain hydrophilous wadding therefrom. P. HADDAN. From Soc. DES TEXTILES ROANNAIS (B.P. 305,709, 10.11.27).—The material is boiled for 1-2 hrs. with an alkaline solution containing, e.g., 2 pts. of caustic soda, 2 pts. of sodium carbonate, 1 pt. of Castille soap, and 0.2 pt. of milk of lime per 100 pts. of water, and, after rinsing, is soaked in 0.2% sodium hyposulphite solution at not above 80° for 1 hr. After washing and drying, the fibres are treated first with 0.5% sulphuric acid, then with a bleaching agent, preferably "chlorozone," and finally for about 1 hr., with agitation, with a solution containing, e.g., water 100 pts., sulphuric acid (d 1.835) 2 pts., sodium hyposulphite 0.2 pt., and nitric acid 0.1 pt. Between each of these treatments the fibre must be thoroughly D. J. NORMAN. washed.

Manufacture of [moiré patterned] fabrics or textile materials. BRIT. CELANESE, LTD. (B.P. 280, 195, 27.10.27. U.S., 4.11.26).—Moiré effects which are permanent may be produced on fabrics containing cellulose esters or ethers by application of heat and pressure according to known processes. [Stat. ref.] F. R. ENNOS.

Manufacture or treatment [cutting] of textile products. BRIT. CELANESE, LTD., and W. A. DICKIE (B.P. 305,257, 2.11.27).—The operation of cutting fabrics containing cellulose esters or ethers by hot knives or dies in order to seal them against fraying is facilitated if, before cutting, the fabric is sprayed with high-boiling softeners or solvents, *e.g.*, diacetone alcohol. D. J. NORMAN.

Manufacture of acetylcellulose. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 305,601, 5.9.27).— Before acetylation, the cellulose is treated continuously, e.g., by percolation, with formic acid of at least 80%concentration at ordinary or raised temperatures in the absence of esterifying agents, until it contains 4-10%by wt. of chemically combined formic acid.

#### F. R. Ennos.

Precipitation of viscose solutions. I. G. FARB-ENIND. A.-G. (B.P. 278,716, 5.10.27. Ger., 5.10.26).— The precipitating bath used has a high content of sulphuric acid and sodium salts, the concentrations of which are so selected that on cooling to the crystallising point "trisodium hydrosulphate,"  $Na_3H(SO_4)_2, H_2O$ , forms the first deposit. F. R. ENNOS.

Manufacture of artificial threads, filaments, ribbon, etc. COURTAULDS, LTD., E. HAZELEY, and E. A. MORTON (B.P. 305,279, 7.11.27).—In order to prevent the threads from wrapping round the spraying device when centrifugally-spun artificial silk is washed in a rotating spinning box by means of a non-rotating spray, two or more rods, or a cylinder of wire gauze, interposed between the cake of silk and the spraying device, are so arranged that they rotate with the box.

D. J. NORMAN.

Treatment of sheets of cellulose pulp for use particularly in the manufacture of viscose, and apparatus therefor. L. C. P. JARDIN (B.P. 286,619, 5.3.28. Fr., 4.3.27).—In order to avoid the use of hydraulic presses in the preparation of alkali-cellulose, the pulp sheets are uniformly impregnated with just that quantity of caustic soda solution necessary for the formation of alkali-cellulose. D. J. NORMAN.

Manufacture of cellulose esters and conversion products therefrom. C. EBERT and T. BECKER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,701,229, 5.2.29. Appl., 17.2.27. Ger., 22.2.26).—See B.P. 289,973; B., 1928, 920.

Preparation and spinning of fibrous materials for production of yarns. M. Conlan (B.P. 305,797, 24.1.28).

Apparatus for spinning artificial threads. RUTH-ALDO CO., INC., Assees. of (MISS) M. KELIN (B.P. 293,424, 4.7.28. Fr., 6.7.27).

**Production of twisted artificial silk threads etc.** O. SINDL (B.P. 305,280, 28.11.27).

Devices for stretching artificial threads made by the dry-spinning method. RUTH-ALDO CO., INC., Assees. of R. A. J. THENOZ (B.P. 283,139, 4.1.28. Fr., 4.1.27).

Apparatus for manufacture of films from cellulose solution. FELDMÜHLE PAPIER- U. ZELLSTOFFWERKE A.-G. (B.P. 290,233, 10.5.28. Ger., 10.5.27).

Drying apparatus for cellulose sheets etc. H. HAAS (B.P. 306,359, 17.7.28. Ger., 22.2.28). Manufacture of plastic material [from fibres and asphalt etc.]. E. THOMAS (B.P. 305,973, 11.11.27).

Drying of paper (B.P. 279,816). Friction material (B.P. 284,269).—See I. Nitrocellulose solutions (B.P. 305,148).—See XIII. Bonding of india-rubber and cellulose derivatives (B.P. 305,745).—See XIV.

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

Bleaching of Phormium tenax. P.W. AITKEN (New Zealand J. Sci. Tech., 1928, 10, 236-240).-Methods of bleaching the material by means of hypochlorite and permanganate solutions are compared with the usual wiring and grassing method from the point of view of cost, ease of manipulation, permanency of the colour obtained, and conservation of the strength of the product. When adequate allowance is made for cost of the bleaching agents, labour, and other charges, considerable economy is still effected by use of the above solutions in place of the grassing. Bleaching may be carried out in the vat, or by the chain method as a continuous process, and if in the case of the permanganate solutions the temperature is raised above 54° only a few minutes' treatment is required and labour charges for a continuous process are reduced to a minimum. No loss of strength occurs as a result of these treatments beyond that occurring during grassing. The colour of the material is different from that obtained by weathering, but is reasonably permanent, and is considered superior. Use of sulphur dioxide as a bleaching agent is not a success. B. P. RIDGE.

Colour of light sources. I. Carbon arcs. II. Daylight lamps. P. W. CUNLIFFE and H. LANIGAN. III. Sunlight and skylight. P. W. CUNLIFFE (J. Text. Inst., 1929, 20, 21-46 T).—Preliminary work to the study of the fastness to light of dyes on textile fabrics. B. P. RIDGE.

Influence of titanium white on coal-tar dyes. KEIDEL; WAGNER.—See XIII.

#### PATENTS.

Bleaching of paper pulp. J. RUTHS, ASST. to RUTHS ACCUMULATOR ARTIEBOLAG (U.S.P. 1,700,647, 29.1.29. Appl., 20.10.21. Swed., 9.1.20).—Steam required in the short-time bleaching of paper pulp is supplied from a steam accumulator. D. J. NORMAN.

Production of dyeings and printings by means of vat dyes. DURAND & HUGUENIN Soc. ANON. (B.P. 281,336, 28.11.27. Ger., 29.11.26. Addn. to B.P. 220,964; B., 1925, 879).—In the process of the prior patent the acid required for accelerating oxidation of the ester salt of a leuco-vat dye is derived from a substance such as mono- or di-chlorohydrin, chloroethyl alcohol, or potassium methyl (or ethyl) sulphate which is included in the printing or dyeing composition.

A. J. HALL.

Transference of dyestuffs to fabrics. H. S. SADTLER (B.P. 284,676, 3.2.28. U.S., 3.2.27).—Coloured designs on textile fabrics are obtained by use of transfer paper consisting of grease-proof paper printed with inks prepared with water-soluble dyes and glycerin. The transfer paper is pressed upon the wet fabric after dusting

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with a carbohydrate such as starch, and then is covered with a damp cloth and ironed; increased fastness of the colours on the fabric is obtained. A. J. HALL.

Production of reserves on wool and silk. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 305,269, 4.11.27).—Wool and silk are reserved against acid or neutral dyes by adding sulphonated natural resin (colophony) to the dye-bath. C. HOLLINS.

Coloration of materials made with or containing cellulose acetate. BRIT. CELANESE, LTD., G. H. ELLIS, and H. C. OLPIN (B.P. 305,560 and 305,566, 7.11.27, Addn. [A] to B.P. 239,470 [B., 1925, 879], and [B] to B.P. 237,943 [B., 1925, 801]).-(A) The following diphenylamines, selected from the general claim of the prior patent, are used for dyeing acetate silk : 4-chloro-2nitro-3'-methyl-, 4-chloro-2-nitro-4'-ethoxy-, 4-chloro-2nitro-4'-acetamido-, 4-bromo-2-nitro-, 4-bromo-2-nitro-4'-methoxy-, 2-nitro-, 2-nitro-4-methyl-, 2-nitro-4'ethoxy-4-methyl-, 4-chloro-2'-nitro-, 4-bromo-2'-nitro-, 2-nitro-4'-acetamido-diphenylamines. (B) The same compounds are used in the form of dispersions with the agents recommended in B.P. 219,349 and 224,925 (B., 1924, 906; 1925, 39). C. HOLLINS.

Revival of the colour of flowers or other patterns on carpets, rugs, mats, etc. M. E. DEANE (B.P. 304,172, 8.9.27).—The faded parts of coloured materials are painted with a suitable dye dissolved in methylated spirit. A. J. HALL.

Manufacture of nitrosoamine printing colours. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 305,787,11.1.28. Addn. to B.P. 303,942; B., 1929, 239). -A nitroscamine and a coupling component are dissolved separately in water, filtered from impurities, mixed, and evaporated to give a dyeing preparation of the Rapid Fast type. Premature colour-formation is prevented if the solutions are sufficiently concentrated before mixing, and a considerable amount of alkali is present. E.g., Naphthol AS (100 pts.) and concentrated aqueous sodium hydroxide (110 pts.) in hot water (300 pts.), and o-nitroaniline (52.6 pts.) in the form of nitrosoamine salt and sodium hydroxide (8 pts.) in hot water (200 pts.), are filtered separately, mixed, and evaporated in a vacuum. The solution of the coupling component may be evaporated separately to dryness before adding the nitrosoamine solution. C. HOLLINS.

Mercerisation of vegetable materials with alkaline liquids. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 305,509, 6.9.27).—Wetting-out agents suitable for use with highly concentrated alkaline liquors and prepared by adding a sulphonated aromatic compound to sulphonated oleic or ricinoleic acid are added to the mercerising lyes. Examples are dibutylnaphthalenesulphonic acid with sulphonated dihydroxystearic acid, or the sodium salt of tetrahydronaphthalenesulphonic acid with sulphonated oleic acid.

## A. J. HALL.

Treatment [delustring] of [viscose] artificial silk. C. and T. TERRELL (B.P. 305,828, 17.2., 14.3., and 13.6.28).—The material is impregnated with an aqueous solution of a salt, *e.g.*, barium chloride, sodium silicate, or a soluble calcium salt, dried, and treated with an aqueous solution of another salt, *e.g.*, ammonium sulphate, aluminium nitrate, or ammonium carbonate, which reacts with the first to form an insoluble, colourless, opaque precipitate in and/or on the filaments, which are finally softened by washing in a soap solution.

F. R. ENNOS.

Treatment [fireproofing] of fabrics or other materials or articles made from combustible material. BRIT. CELANESE, LTD. (B.P. 296,344, 10.8.28. U.S., 29.8.27).—Textile materials, e.g., cellulose esters or ethers, are steeped in an aqueous solution at 15—16° containing arsenic, stibinic, or phosphoric acid, rinsed, and dried. Cellulose acetate silk after steeping for 1 hr. at 40° in a 10% solution of phosphoric acid melts, but does not ignite when held in a flame. Aluminium acetate may also be added to the steeping liquor.

A. J. HALL.

Dyeing and like machines. C. S. BEDFORD (B.P. 305,901, 27.6.28. Addn. to B.P. 290,138).

[Winding] process for retting, boiling, bleaching, or otherwise treating yarn or rove with liquids. M. WADDELL and H. C. WATSON (B.P. 306,206, 25.11.27).

[Apparatus for] drying or drying and bleaching fabrics. M. M. KASANOF (B.P. 301,783, 5.9.27).

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

Manufacture of synthetic nitric acid from nitrogen oxides under pressure. V. I. MALVAREVSKI and V. V. PAPKOV (J. Chem. Ind. Moscow, 1928, 5, 682-689).—The absorption of nitrogen oxides in water under pressure is advocated; 65—67% nitric acid could thus be obtained. Water-cooling is necessary. A projected plant is described. CHEMICAL ABSTRACTS.

Determination of small amounts of silica in orthophosphoric acid. P. ALBRECHT (Chem.-Ztg., 1929, 53, 118—119).—Phosphoric acid is mixed with 5 times its weight of silver nitrate and heated in a platinum dish at 100—120° for 2 hrs., when the silicophosphoric acids are irreversibly decomposed with formation of silver phosphate and insoluble silicic acid. Soluble salts are dissolved in warm dilute nitric acid, and the solution is filtered. The insoluble matter is ignited and weighed before and after treatment with hydrofluoric acid. J. S. CARTER.

New process for the manufacture of caustic soda and sodium carbonate. W. SIEGEL (Chem.-Ztg., 1929, 53, 145-147).-The process of the "Ring" Ges. chem. Unternehmungen for the manufacture of caustic soda from common salt consists of three steps: (1)  $CaF_2 + SiF_4 + 2NaCl = Na_2SiF_6 + CaCl_2$ ; (2)  $Na_2SiF_6 = 2NaF + SiF_4$ ; (3)  $2NaF + Ca(OH)_2 =$  $2NaOH + CaF_2$ . The silicon fluoride from step (2) and the calcium fluoride from step (3) are used in step (1). If sodium carbonate is required the milk of lime in step (3) is replaced by calcium carbonate. Step (3) presents no difficulties, and the caustic soda can be produced at a higher concentration than from the carbonate. Step (1) is carried on in solution acidified with hydrochloric acid, and may be written as follows : (A)  $3 \operatorname{SiF}_4 + 4\operatorname{H}_2O = 2\operatorname{H}_2\operatorname{SiF}_6 + \operatorname{Si}(O\operatorname{H})_4$ ; (B)  $\operatorname{H}_2\operatorname{SiF}_6 + 2\operatorname{NaCl} = \operatorname{Na}_2\operatorname{SiF}_6 + 2\operatorname{HCl}$ ; (c)  $3\operatorname{CaF}_2 + \operatorname{Si}(O\operatorname{H})_4 + 2\operatorname{NaCl} + 4\operatorname{HCl} = \operatorname{Na}_2\operatorname{SiF}_6 + 3\operatorname{CaCl}_2 + 4\operatorname{H}_2O$ . No acid

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is therefore consumed, apart from small losses. The reaction is carried out in a conical vessel, into the lower part of which silicon fluoride is led. It is important to obtain a good suspension of the calcium fluoride. Step (2) presents the chief difficulties. The dissociation pressure of sodium silicofluoride reaches a workable figure at about 700°, but as decomposition proceeds the pressure falls off owing to the formation of a solid solution of m.p. 717°. The final driving-off of the last traces of silicon fluoride is a slow process, and any which may remain results in the caustic soda becoming contaminated with sodium silicate. It might be possible to work below 700° with injection of indifferent gas or superheated steam, but this would lead to difficulty in recovering the silicon fluoride. The theoretical heat balance shows a consumption of only 260 kg.-cal. per kg. of NaOH. A modification of step (3) employs calcium cyanamide, whereby sodium carbonate and ammonia are obtained ; a further variation involves the reaction  $2CaF_2 + SiO_2 + 6C = SiF_4 + 2CaC_2 + 2CO$ , carried out in an electric furnace. By incorporating this latter in the cycle, hydrochloric acid is obtained as a by-product. The advantages of this complication are doubtful. The process here described has not been proved on a works' scale. C. IRWIN.

Manufacture of oxygen from lime and chlorine. O. R. SWEENEY and A. W. RALSTON (Proc. Iowa Acad. Sci., 1927, 34, 215).—For the reaction of chlorine with a suspension of lime the optimal temperature is  $94^{\circ}$  and the optimal concentration of the nickel nitrate catalyst is 0.02 g. per 100 c.c. The nickel or cobalt catalyst is not easily poisoned. CHEMICAL ABSTRACTS.

Determination of ozone in air. M. S. EGOROV (Z. Unters. Lebensm., 1928, 56, 355-364).-The iodometric methods of Wartenberg and Podjaski (A., 1925, ii, 1198) and of Macdonnell (B., 1926, 273) for determining traces of ozone are not sufficiently sensitive, and the fluorescein method of Benoist (B., 1919, 285 A) is unsuitable owing to the slowness of the reaction. The following method was found to be suitable. Fluorescein (1-2 mg.) is dissolved in several drops of 10% sodium hydroxide solution and 10 c.c. of saturated sodium hydroxide are added. The mixture is shaken for several minutes with zinc dust, filtered through asbestos, and used fresh or stored in sealed tubes. 10 c.c. of 0.5% sodium hydroxide solution are placed in a test-tube with one drop of fluorescein solution prepared as described, and air is drawn through the liquid until the intensity of its fluorescence is equal to that of a standard fluorescein solution (standardised iodometrically). Not more than 8 litres of air should be drawn through at a rate of 12-15 litres/hr. The standard fluorescein solution should not be more dilute than 1:108, and the standardisation should be carried out at a dilution of 1:106. Nitrogen oxides and hydrogen peroxide do not affect the reagent. W. J. BOYD.

Automatic apparatus for determination of small concentrations of sulphur dioxide in air. II. M. D. THOMAS and J. N. ABERSOLD (Ind. Eng. Chem. [Anal.], 1929, 1, 14—15; cf. Thomas and Cross, A., 1928, 862).—The automatic recording of the change in iodine content of a solution is not easy, but the substi-

tution for it of distilled water, slightly acidified and containing 0.003-0.006% of hydrogen peroxide, makes it possible to determine the sulphuric acid produced by conductivity. The method is suitable for concentrations up to 60 p.p.m. of sulphur dioxide in air, and results agree well with those given by other methods. C. IRWIN.

Extraction of bromine by solvent. II. B. G. PANTELEIMONOV (J. Chem. Ind. Moscow, 1928, 5, 484– 489).—When kerosene is used for the extraction of bromine from aqueous solutions, solvent containing bromine is lost as an emulsion, the loss of bromine being 14-26% after 15 extractions. Absorption in sponge reduces the loss of kerosene from 1-2% to 0.5% per extraction. The bromine is thereby converted into hydrogen bromide, and the kerosene can thus be used again without treatment with alkali, the hydrogen bromide remaining in the aqueous liquid.

## CHEMICAL ABSTRACTS.

See also A., Mar., 256, Adsorption of gases and vapours by silica gels (BOSSHARD and JAAG). 257, Siloxen as adsorbent (KAUTSKY and BLINOFF). Adsorption of carbon dioxide by ferric hydroxide (TILLMANS and others). 267, Equilibrium between water and the nitrates and sulphates of sodium [and potassium] at 50–90° (CORNAC and KROMBACH). 274, Copper catalysts from precipitated hydroxide (FROLICH and others). 275, Electrolytic preparation of sodium perborate (GIORDANI and INTONTI). 284, Detection of thiocyanate, fluoride, and copper, and drop reaction for ammonia (FEIGL and others).

Soda lyes from treatment of esparto. PADOVANI and SALMOIRAGHI.—See V. Evaluation of stibnite. MCNABB and WAGNER.—See X. Determination of iodine and potassium iodide. WINTERFELD and SIECKE.—See XX. Toxicity of hydrogen cyanide. ALLISON.—See XXIII.

# PATENTS.

Manufacture of hydrogen cyanide from cyanides of the alkali metals. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 305,860, 23.3.28).—Alkali cyanides are treated in the presence of water with a quantity of sodium bicarbonate at least 10% in excess of that required by the equation, NaCN + NaHCO<sub>3</sub> = Na<sub>2</sub>CO<sub>3</sub> + HCN, and the hydrogen cyanide is removed below 50° in vacuo. L. A. COLES.

Synthesis of ammonia. G. F. UHDE (B.P. 272,930, 28.12.27).—Catalysts active at comparatively low temperatures (380—450°) are prepared from material containing complex cyanides by heating it in the contact chamber by the admission of an externally heated mixture of hydrogen and nitrogen from which all traces of oxygen and compounds containing it have been removed. The proportion of hydrogen in the mixture, which may be initially as low as 10%, and the temperature and pressure of the gases are raised during the preparation of the catalyst until normal working conditions are attained. L. A. COLES.

Means for obtaining crystals of uniform coarse grain, especially of fertiliser salts. J. Y. Johnson. From I. G. FARBENIND. A.-G. (B.P. 304,872, 24.11.27).— A hot, saturated solution of, *e.g.*, ammonium sulphate is passed from a saturator through a crystallising tower in which the liquid is kept moving upwards by means of bubbles of air admitted at suitable intervals and by a spiral propeller. Coarse crystals fall to the bottom and are removed, whilst fine crystals pass out at the top of the tower and are returned to the saturator. In the production of ammonium bicarbonate etc., ammonia solution is charged in from the saturator, and carbon dioxide and ammonia gas are forced into the tower.

L. A. COLES.

Manufacture and purification of alumina. J. C. SEAILLES (B.P. 293,392, 3.7.28. Fr., 5.7.27).-Modifications for reducing the proportion of silica in the product obtained as described in B.P. 277,697 and 283,509 (B., 1928, 603, 230) comprise reduction of the temperature and/or time of treatment of the bauxite etc. with lime in the autoclave; addition of catalysts, e.g., calcium benzenesulphonate, calcium sulphoricinoleate, etc., during digestion; prevention of an excess of free alkali during treatment of the alkaline-earth aluminate with sodium carbonate, e.g., by previous extraction of free lime with water, by direct carbonation, or by the addition of sodium bicarbonate; and treatment of the sodium aluminate solution with material containing calcium aluminate, e.g., with material freshly discharged from L. A. COLES. the autoclave.

Production of green hydrated chromium oxide. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 304,809, 24.10.27).—Chromic acid or a chromate is heated with water and a reducing agent, *e.g.*, carbon monoxide, hydrogen, or mixtures of these (sulphur dioxide being excepted), under a raised pressure not exceeding 150 atm. L. A. COLES.

Manufacture of pure hydrated chromic chloride. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 306,305, 23.3.28. Addn. to B.P. 271,016; B., 1927, 522). —Anhydrous chromic chloride is treated with a quantity of water equal to or not greatly exceeding that required to form the crystals, CrCl<sub>3</sub>,6H<sub>2</sub>O, in the absence of air and in the presence of catalytic reducing agents, preferably chromous chloride. L. A. COLES.

Impregnated gel for adsorbing water vapour. SILICA GEL CORP., Assees. of E. B. MILLER and G. C. CONNOLLY (B.P. 280,934, 17.11.27. U.S., 19.11.26).—The material comprises silica gel, with pores of such a size that it will adsorb not less than 10% of its weight of water vapour at 30° and 22 mm. pressure, impregnated with a substance which will combine reversibly with water vapour, e.g., copper sulphate. L. A. COLES.

Manufacture of carbonic snow [solid carbon dioxide]. E. DU BOIS (B.P. 304,958, 3.4.28).—Liquid carbon dioxide is passed through a spiral coil terminating in a jet with an enlarged mouthpiece, situated at the top of a suitable receptacle in which the solid collects and whence it can be removed through an opening at the bottom. Carbon dioxide gas liberated by evaporation passes through a screen of fine mesh placed above the spiral coil, and, after passage through a double cavity in the walls of the receptacle, is returned to the compressor. L. A. COLES. Catalytic oxidation of sulphur dioxide. A. O. JAEGER, ASST. to SELDEN CO., and J. A. BERTSCH (U.S.P. 1,695,285, 18.12.28. Appl., 6.2.26).—Sulphur dioxide gases containing hydrogen chloride or catalyst poisons other than arsenic are oxidised by zeolite catalysts free from platinum and containing vanadium or other catalytically active elements. R. BRIGHTMAN.

Recovery of ammonia from waste waters in manufacture of artificial filaments by the cuprammonium process. Recovery of ammonia from dilute solution. H. HOFMANN, Assr. to AMER. BEMBERG CORP. (U.S.P. 1,701,110 and 1,701,265, 5.2.29. Appl., [A] 21.4.25, [B] 29.1.27. Renewed [A] 13.6.28. Ger., [A, B] 6.5.24).—See B.P. 233,669; B., 1925, 955.

Manufacture of ammonium nitrate in water solution and simultaneous concentration thereof. C. TONIOLO (U.S.P. 1,700,914, 5.2.29. Appl., 27.1.26. It., 7.2.25).—See B.P. 247,227; B., 1927, 408.

**Production of aluminium oxide.** S. E. SIEURIN (U.S.P. 1,701,510, 12.2.29. Appl., 4.6.24. Swed., 15.6.23).—See B.P. 217,568; B., 1925, 545.

Cyanides (B.P. 304,421).—See II. Catalytic oxidation (B.P. 291,419).—See III. Chromium compounds (B.P. 305,712). Vessels with acid-proof linings (B.P. 290,189).—See X. Fertilisers (B.P. 305,760).—See XVI.

# VIII.-GLASS; CERAMICS.

White stoneware and other white ceramic masses. F. SINGER (Chem.-Ztg., 1929, 53, 105—106, 126—129).—The methods of manufacturing chemical stoneware and porcelain are briefly described, and a short account of their properties and microstructure is given. The mechanical properties of stoneware or earthenware are dependent to a considerable extent on the form and distribution of the mullite produced during firing. A. R. POWELL.

Slagging of refractory materials. II. Influence of the chemical composition of the slags. H. SALMANG and F. SCHICK (Arch. Eisenhüttenw., 1928-9, 2, 439-447; Stahl u. Eisen, 1929, 49, 187-189).-The corrosive action of various slags on a highgrade fireclay crucible at 1410° has been examined. Slags with a high content of ferrous oxide are very fluid, but have a low wetting power and high surface tension, whereas those with a high content of manganous oxide are more viscous and have a low surface tension and high wetting power; both types are highly corrosive. The slagging action of various metal oxides on fireclay decreases in the order : lead, ferrous, manganous, calcium, ferric, potassium, sodium, manganic oxide, but in molten slags the order of decreasing activity of the basic oxides is lime, ferrous oxide, manganous oxide, ferric oxide, magnesia. In highly basic slags from iron smelting, alumina acts as an acidic constituent and tends to reduce the corrosive action of the bases. Singlesilicate slags containing less of the base than corresponds with RO,SiO, have no corrosive action on fireclay, but with magnesium aluminosilicate slags the addition of lime causes a progressive increase in the corrosive action, as also does addition of ferrous or manganous oxide to

calcium aluminosilicate slags. The slagging power for fireclay at 1410° in 1 hr. of a slag may be expressed by the formula  $K = 9F[(CaO) + 5(FeO) + 4(MnO) + (MgO) + (Fe_2O_3)]/[(Al_2O_3) + 2(SiO_2) + 4(P_2O_5) + (S) + (SO_3)],$ where F is a constant depending on the clay, and the analysis figures (%) are substituted for the oxide formulæ. A. R. POWELL.

See also A., Mar., 280, Rehydration of metakaolin and synthesis of kaolin (VAN NIEUWENBERG and PIE-TERS).

## PATENTS.

Moulding of [powdered] ceramic material. H. L. CROWLEY, ASST. to ISOLANTITE CO. OF AMERICA, INC. (U.S.P. 1,699,502, 15.1.29. Appl., 11.1.27).—The material is formed into a block under great pressure while the air is evacuated. The block is then subjected *in vacuo* to a second and greater pressure, which changes the shape without causing the block to crumble. F. SALT.

Manufacture of light-weight ceramic material. R. ERICSON, ASST. to U.S. GYPSUM CO. (U.S.P. 1,702,076, 12.2.29. Appl., 14.10.27).—After incorporating a tenacious foam in a fluid plastic mixture of finely-divided argillaceous material and water, the mixture is shaped, dried, and finally heated to form a hard, cellular product. H. ROYAL-DAWSON.

Pottery decoration. LOVATT & LOVATT, LTD., and A. E. LOVATT (B.P. 303,679, 20.2.28).—Pottery ware is subjected to a light biscuit fire and then dipped in a mixed glaze, prepared by mixing colouring matter, which has been previously fired and crushed to suitable particle size, with raw or unfired glaze. The glaze is then fired on at a comparatively high temperature.

F. SALT.

Compound glass sheets. J. A. WATT (B.P. 305,404, 30.3.28).

Production of graded tinting on glass. O. P. RAPHAEL (B.P. 305,436, 5.7.28).

Apparatus for making glass threads or filaments. S. G. S. DICKER. From GOULD STORAGE BAT-TERY Co., INC. (B.P. 306,332, 26.4.28).

# IX.—BUILDING MATERIALS.

Determination of iron and aluminium oxides, magnesium oxide, and calcium oxide in Portland cement. J. S. PIERCE and W. C. SETZER (Ind. Eng. Chem. [Anal.], 1929, 1, 25-26).-After removal of silica, iron and aluminium oxides are precipitated at  $p_{\rm H}$  7, using bromothymol-blue as indicator. The slightly blue filtrate is acidified, boiled, and 10% of its volume of saturated alcoholic trinitrobenzene added. Carbonate-free sodium hydroxide solution is run in until a deep red colour is obtained. The precipitated magnesium hydroxide is dissolved in excess of standard acid, and the excess acid is titrated, using dimethylaminoazobenzene as indicator. The calcium in the filtrate is determined similarly. The method is an approximate one suitable for rapid control tests. C. IRWIN.

Effect of temperature on the strength of concrete. C. C. WILNY (Eng. News-Rec., 1929, 102, 179—181).— Equal strength of concrete matured at 38° is obtained in about half the period required for that matured at 21°, which again requires only half the time of concrete matured at 2°. At all ages between 3 and 28 days the difference in strength was represented by 54 lb./in.<sup>2</sup> per degree. Frozen concrete showed practically no increase in strength, and it is recommended that all concrete should be protected from freezing for at least a week at 21°, or for twice as long at 2°. Loss in strength of concrete after 7 days' hardening was small, and it was indecisive whether freezing after this period resulted in any permanent damage, but when frozen at the age of 1 day the strength was less than half that of unfrozen material, and freezing at 3–5 days indicated a permanent loss of strength of 15–20%. C. A. KING.

Prolonged tests on concrete made with various ballasts, especially blast-furnace slag. A. GUTT-MANN (Arch. Eisenhüttenw., 1928—9, 2, 401—403; Stahl u. Eisen, 1929, 49, 213).—A series of tests extending over ten years on concretes made with blast-furnace slag, granite, limestone, and gravel as ballast showed that slag ballast was slightly more satisfactory than natural ballast. The sulphur content of the slag appeared to have no accelerating effect on the corrosion of iron embedded in the concrete. A. R. POWELL.

Absorption of wood preservatives. J. D. Mac-LEAN (Eng. News-Rec., 1929, 102, 176-179).-Considerable variation in results occurs when timbers of different lengths and cross-sections are treated with preservatives in accordance with present specifications. The most satisfactory and convenient method of specification is in weight (lb.) of preservative per cub. ft. of timber, provided that consideration is taken of the ratio of surface area to volume. This proviso is unnecessary in the case of sapwood or porous wood, but the heartwood of most species will take only a limited penetration, so that absorptions widely differing from an average value are obtained with different sizes of timber. The side and longitudinal penetrations of a number of airseasoned heartwood specimens gave an average ratio of about 1:15 for preservative oils and 1:20 for water solutions. C. A. KING.

Bituminous substances. SUIDA and SCHMÖLZER. Road asphalts. BURSTYN.—See II.

# PATENTS.

Production of Portland cement. H. R. DURBIN, Assr. to Internat. Cement Corp. (U.S.P. 1,700,032-3, 22.1.29. Appl., [A] 12.6.26, [B] 22.1.27).-(A) Portland cement, having a tensile strength of not less than 240 lb./in.2 after 24 hrs. in a 1:3 sand mixture, is prepared by burning Portland cement clinker containing a large proportion of tricalcium silicate and incompletely lime-saturated silicate intimately mixed with just sufficient lime to complete the saturation of the silicate, and grinding this clinker to cement. (B) A similar cement, having a tensile strength greater than 300 lb./in.<sup>2</sup> after 24 hrs. in a 1:3 sand mixture, is prepared by adding silica, alumina, and iron, together with an amount of lime which is theoretically necessary to saturate the silica, alumina, and iron, but which is considerably in excess of that which will combine with these elements in one burning operation. The mass is burned to incipient fusion, ground, re-burned to incipient F. SALT. fusion, and again ground.

Direct vitreous coating of surfaces such as walls, constructional ironwork, roofing, fencing, etc. R. F. BANKS (B.P. 306,256, 21.1.28).—The surfaces are sprayed with a mixture of cement and solutions of sodium silicate and barium chloride, with or without a solution of acid potassium oxalate, and are thereby glazed and rendered waterproof.

H. ROYAL-DAWSON. Aggregating particles of wood. F. LAUTER, ASST. to LIGNEL CORP. (U.S.P. 1,702,013, 12.2.29. Appl., 18.11.27).—Wood particles containing substantial amounts of natural resin are treated with an aldehyde solution, dried, and subjected simultaneously to the action of heat and pressure ; the artificial resin produced acts as a binder for the wood. H. ROYAL-DAWSON.

Manufacture of [recessed] bricks or building blocks. P. W. STEWART and G. H. C. RACTLIFFE (B.P. 306,210, 29.11.27).

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

Corrosion of iron in sodium chloride solution. W. VAN WÜLLEN-SCHOLTEN (Arch. Eisenhüttenw., 1928-9, 2, 523-530; Stahl u. Eisen, 1929, 49, 212-213).-When a smooth and a rough iron plate are immersed in sodium chloride solution and connected outside the vessel through a galvanometer a current flows from the smooth to the rough plate inside the cell. Agitation of the solution causes a reversal of this direction of flow, which is permanent with vigorous agitation, but gradually reverts to the original direction with slow stirring. In an atmosphere of hydrogen agitation has no effect. Potential measurements in the non-agitated solution show that the potential of iron to ferrous hydroxide is -0.569 volt and to ferric hydroxide -0.220 volt, but in the presence of oxygen a solid solution of these two oxides is obtained as a single phase, the ratio Fe"/Fe" in the phase being dependent on the oxygen concentration in the solution. A. R. POWELL.

Sulphur in electrolytic iron and its removal. K. OMA (Bull. Inst. Phys. Chem. Res. Tokyo, 1929, 8, 126—130).—Iron deposited electrolytically from ferrous ammonium sulphate baths may contain 0.001% of sulphur as sulphide derived from the anode material, and 0.02-0.03% as sulphate derived from the electrolyte. At high temperatures the latter may be reduced to sulphide and thence to hydrogen sulphide, and the rate of desulphurisation effected by heating in hydrogen above 800° is a function of time, temperature, and grain size. J. GRANT.

Manufacture of hollow drill steel in the electric furnace. T. M. PUGSLEY (Chem. Eng. Min. Rev., 1929, 21, 131-132).—The furnace is charged with scrap steel covered with 3% of lime and some fluorspar, and heating is continued until the metal contains less than 0.02% P, more lime and fluorspar being added if necessary. The slag is removed and the metal carburised to 0.6% C by stirring in anthracite; more lime-fluorspar slag is added, together with anthracite, to produce carbide in the slag, followed by the requisite quantity of manganese. The steel is then deoxidised

with a small quantity of ferrosilicon and poured. Details of the method of pouring and of working-down the ingots are given. A. R. POWELL.

Effect of the final rolling temperature on the properties of steel rails. R. STUMPER (Stahl u. Eisen, 1929, 49, 177–187).—The mechanical properties and structure of steel rails containing 0.4% C, 0.1% Si, 1.0% Mn, 0.06% P, and 0.04% S have been examined after finishing the rolling at temperatures between 950° and 1150°. The tensile strength, elongation, yield point, and impact strength decrease generally with increase of rolling temperature, which also causes a rapid increase in grain size. Hence the rails should be finished at as low a temperature (above 950°) as is compatible with convenient handling in the rolling mill. A. R. POWELL.

Resistance of some steels to chemical action in relation to their nickel, chromium, and carbon content. F. SCHMITZ (Z. Metallk., 1929, 21, 64-65).— The work of Guertler and Ackermann on carbon-free iron-chromium-nickel alloys (B., 1928, 753) has been repeated on nickel-chromium steels with similar results, chromium being the sole protective agent against corrosion in oxidising media and nickel in non-oxidising media. A high carbon content improves the resistance of plain carbon steels to mild corrosive agents, but tends to reduce the passivity of alloy steels, although it decreases scaling at high temperatures.

A. R. POWELL.

Systems iron-silicon, iron-chromium, and ironphosphorus. P. OBERHOFFER and C. KREUTZER (Arch. Eisenhüttenw., 1928-9, 2, 449-456; Stahl u. Eisen, 1929, 49, 189-190).-Röntgenographic examination of the iron-silicon system shows the y-field to be bounded by a smooth curve joining 1400° and 900° on the temperature axis and extending to 2.2% Si at 1150°. The heterogeneous  $(\alpha + \gamma)$ -field is bounded by a similar curve extending to 2.5% Si at 1180°. X-Ray analysis affords no evidence of the formation of an intermetallic compound in the iron-chromium system; the  $\gamma$ -field extends to about 15% Cr. In the iron-phosphorus system, alloys containing up to 1.11% P have the simple a-lattice structure, those with more than 2.28% P show in addition the lines of the compound Fe<sub>3</sub>P, and alloys with 14.2 - 21.2% P contain both Fe<sub>3</sub>P and Fe<sub>2</sub>P. With larger proportions of phosphorus new lines appear in the röntgenogram due to the presence of an, as yet, unknown constituent. A. R. POWELL.

Anomalies of annealing cold-worked copper and brass. F. EUGÈNE (Rev. Mét., 1928, 25, 685-706; 1929, 26, 29-42; cf. B., 1928, 861).—The changes in the physical and mechanical properties of cold-worked copper and brass on annealing through the recrystallisation zone are shown in a series of tables and graphs, and photomicrographs of the structural changes that take place in the zone between the two anomalies A1 and A2 are reproduced. The maximum softness and Erichsen value of copper are obtained by annealing at A2, whereas the malleability of brass increases with rise of annealing temperature between A2 and the temperature at which recrystallisation is complete. Micrographic examination shows that all the strained crystals have disappeared completely at A2, but internal stress is relieved as soon as the individual strained crystals are completely isolated from one another by areas of recrystallised metal. Dilatometric tests on cold-worked copper showed contraction to take place on annealing in all directions simultaneously, whereas with brass contraction takes place in a direction transverse to the direction of rolling and expansion in the direction of rolling; this effect is the more pronounced the higher is the zinc content.

A. R. POWELL.

Copper-cuprous oxide eutectic in copper refining. W. STAHL (Chem.-Ztg., 1929, 53, 137).—The formation of a cuprous oxide-copper eutectic containing  $3\cdot 4$ —  $3\cdot 5\%$  Cu<sub>2</sub>O is responsible for the fibrous structure which copper acquires during refining by poling. Similar behaviour is observed when pure copper is oxidised.

J. S. CARTER.

Brittleness in arsenical copper. II. C. BLAZEY (Inst. Metals, March, 1929. Advance copy. 6 pp.).— Following an observation of a type of brittleness in copper which disappeared on annealing, and re-appeared when the metal was cold-worked (cf. B., 1927, 280), the influence of phosphorus, lead, and bismuth has been determined. Phosphorus or antimony up to 0.1% was without ill-effect, but 0.004% of bismuth was harmful when no phosphorus was added before pouring, and the effect was characteristic of low-temperature annealing only. C. A. KING.

Testing of electrodeposits on aluminium. G. B. BROOK and G. H. STOTT (Inst. Metals, March, 1929. Advance copy. 9 pp.).-Methods designed to determine the probable durability of electroplating on aluminium included the determination of weight, thickness, and degree of porosity of the deposit. The measure of adhesion was shown by bending a strip of the plated metal helically around an iron rod under standard conditions, and the resistance to corrosion by immersing specimens in brackish water at half-tide level for three months. Considerable variation in thickness, soundness, and crystalline structure was found in specimens representing commercial practice. Generally, failures, e.g., cracking, blistering, peeling of the plating, were confined to single-metal deposits, some multiple deposits remaining free from these defects for long periods. A particular chromium-plated specimen showed evident poor adhesion. C. A. KING.

Rapid determination of tin in tinplate. W. A. MASEL (Chem.-Ztg., 1929, 53, 108).—A weighed sample is placed in a short test-tube with a small hole in the bottom and the tube is lowered, by means of a piece of iron wire round the neck, into a cylinder filled with a solution of chlorine in tin tetrachloride. Dissolution of the tin coating is complete in 3—4 min. when no further bubbles develop on the iron. The tube is removed and the iron thoroughly washed with benzine, dried, and weighed. The loss in weight, plus 0.1% for tin retained as a compound in the outer iron layer, is the amount of tin in the sample taken. A. R. POWELL.

Evaluation of stibnite. I. Determination of sulphur. W. M. McNABB and E. C. WAGNER (Ind. Eng. Chem. [Anal.], 1929, 1, 32-35).—The evolution method for the determination of sulphur in stibnite

gives results which, though concordant in themselves, are lower than those given by bromine oxidation. The difference was suspected to be due to the presence of free sulphur. Specimens of Chinese stibnite gave on extraction with carbon tetrachloride up to 0.10% of free They contained about 0.01% of sulphate sulphur. The same samples were analysed by the sulphur. bromine oxidation and evolution methods, and differences approximating to the actual free sulphur content were found. The evolution method, in which the hydrogen sulphate is evolved in a current of carbon dioxide or hydrogen, absorbed in ammoniacal cadmium solution, and the cadmium sulphide titrated with iodine, is therefore accurate. It permits of the antimony being determined from the same sample. C. IRWIN.

Variations in silver assaying. F. C. JOHNS (Chem. Eng. Min. Rev., 1929, 21, 152—154).—In the assay of lead-zinc sulphide ores and concentrates for silver equally good results are obtained by the scorification and pot-running (nitre) methods, whereas slightly low results are obtained by using an iron rod in the pot assay. With bone ash cupels there is a greater absorption of silver than with Mabor or Morganite cupels. Examples are given of the variations that may be expected in the assay of these products for silver; even with careful adjustment of the cupellation conditions differences of 1-4 oz./ton on material assaying 70—80 oz./ton may be obtained. A. R. POWELL.

Detection of gold and the platinum metals [in ores]. SPORCQ (Bull. Soc. chim. Belg., 1929, 38, 21-24). -The ore is smelted in the usual way to obtain a lead button, which is cupelled and the resulting bead is heated in a porcelain crucible with a few drops of nitric acid. When no further action is seen, a few drops of hydrochloric acid are added and the solution is evaporated on the water-bath to dryness. The residue is moistened several times with hydrochloric acid and dried to expel nitric acid completely. The final residue is moistened with 1 drop of hydrochloric acid, and 2 drops of a 20% solution of stannous chloride in 1:1 hydrochloric acid are added. If gold is present a blackishblue colour is obtained, and after keeping for some time a black precipitate separates; if platinum is present the supernatant liquid will be brown, and if palladium is present brick-red. Detection of as little as 0.01 g. of these metals per ton of ore is claimed when 100 g. are taken for the test. A. R. POWELL.

Annealing of metals in vacuo. L. GUILLET and A. Roux (Rev. Mét., 1929, 26, 1–11).—Three samples of commercial steel were annealed in vacuo and the rate of evolution of gas and its composition determined. With mild steel (0.04% C) gas continued to be evolved for 23 hrs. at 950°, the total volume expelled being 3.3 times the volume of the steel; the gas contained 40% H<sub>2</sub>, 37% CO, 22% N<sub>2</sub>, and 1% CO<sub>2</sub> by vol. From a cementation steel (0.13% C) 0.67 vol. of gas was evolved in 5 hrs., the gas containing 43% H<sub>2</sub>, 40% CO, 8% N<sub>2</sub>, and 9% CO<sub>2</sub> by vol., and from a steel with 0.45% C, 0.53 vol. of gas containing 41% H<sub>2</sub>, 39% CO, 14% N<sub>2</sub>, and 6% CO<sub>2</sub> by vol. was evolved in 4 hrs. Except in the case of mild steel, annealing *in vacuo* had little effect on the mechanical properties. The resistance to shock of mild steel was, however, considerably increased by vacuumannealing. Brass containing 67% Cu evolved 0.55 vol. of gas containing 65% H<sub>2</sub>, 14% CO, 7% N<sub>2</sub>, and 14% CO<sub>2</sub> by vol. on annealing for 2 hrs. at 720°, whereas 60:40 brass evolved only 0.39 vol. of gas containing 56% H<sub>2</sub>, 43% N<sub>2</sub>, and 9% CO<sub>2</sub> by vol. under the same conditions. A pure nickel wire evolved 22 times its volume of gas in 2 hrs. at 950° *in vacuo*; the gas contained by vol. 36% H<sub>2</sub>, 33% CO, 26% N<sub>2</sub>, and 5% CO<sub>2</sub>. Aluminium and duralumin commence to evolve gas at 300—400° *in vacuo*; the volume and composition of the gas vary very widely according to the previous history of the metal, but usually the gas is very rich in hydrogen and poor in nitrogen, and it frequently also contains methane. A. R. POWELL.

Determination of gases in metals. W. HESSEN-BRUCH (Z. Metallk., 1929, 21, 46-55).—A review of recent work, with especial reference to steel, giving diagrams of suitable apparatus and a summary of the results obtained by previous authors. A. R. POWELL.

Explosibility of sulphide dusts in metal mines. E. D. GARDNER and E. STEIN (U.S. Bur. Mines Rep. Invest., 1928, No. 2863, 11 pp.).—Sulphide dusts can be ignited by blasting, and combustion may proceed with explosive violence. CHEMICAL ABSTRACTS.

Cathodic yield in nickel-plating with high current density. Influence of oxidising agents and hydrogen-ion concentration. BALLAY (Compt. rend., 1929, 188, 556-559).—Nickel-plating on steel from a solution containing 450 g. of nickel sulphate, 22 g. of nickel chloride, and 22 g. of boric acid per litre for 15 min. at 50° with a current density of 10 amp./dm.<sup>2</sup> gives a yield of over 95%, which is only slightly influenced by small amounts of oxidising agents, though excess of nitric acid or nitrates, or to a smaller extent potassium permanganate, diminishes it considerably. The optimum  $p_{\rm H}$  range is  $4 \cdot 4 - 5 \cdot 5$ . J. GRANT.

Importance of design and setting of large kettles used for refining and low m.p. alloys. H. C. LANCASTER (Inst. Metals, Mar., 1929. Advance copy. 6 pp.).

See also A., Mar., 248, Recrystallisation of metals (KARNOP and SACHS). 259, Colloidal gold (NICOL). 270, Passivity of metals (EVANS). 271, Mechanism of corrosion (EVANS). 275, Electrolytic precipitation of metals (ARNDT). Theory of electrodeposition of chromium (Müller and Ekwall). 287, Microtitration of bismuth (STRAUB). Determination of tantalum and niobium (SEARS). Determination of osmium (CROWELL and KIRSCHMAN). 289, Iron ores of S. Africa (KRENKEL).

Gas for cutting and welding. SCHOLTZ.—See II. Testing for corrosion. DUFFEK.—See XI.

# PATENTS.

Apparatus for roasting and/or sintering ores etc. COMP. DES MÉTAUX OVERPELT-LOMMEL (B.P. 294,197, 30.3.28. Belg., 20.7.27).—Sintering apparatus of the Dwight-Lloyd type is provided with one or more joints in series with the usual joint between the movable grate and the suction or blowing box so as to form one or more chambers inside which a pressure is maintained, by means of a fan, equal to that in the suction or blowing box. An automatic regulating device is provided for keeping these pressures in equilibrium.

A. R. POWELL.

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Reduction of ores. F. L. DUFFIELD (B.P. 304,174, 10.10.27).—A charge of iron ore, carbonaceous material, and lime is introduced into the base of a vertical furnace by means of a horizontal ram working in conjunction with a vertical ram which intermittently elevates the charge into progressively hotter zones of the furnace, the reduced metal being discharged at or near to the top of the chamber. A one-way stop-valve supports the charge on the downward stroke of the ram. Gases evolved in the reducing chamber are burned in an adjacent heating chamber, the gases passing in a downward direction and providing the necessary heat for the reducing process. C. A. KING.

Chill-cast iron alloys. Grey-iron alloys. INTER-NATIONAL NICKEL CO., Assees. of P. D. MERICA, J. S. VANICK, and T. H. WICKENDEN (B.P. 279,414—5, 29.9.27. U.S., 21.10.26).—(A) A chilled cast-iron alloy, a portion at least of which is of chilled white iron, contains 0.25-2% Si, 2—10% Ni, 1—4% Cr, and 0.25-2% Mn, besides the usual carbon content of cast iron. (B) A cast-iron alloy resistant to wear contains 0.25-3% Si, 1—12% Ni, up to 3% Cr, 2—4%C, and the usual content of manganese, sulphur, and phosphorus. [Stat. ref.] A. R. POWELL.

Manufacture of grey cast iron with low carbon content and of any desired composition. MAS-CHINENFABR. ESSLINGEN (B.P. 291,112, 25.5.28. Ger., 27.5.27. Addn. to B.P. 260,990; B., 1927, 960).—On the basis of the relation between the thickness of wall, the composition of metal, and the formation of structure of grey cast iron, the carbon content is maintained constant and the structure determined by variation of the silicon content in accordance with a graph correlating the wall thickness and the silicon content for a given percentage of carbon. C. A. KING.

Manufacture of malleable cast iron. C. E. KLUJJTMANS and W. H. W. PROCTOR (B.P. 288,980, 14.4.28. Fr., 16.4.27).—A charge consisting of equal parts of steel scrap and the residues from previous melts is smelted in a cupola with the addition of ferrosilicon and ferromanganese to obtain an iron containing up to  $2 \cdot 9\%$  C,  $1 \cdot 1 - 1 \cdot 5\%$  Si,  $0 \cdot 42 - 0 \cdot 75\%$  Mn,  $0 \cdot 1 - 0 \cdot 2\%$  P, and less than  $0 \cdot 2\%$  S. The castings are embedded in sand or waste iron ore, heated for 12 hrs. at 680—730°, 12 hrs. at 800—830°, and 48 hrs. at 880—900°, cooled to 700° at the rate of 5—8°/hr., and then cooled rapidly to produce black-core malleable iron. A. R. POWELL.

Increasing the elastic limit or yield point of tough or plastic metals. G. KERÉKGYÁRTÓ (B.P. 295,390, 7.8.28. Hung., 11.8.27).—Wrought iron or steel shafts, tubes, etc. are subjected to a torsional stress beyond the elastic limit or yield point, and then annealed at a low temperature to relieve internal stress.

A. R. POWELL.

**Proofing of iron and steel against rust.** W. H. COLE (B.P. 305,386, 28.2.28. Addn. to B.P. 289,906; B., 1928, 488).—The pickling solution is made by

dissolving in hot phosphoric acid (1 litre of acid,  $d \cdot 45$ , diluted to  $d \cdot 09$ ) 150 g. of zinc oxide, 250 g. of ferric oxide, 10 g. of chromium oxide, 10 g. of aluminium powder, 3 g. of potassium dichromate, 4 g. of potassium chromate, 2 g. of ammonium phosphate, and 6 g. of naphthalene. A. R. POWELL.

[Heat-resisting chromium-iron] alloys. T. D. KELLY (B.P. 304,893, 23.12.27).—Iron of a good quality is melted by means of a flame produced from powdered anthracite, and to every 100 lb. of iron is added an alloy consisting of (a) 5—20 lb. of chromium, 5—20 lb. of nickel, and 5—20 lb. of copper, (b) 5—20 lb. of chromium, 10—20 lb. of nickel, 10—20 lb. of copper, and 60—80 lb. of carbon-free iron, or (c) 5—20 lb. of chromium and 5—20 lb. of copper. In addition, the alloy may contain 2—20 lb. of manganese and/or silicon and 0.2-20% of vanadium, molybdenum, or tungsten.

A. R. POWELL.

Prevention of corrosion of articles made of aluminium and aluminium alloys. B. JIROTKA (B.P. 305,149, 28.10.27).—The articles are immersed in nitric acid of a concentration greater than 25% whereby a protective oxide film is formed. Coloured films are produced by addition of salts of heavy metals, *e.g.*, chromium, cobalt, copper, to the nitric acid bath, and the duration of pickling may be shortened by connecting the article to a rod or plate of graphite or of a heavy metal so that electrolytic action ensues. To obtain a rough surface for subsequent varnishing hydrochloric acid is added to the pickling bath. A. R. POWELL.

**Production of coatings on aluminium and its alloys.** B. JIROTKA (B.P. 286,729, 9.3.28).—To the baths containing zinc-cyanogen compounds, or a mixture of zinc salts and alkali cyanides, or zinc compounds and alkali carbonates, an alkali borate is added; *e.g.*, the bath may consist of a mixture of zinc carbonate, alkali cyanide, and borax. The hot solutions may be sprayed or painted on to the metal. M. E. NOTTAGE.

Decomposition of chromium ore and manufacture of chromium compounds free from iron. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 305,712, 10.11.27).—The ore is opened up either by quenching it, or by melting it with an alkali hydroxide or carbonate in a non-oxidising atmosphere. It is then treated with hydrogen chloride and/or chlorine at a temperature not above 800°, preferably 500—650°, in the presence or absence of a reducing agent.

M. E. NOTTAGE. Recovery of tin contained in the residues of tin plate manufacture. Les PETITS FILS DE F. DE WENDEL & CIE. (B.P. 304,639, 15.3.28. Fr., 23.1.28).— The residues are agitated in a perforated rotating trommel immersed in a hot solution containing 2% of sodium hydroxide and 2% of sodium carbonate. The oily material separates as a scum on the surface of the liquid, and the earthy impurities and oxides fall through the perforations in the trommel, leaving a clean metallic residue from which a high-quality tin is recovered by reverberatory liquation. A. R. POWELL.

Metallurgical process [for nickel]. J. H. WHITE, Assr. to Bell Telephone Labs., INC. (U.S.P. 1,700,460, 29.1.29. Appl., 15.12.26).—Small amounts of vanadium and magnesium are added to molten nickel to enable it to be forged. H. ROYAL-DAWSON.

Silicon-containing magnesium alloys for use with pistons for internal-combustion engines. I. G. FARBENIND. A.-G. (B.P. 293,359, 4.7.28. Ger., 4.7.27).—The alloys contain up to 15% Al, 1—6% Si, and copper, zinc, or cadmium, either alone or together, in such amounts that the m.p. does not fall below 400°. A. R. POWELL.

Tantalum-alloy pen. E. W. ENGLE and M. M. AUSTIN, ASSTS. to FANSTEEL PRODUCTS CO., INC. (U.S.P. 1,701,299, 5.2.29. Appl., 27.5.27).—An alloy containing 90% Ta and 10% W is used for the nib.

H. ROYAL-DAWSON.

Decomposition of ores of zirconium and other rare-earth metals, and of titanium. DEUTS. GASGLÜH-LICHT AUER-GES.M.B.H. (B.P. 291,004, 26.4.28. Ger., 23.5.27).-The ore is treated with sulphuric acid and the earths are separated as sulphates or basic sulphates in the usual way; the mother-liquor is evaporated and the residue heated to drive off excess sulphuric acid and to decompose any sulphates, e.g., ferric sulphate. The mixed gases from this operation and from the calcining of the sulphates of the earths are mixed with air and passed over a fresh charge of ore to effect partial sulphatising of the earths. The product is then digested as usual with sulphuric acid. The process effects considerable economies in the amount of acid required to decompose the ores. A. R. POWELL.

Vessels having an acid-proof lining. I. G. FARBENIND. A.-G. (B.P. 290,189, 25.1.28. Ger., 9.5.27. Addn. to B.P. 283,964; B., 1929, 100).—The lining consists of silicon alloy slabs containing not less than 50% Si. Alloys of silicon with chromium, copper, manganese, or tungsten are suitable.

H. ROYAL-DAWSON.

Treatment of minerals. McK. STOCKTON, Assr. to CELITE Co. (Re-issue 17,212, 12.2.29, of U.S.P. 1,611,791, 21.12.26).—See B., 1927, 114.

Processing sheets to be used in can making. E. W. BLISS Co., Assees. of P. KRUSE (B.P. 289,052, 12.4.28. U.S., 22.4.27).

Disintegration of [low-grade, ore-bearing] clays and similar materials. J. F. NEWSOM (B.P. 302,420, 24.9.27).

Separation of materials (B.P. 275,970).—See I. Refining of aluminium (B.P. 293,353). Electrodeposition of chromium (B.P. 301,478). Metal foils and sheets by electroplating (U.S.P. 1,698,472 and 1,698,486).—See XI.

# XI.—ELECTROTECHNICS.

Improved form of electric resistance furnace. W. ROSENHAIN and W. E. PRYTHERCH (Inst. Metals, March, 1929, Advance copy. 5 pp).—A resistance furnace developed primarily for laboratory use at temperatures above those obtained by means of wirewound furnaces is heated by mean of carbon resistor elements. These elements consist of pellets or short rods of carbon or graphite loosely fitting into a sheath of refractory material. High temperatures are obtained by the passage of a relatively small current through the contact surfaces between successive rods or pellets, and the use of solid end-pieces provides the resistor with comparatively cool ends without the necessity of water-cooling. Oxidation of the carbon is very slow even when working at 1300° or 1400°. A small muffle and an experimental steel-melting furnace have been heated by means of a number of sheathed resistors, and the type evolved may be suitable for more extended use. C. A. KING.

Recent developments in electric furnaces. D. F. CAMPBELL (Inst. Metals, March, 1929. Advance copy. 20 pp.).-In melting practice, as applied largely to brass works, the development of the Ajax-Northrup and Ajax-Wyatt induction furnaces has eliminated the use of expensive crucibles. In the modern furnace the lining is formed by filling the space between a metal liner and the induction coil with a suitable refractory composition, and fritting the material by means of heat induced in the liner, which is later melted out in the first charge. Ajax-Wyatt furnaces of 600 or 1200 lb. capacity are now preferred for intermittent working rather than the heavier types, and the type having a vertical secondary channel is in most common use. Mention is made of furnaces for heat-treatment and also of a double-track, tunnel pottery kiln heated by nickelchromium spiral resistors inserted into grooved bricks.

C. A. KING.

Theoretical considerations in electric tunnel kiln design. J. KELLEHER (Amer. Electrochem. Soc., May, 1929. Advance copy. 6 pp.).—A method is described for designing an electric tunnel kiln for heattreating materials according to a specified temperaturetime cycle. H. J. T. ELLINGHAM.

Control of the current density in electrolytic baths. W. PFANHAUSER (Chem.-Ztg., 1929, 53, 129– 130).—An instrument for measuring the current density in electrolytic baths comprises two circular metal electrodes each 0.5 dm.<sup>2</sup> in area set into a hard rubber case so that they are parallel to but insulated from one another. The plates are connected, by means of wires inside a tube attached to the rubber case, to a measuring instrument which records directly the current density when the plates are immersed between the anode and the cathode. A second scale shows the time required to obtain a definite thickness of deposit, using the current density recorded on the first scale.

A. R. POWELL.

Electrical resistance of liquid insulating materials. A. GYEMANT (Physikal. Z., 1929, 30, 33-58).— A critical review of the literature relating to the breakdown of liquid dielectrics. The relations between chemical constitution and physical structure and the breakdown field strength of organic liquids are considered, together with the rôle of electrical conditions, temperature, and pressure. R. A. MORTON.

Dissolution of plain and amalgamated zincs in electric batteries. J. N. FRIEND (Inst. Metals, March, 1929. Advance copy. 4 pp.).—The results of comparative treatments of plain, high-grade  $(99 \cdot 9\%)$  zinc and ordinary commercial (98-99%) zinc in 10% sulphuric acid, and in solutions of ammonium chloride in ordinary use in Leclanché cells, showed that the use of pure zinc was not warranted. In the dilute acid amalgamated pure zinc may be less resistant to attack than ordinary amalgamated rod, though little difference was noted in ammonium chloride solutions. Plain highgrade zinc is not recommended in place of amalgamated commercial metal. C. A. KING.

Apparatus for testing liability to corrosion. V. DUFFEK (Gas- u. Wasserfach, 1929, 72, 127-129) .---The material to be tested is immersed, together with a mercury electrode, in a vessel containing the electrolyte (e.q., water) the action of which is to be examined. External connexion is made through a milliammeter and a resistance, the latter being sufficiently great that the normal corrosive action of the electrolyte on the material is not affected by the current passing. The whole is placed inside a closed vessel, and provision is made for passing a stream of oxygen through the electrolyte during the test. If no corrosion takes place the current passing between the material and the mercury electrode gradually falls, and a sudden increase in the latter indicates the beginning of corrosive action. Working with 60 mm. pressure (water-gauge) of oxygen in the apparatus, the velocity of corrosion is about 120 times that under normal atmospheric conditions. The apparatus may be used for determining the liability of metals and alloys to corrosion by liquids, in the presence of air or other gases, at various pressures, or the efficiency of various protective coverings on metals W. T. K. BRAUNHOLTZ. etc.

See also A., Mar., 240, Deterioration of quartz mercury-vapour lamps (GILLAM and MORTON). 274, Preparation of hydroxylamine (STSCHERBAKOV and LIBINA). 275, Preparation of sodium perborate (GIORDANI and INTONTI). Electrolytic precipitation of metals (ARNDT). Theory of electrodeposition of chromium (MÜLLER and EKWALL).

Removal of tar fog from gases. FISCHER. Analysis of gasoline. Aborn and Brown.—See II. Colour of light sources. CUNLIFFE and LANIGAN.—See VI. Nickel-plating. BALLAY.—See X.

## PATENTS.

Electric induction furnace. E. F. NORTHRUP (B.P. 305,620, 8.9.27. Cf. B.P. 201,954; B., 1929, 101).— Current is supplied to the furnace coil in a number of phases by a polyphase transformer in which the number of secondary phases is a multiple of the number of primary phases, whereby greater dissipation of energy in the charge is attained and the power factor improved.

J. S. G. THOMAS.

Induction furnace. E. F. Russ (B.P. 306,172, 15.11.27).—Part of the secondary circuit is used as a heating element, the secondary circuit comprising, in series, substantially smooth, corrugated, or ribbed surfaces of high resistance forming a substantially rectilinear muffle or trough, and a secondary low-resistance member surrounding the primary and heating a preheating chamber. J. S. G. THOMAS.

[Electrodes for] alkaline accumulators. J. BACSA (B.P. 305,680, 8.8.27 and 12.4.28).—Electrodes

are prepared by the repeated anodic oxidation of nickel, cobalt, or iron electrodes in alkaline solution and cathodic deposition of the metal from neutral solution and simultaneous conversion from the higher into the lower state of oxidation. Alternatively, cadmium repeatedly deposited from a neutral cadmium salt solution upon a cadmium or zinc electrode is subjected to anodic oxidation and subsequent cathodic reduction in an alkaline electrolyte. J. S. G. THOMAS.

Calomel electrode vessel. D. S. HAKE, ASST. to ACHESON GRAPHITE CO. (U.S.P. 1,700,796, 5.2.29. Appl., 2.11.26).—The cell has an inlet tube near its upper end for the introduction of potassium chloride solution, and a contact tube extending from the cell at a point below the inlet the end of which extends into the contact tube. H. ROYAL-DAWSON.

**Production of cathodes for electric discharge tubes.** VEREIN. GLÜHLAMPEN U. ELECTRICITÁTS A.-G. (B.P. 289,763, 18.11.27. Austr., 30.4.27).—Vapours of alkaline-earth metals liberated by a reduction process started by heating, *e.g.*, by reduction of barium hydroxide with magnesium or calcium, are deposited upon a core of platinum, copper, nickel, or chrome-nickel.

J. S. G. THOMAS.

Manufacture of thermionic cathodes for electric discharge devices. M. O. VALVE Co., LTD., A. L. REIMANN, and J. W. RYDE (B.P. 306,281, 20.2.28).— A metallic core, e.g., of platinum-rhodium alloy, is coated with a mixture of alkaline-earth oxides and metallic thorium. J. S. G. THOMAS.

Manufacture of liquid or solid products by gaseous reaction under the influence of silent electrical discharge. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 304,623, 20.10.27).—The cooling fluid of the discharge chamber is controlled so that the temperature rises above the saturation point of the gaseous mixture, whereby deposition of reaction products upon the walls of the chamber is prevented or reduced. If desired, a liquid not taking part in the reaction is caused to flow over the chamber walls in order to dissolve or wash away reaction products. [Stat. ref.] J. S. G. THOMAS.

Glow-lamp filaments. Soc. FRANÇ. DES LAMPES À INCANDESCENCE "LUXOR" (B.P. 288,557, 11.4.28. Fr., 11.4.27).—A compound filament is made of two layers of the same metal having different resistances and bent preferably into U-shape. J. S. G. THOMAS.

Electrolytic apparatus and method of operation. E. THOMSON, ASST. to GEN. ELECTRIC CO. (U.S.P. 1,701,346, 5.2.29. Appl., 25.3.26).—Annular electrolytic cells subdivided by permeable partitions are arranged in stacked relation at right angles to the axis of rotation of a receptacle having a hollow shaft which is used to collect separately the gases evolved from the positive and negative parts of the cells. J. S. G. THOMAS.

Electrolytic apparatus for refining aluminium etc. Soc. ANON. COMP. DE PROD. CHIM. & ELECTRO-MÉTALLURGIQUES ALAIS, FROGES, & CAMARGUE, Assees. of P. L. HULIN (B.P. 293,353, 19.6.28. Fr., 4.7.27).— In electrolytic apparatus in which the cathode metal is removed periodically while the anode is reconstituted suitably, either by admixture of impure light metal or by partial or complete replacement of the mass, there are three superposed fused layers, viz., an anode of a dense alloy of the metal to be extracted, a halogenated bath forming the electrolyte, and a light metal forming the floating cathode. The inner lining comprises a horizontal, refractory frame containing the anode, a horizontal insulating frame containing electrolyte which solidifies on a frame through which water is circulated, and a frame composed substantially of carbon surrounding the cathode and insulated from the metal shell.

J. S. G. THOMAS.

Electrolytic deposition of chromium. LANGBEIN-PFANHAUSER-WERKE A.-G. (B.P. 301,478, 2.1.28. Ger., 1.12.27).-Chromium is electrolytically deposited from neutral or slightly acid solutions of chromium compounds, e.g., chromium acetate, sulphate, chloride, etc., to which is added sodium citrate, Rochelle salt, or other substance, e.g., oxalic acid, phenolsulphonic acid, alcohol, glycerin, sugar, etc., for retaining chromium oxide in solution. The  $p_{\rm H}$  value of the electrolyte is maintained between 4 and 6, and, if desired, colloids or other substances having a capillary action, e.g., derivatives of aromatic hydrocarbons containing hydroxyl groups, which prevent the growth of crystals, may be added to the bath when bright, polished deposits are required. The current density employed is greater than, but of the order of, 0.25 amp./dm.<sup>2</sup> Bags containing chromium carbonate may be suspended in the catholyte, which is separated from, and has a higher concentration than, the anolyte. J. S. G. THOMAS.

Making metal foils. J. R. CAIN, ASST. to RICHARD-SON CO. (U.S.P. 1,698,472, 8.1.29. Appl., 3.5.26).--A film of metal powder deposited and floating on the surface of an electrolyte is maintained in contact with a spongy electrode, and an electric current is passed between this electrode and an anode. J. S. G. THOMAS.

Making metallic sheets by electroplating. G. YUNGBLUT, ASST. to RICHARDSON CO. (U.S.P. 1,698,486, 8.1.29. Appl., 19.9.27).—A cathode shaped like the desired article but of a different metal is plated on one face with the required metal. The cathode metal is then stripped from the plated body in an electrolytic cell in which a similar cathode is used and upon which the stripped metal is plated. J. S. G. THOMAS.

Apparatus for the determination of the amount of carbonic acid in flue gases. H. GRÜSS, ASST. to SIEMENS & HALSKE A.-G. (U.S.P. 1,701,181, 5.2.29. Appl., 11.10.27. Ger., 25.10.26).—In an apparatus of the catharometer type, a double coil is inserted in the diagonal branch of the Wheatstone bridge, and the current supply to the bridge is such that the testing wire surrounded by flue gases and the wire surrounded by the comparison gas are at the same temperature between 200° and 350°. J. S. G. THOMAS.

Manufacture of electrical condensers and materials therefor. E. W. JODREY (B.P. 305,515, 3.11.27 and 1.2.28).—Paper coated with an adhesive, continuous, conducting layer of copper or other metal foil is coated with cellulose esters, *e.g.*, cellulose acetate or collodion. J. S. G. THOMAS. Manufacture of moulded articles such as switch bases, fuse holders, etc. J. A. CRABTREE and J. R. DOLPHON (B.P. 306,249, 12.1.28).—Shields of refractory and insulating material, e.g., asbestos, are caused to adhere (a) by heat, pressure, shrinkage, etc. to parts of the moulding liable to become hot due to arcing etc., or (b) by means of a cement after treatment with chemicals, e.g., dilute hydrochloric acid, for extraction of organic and/or inorganic constituents. J. S. G. THOMAS.

[Cathode for] electric discharge tube. G. HOLST and E. OOSTERHUIS, ASSIS. to N. V. PHILIPS' GLOEILAMP-ENFABR. (U.S.P. 1,701,849, 12.2.29. Appl., 28.7.23. Fr., 21.2.23).—See B.P. 211,825; B., 1924, 551.

Electric discharge device. BRIT. THOMSON-HOUS-TON CO., LTD., Assees. of A. W. HULL (B.P. 282,766, 23.12.27. U.S., 23.12.26).

Filament for incandescence lamps etc. F. SKAUPY, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,701,342, 5.2.29. Appl., 20.4.25. Ger., 3.5.24).—See B.P. 233,294 ; B., 1925, 952.

[Coir-bound electrodes for] electric secondary batteries or accumulators. H. F. JOEL (B.P. 305,738, 21.9.28).

[Holding plates in] electric storage batteries. R. BOSCH A.-G. (B.P. 283,490, 3.8.27. Ger., 11.1.27).

Conveyors for baths for treatment of objects, more particularly electrolytic baths. SIEMENS & HALSKE A.-G. (B.P. 298,184, 17.2.28. Ger., 5.10.27).

High vacua (B.P. 280,560).—See I. Transformation of gaseous hydrocarbons (B.P. 304,914). Lubricating and insulating oils (B.P. 305,553).—See II. Insulated wire (B.P. 305,084).—See XIII.

# XII.—FATS; OILS; WAXES.

Density of lard. W. HALDEN and R. KUNZE (Chem. Umschau, 1929, 36, 61-62). The recorded observations of the density of lard were collected and corrected by calculation to the values for  $d_{1s}^{1s}$  for comparison: according to the large majority of observations this value falls between the limits 0.915-0.923 (never exceeding 0.93), and this range should be substituted for the higher values current in many text-books.

## E. LEWKOWITSCH.

Analysis of fatty substances in Wood's light. A. G. NASINI and P. DE CORI (Annali Chim. Appl., 1929, 19, 46-54).—Addition of natural pigments soluble in oils (carotinoids, chlorophyll) may impart to refined olive oil the optical properties in Wood's light characteristic of virgin olive oil. Results are given of a comparison of the fluorescence and absorption spectra of solutions of chlorophyll in refined olive oil and other natural oils. The fluorescence of various cholesterols is due to the presence of impurities. T. H. POPE.

[Catalytic] hydrogenation of highly unsaturated acids. II. Course of hydrogenation of methyl esters of highly unsaturated acids with a platinum catalyst. Y. TOYAMA and T. TSUCHIVA (Chem. Umschau, 1929, 36, 49-52; cf. B., 1926, 286).—The methyl esters of the highly unsaturated acids from Japanese sardine oil were hydrogenated in alcoholic solution in the presence of platinum-black at ordinary pressure ; by this method the formation of solid acids proceeds from the beginning of the reaction. The amount of acids yielding insoluble bromides decreases steadily during hydrogenation, and it is shown that practically no acids of the series  $C_nH_{2n-6}O_2$  and  $C_nH_{2n-5}O_2$  yielding etherinsoluble bromides are formed; on the other hand, acids of the series  $C_nH_{2n-6}O_2$ , and possibly of  $C_nH_{2n-4}O_2$ , yielding bromides insoluble in light petroleum, are produced in appreciable quantities. Solid acids of the series  $C_nH_{2n-2}O_2$  are formed, having m.p. notably higher than those of the naturally occurring acids of this series ; solid acids of the series  $C_nH_{2n-4}O_2$  with 20 and 22 carbon atoms are also formed.

E. LEWKOWITSCH.

Polymerisation of highly unsaturated [fatty] acids. I. Y. TOYAMA and T. TSUCHIYA (Chem. Umschau, 1929, 36, 45-49) .- The highly unsaturated fatty acids (iodine value 351, ether-insoluble bromides 129%), isolated from Japanese sardine oil by the sodium salt-acetone method and fractionation of the methyl esters, were polymerised by heating at 240-320° in an atmosphere of carbon dioxide; the products were examined at various stages of the reaction. As heating proceeds the iodine value falls, the sp. gr. and refractive index rising approximately proportionally; the specific viscosity, however, rises more rapidly, the curve being almost vertical when the iodine value has dropped to 250. The acid value and saponif. value decrease, whilst the ester value increases. The mol. wt. of the fatty acids (camphor f.p. method) rises to about double its original value. The actual degree of temperature to which the oil is raised produces greater effect than prolonged heating. By extraction of the syrupy polymerisation products with light petroleum, insoluble resinous substances (m.p. 69-70°) were obtained. Since the yield of ether-insoluble bromides decreases more rapidly (none, when iodine value is 198) than the mol. wt. increases, it is concluded that, in the first stages of polymerisation, intramolecular change-probably ring-formation-preponderates over extramolecular polymerisation. E. LEWKOWITSCH.

Fats. XIII. Partial addition of halogen to polyunsaturated fatty acids. Glyceryl β-elæostearate and wood [tung] oil. H. P. KAUFMANN [with C. LUTENBERG] (Ber., 1929, 62, 392-401; cf. B., 1926, 447, 758).-Glyceryl β-elæostearate when treated with a 100% excess of bromine in carbon tetrachloride in the absence of light absorbs 2 mols. of halogen in the interval 3-8 hrs., after which addition commences at the third double linking. Determination of the corresponding iodine value in tung oil is effected by mixing 0.1-0.15 g. of the sample in 10 c.c. of pure carbon tetrachloride with 30-40 c.c. of 0.1N-bromine in the same solvent, preservation of the mixture in the dark for 4-5 hrs., addition of aqueous potassium iodide, and titration of the liberated iodine. Subsequent addition of potassium iodate to the solution causes only very slight liberation of iodine, so that substitution has not occurred to an appreciable extent. For the determination of a single unsaturated linking, glyceryl β-elæostearate or tung oil (0.1 - 0.12 g.) is dissolved in a mixture of equal volumes of chloroform and carbon tetrachloride (distilled over phosphoric oxide). A solution (20 c.c.) of bromine and iodine is added, prepared by addition of the requisite weight of iodine to a  $0 \cdot 1N$ -solution of bromine in methyl alcohol containing sodium bromide. After 3—4 hrs. the mixture is treated with 5% aqueous potassium iodide and the liberated iodine is titrated. The "partial iodine value" thus determined coincides with or differs by 1—2 units from the thiocyanogen value. H. WREN.

Catalytic polymerisation of fatty oils. J. MAR-CUSSON (Chem. Umschau, 1929, 36, 53-54).-Anhydrous tin tetrachloride was used as catalyst (cf. Staudinger and Bruson, B., 1926, 719) for the polymerisation of tung, linseed, and fish-liver oils. Solid polymerised products, insoluble in acetone, were obtained from tung, linseed, and cod-liver oils yielding thick oils from which the fatty acids, recovered by saponification, had mol. wts. of 590, 494, and 330, respectively. The polymerised fatty acids from tung oil could be separated into (1) solid resinous acids (acid value 186, saponif. value 199.5, iodine value 81) yielding sodium salts which were soluble in water but insoluble in alcohol; and (2) pale, unimolecular, semi-liquid acids soluble in light petroleum and forming sodium salts soluble in alcohol. The solid acids of (1) on being heated at 105° or on exposure to air became partially insoluble in organic solvents due to anhydride formation.

E. LEWKOWITSCH.

Negative autoxidation catalysts for fatty oils. V. RUTSCHKIN (Oil Fat Ind. Russia, 1928, No. 6, 27–30: Chem. Zentr., 1928, ii, 1504).—Heating and spoilage of moist oil seeds is prevented by 0.01% of  $\beta$ -naphthol, but not phenol. Addition of phenol, resorcinol, or  $\beta$ naphthol (0.01%) to moistened seeds retards their growth. A. A. ELDRIDGE.

Determination of the oil content of seeds without heating. N. SPASSKI (Oil Fat Ind. Russia, 1928, No. 6, 19; Chem. Zentr., 1928, ii, 1506).—By means of the author's apparatus an accuracy of 0.1% is attained. A. A. ELDRIDGE.

Dependence of chemical composition of oils on climate. J. DAVIDSOHN (Chem. Umschau, 1929, 36, 64-67).--Ivanov's work ("Climate of the World and the Chemical Functions of Plants") is reviewed. A notably higher proportion of linolenic and linoleic acids is found in the oils from seeds (linseed, sunflower seed, etc.) that have ripened in cold climates (northern latitudes or high altitudes), which give oils of high iodine value, than in the oils from the same seed grown in hotter climates, which favour the production of oleic acid. The decrease in linolenic acid content may also be correlated with the increasing intensity of the light in low latitudes. (Cf. Eibner and Brosel, B., 1928, 761.) E. LEWKOWITSCH.

See also A., Mar., 245, Higher fatty acids (MORGAN and HOLMES). 293 and 294, Determination of higher unsaturated fatty acids (ESCHER). 294, Distillation of palmitic acid (SHOYAMA). Conversion of higher fatty acids into barium salts (ESCHER). Distillation of oleic acid (OGATA). Acids of high mol. wt. from arachis oil (HOLDE and others). 312, Cholesterol (MONTIGNIE). Colour reaction for ergosterol and its transformation products (Häussler and Brauch-

LI). 337, Micro-detection of glycerol (ALBER). 359, Vitamin content of irradiated olive oil (RIGOBELLO). Colour reaction for ergosterol (ROSENHEIM).

Lipolysis of worked butter. GRATZ.-See XIX.

## PATENTS.

Production and employment of cleansing, emulsifying, and wetting agents. CHEM. FABR. MILCH A.-G. (B.P. 275,267, 2.8.27. Ger., 2.8.26).— Neutral fats, fatty acids, naphthenic acids, etc. (e.g., technical oleic acid) are sulphonated with excess of chlorosulphonic acid especially in presence of aromatic hydrocarbons (e.g., benzene) to yield products with soaplike properties. C. HOLLINS.

Treatment of tall oil. OEL-U. FETT-CHEMIE G.M.B.H., Assees. of A. SCHULTZE & CO. OELFABR. (B.P. 278,697, 26.9.27. Ger., 11.10.26).—Tall oil is subjected to esterification (e.g., by refluxing with alcohol and sulphuric acid) to such an extent that the fatty acids alone are esterified. The mixture is then treated with soda lye to neutralise the excess of mineral acid and the unattacked resinic acids: the esters are extracted with light petroleum and the fatty acids recovered by hydrolysis.

E. LEWKOWITSCH.

Oil from hops (B.P. 305,066).—See XVIII.

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

Detection of adulteration in oil of turpentine. M. H. BARRAUD (Ann. Falsif., 1929, 21, 5-18).-Oil of turpentine in France should fulfil the official definition adopted by the 4th Congress of Industrial Chemistry at Bordeaux, 1924. 200 c.c. of the sample are first fractionally distilled, and four fractions of 40 c.c. collected, the fifth consisting of the residue in the flask and a few drops from the end of the condenser. Values for d,  $[\alpha]$ ,  $n_{\rm D}^{25}$ , and acidity of the whole sample and of each of the five fractions are then determined. In the first fraction any non-terpene adulterant will be present (usually white spirit) and the normal adulterants (rosin oil and rosin) will be in the last. From variations in the differences between each of these values for the various fractions adulteration may be detected. Standard values for the turpentine oils and of an oil adulterated with white spirit are tabulated. 2% of white spirit may thus be detected. If oil of turpentine is mixed with glycerin the Christianson effect results, and the temperature of the colour change from indigo-blue to violet indicates the point where the refractive indices of turpentine and glycerin are the same in yellow light. 1-2 c.c. of glycerin are added to 10 c.c. of turpentine oil in a test tube in which a thermometer is fixed. The temperature of the change is lowered for a very pure oil and rises with the proportion of impurities in the last fraction, particularly with products of oxidation. It is necessary always to compare oils from the same type of pine and to standardise the temperature change with a sample of known purity. 50 samples of standard maritime pine oil gave the value 23-26°. D. G. HEWER.

Adhesives and paint vehicles. E. STERN (Farben-Ztg., 1929, 34, 1312-1313).—The nomenclature adopted by Wagner and Kesselring (B., 1929, 218) in their communication on the subject of casein emulsions is discussed; the author restates his conception of the staticcolloid paint system and the dynamic-colloid processes of the paint film. S. S. WOOLF.

[Adhesives and paint vehicles.] H. WAGNER (Farben-Ztg., 1929, 34, 1313).—A reply to Stern (cf. preceding abstract). S. S. WQOLF.

Paint testing. H. WOLFF, W. TOELDTE, and G. ZEID-LER (Farben-Ztg., 1929, 34, 1179—1181).—Paint testing should not be modified in the direction of increasing the accuracy of existing methods to a point where undue significance is given to quantitative differences that are not justified in practical tests, subject at the best to accidental influences. Records of paint failures should take the form of well-marked stages in the deterioration, recognisable with certainty. A sand-abrasion test and a test wherein the electrical conductivity of the whole surface of a paint film is examined are detailed. Five "degrees of failure" are indicated for each test. S. S. WOOLF.

Temperature-humidity control cabinet for testing paint, varnish, and lacquer films. G. G. Sward (Amer. Paint & Varnish Manufrs.' Assoc., Feb., 1929, Circ. No. 345, 91—98).—Drying, hardness, elasticity, etc. tests on films are carried out in the laboratory of the above Association in a cabinet fitted with devices for controlling temperature and humidity, details of which are given. S. S. WOOLF.

Influence of titanium white on the fastness to light of coal-tar dyes. E. KEIDEL (Farben-Ztg., 1929, 34, 1242—1243).—Exposure tests indicate that coal-tar dyes bleach much more rapidly when mixed with titanium white than with zinc oxide, lithopone, or white lead. The results are very definite in water-paint vehicles, but similar tendencies are indicated in oil media. S. S. Woolf.

Titanium white catalysis ? H. WAGNER (Farben-Ztg., 1929, 34, 1243—1244; cf. preceding abstract).— The abnormal bleaching of coal-tar dyes in the presence of titanium white (which may be attributable to autoxidation and to the sulphuric acid content of titanium white) may be remedied by the addition of zinc oxide, but this in turn causes a slight tendency towards darkening. The preparation of a "neutral pigment" is proposed. S. S. WOOLF.

Rapid drying finishes for wood surfaces. F. W. HOPKINS (Amer. Paint & Varnish Manufrs.' Assoc., Feb., 1929, Circ. No. 343, 61—64).—A brief survey of the scope of lacquers in the wood-finishing field. Types of failure of nitrocellulose lacquers are discussed, and the desirable qualities of a new type of material are indicated. S. S. WOOLF.

Systematic formulation of nitrocellulose lacquers. S. DE LANCE (Farben-Ztg., 1929, 34, 1245— 1246).—The work of Hofmann and Reid (B., 1928, 648) is criticised. The extension of the method to threedimensional graphs (*i.e.*, triangular prism co-ordinates) is considered to afford much more significant information. Thus flexibility may be correlated with proportion of nitrocellulose, resin, and plasticiser in lacquers, or compatibility with quantity of ester, alcohol, and hydrocarbon in the solvent mixture. S. S. WOOLF.

Use of ultra-violet radiations in distinguishing between amber and its imitations. G. KOSTKA (Chem.-Ztg., 1929, 53, 117-118, 138-139).-Under the influence of ultra-violet radiation (440-280 µµ) amber shows a yellowish-green to bluish-white fluorescence. Pressed amber (ambroid) behaves similarly, the fluorescence being rather less pronounced. The fluorescence of clouded amber differs somewhat from that of the clear variety. The difference is mainly one of degree only, and is due to structural differences and not to the presence of impurities. Phenol-formaldehyde condensation products absorb ultra-violet radiation, but are not excited to fluorescence. Urea-formaldehyde condensation products show a faint fluorescence, easily distinguishable from that of genuine amber. Urea products are transparent to ultra-violet radiation. The differing transparencies towards the radiation permit of a ready distinction between urea and phenol condensation products. The easily-recognisable casein and cellulose derivatives fluoresce under the influence of J. S. CARTER. ultra-violet radiation.

Polyhydric alcohol-polybasic acid reaction. I. Glycerol-phthalic anhydride. R. H. KIENLE and A. G. HOVEY (J. Amer. Chem. Soc., 1929, 51, 509-519). -The isothermal resinification of a mixture of glycerol (2 mols.) and phthalic anhydride (3 mols.) at 195-230° involves esterification only, since water alone is evolved and the saponification value of the mixture remains nearly constant. The reaction proceeds rapidly at first with heat evolution and a corresponding rapid decrease in acid value and increase in ester value of the product, followed by a more gradual change until gelation takes place. The flow-point, colour, and consistency of the product show similar changes. The density increases, but there is little change in the refrac tive index. Esterification is about 50% complete during the first minute, 75-79% at gelation, and 99% on prolonged heating. The time of gelation is inversely proportional to the absolute temperature. There is no indication that the reaction proceeds in stages.

H. E. F. NOTTON.

# PATENTS.

Nitrocellulose solutions. W. J. JENKINS and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 305,148, 26,8.27).—The flash point of nitrocellulose solutions containing petrol or benzene hydrocarbons, *e.g.*, clear lacquers or enamels, may be raised by addition of not more than 8% by wt. of carbon tetrachloride.

F. R. ENNOS.

Cellulose ester or ether compositions. E. I. DU PONT DE NEMOURS & Co. (B.P. 279,520, 25.10.27. U.S., 25.10.26).—Tetrahydrofurfuryl alcohol is claimed as a desirable ingredient of brushing lacquers.

S. S. WOOLF.

Coating of articles [insulated wire]. W. E. BEATTY. From BELL TELEPHONE LABS., INC. (B.P. 305,084, 28.10.27).—A liquid insulating coating is applied to wire etc. and hardened by heat solely obtained from the combustion of its own volatile constituents. The liquid coating material is contained in a suitable sealed chamber through which the wire may be drawn continuously at high speed. Suitable apparatus is described. S. S. WOOLF.

IROHA.

Manufacture of coating compositions [chlorinated rubber varnishes]. C. ELLIS, ASST. to CHADE-LOID CHEM. Co. (U.S.P. 1,695,638-1,695,642, 18.12.28. Appl., [A] 29.8.21, [B] 5.5.23, [C-E] 22.3.24. Renewed, [B] 30.4.25, [E] 10.9.27).-(A) A varnish consists of chlorinated rubber, a volatile solvent or thinner, and a drying oil, e.g., tung oil. (B) A straw-hat varnish consists of chlorinated rubber (over 66% Cl) and a colouring matter dissolved in a volatile solvent. (c) In order to give a dull finish, the varnish contains less drying oil, e.g., linseed oil, than chlorinated rubber. (D) A solution of chlorinated rubber (67% Cl or more) is stabilised by the addition of a soluble organic aminocompound. (E) A varnish reviver consists of chlorinated gutta-percha, an aromatic solvent therefor, and a resinous homogenising agent; a phenolic phosphate may also be present. F. G. CLARKE.

Cement [from chlorinated rubber]. C. ELLIS, Assr. to CHADELOID CHEM. Co. (U.S.P. 1,695,637, 18.12.28. Appl., 22.3.24).—An adhesive consists of a solution of chlorinated rubber. F. G. CLARKE.

**Phenol resin and its manufacture.** BAKELITE CORP. (B.P. 280,521, 27.10.27. U.S., 11.11.26).—A phenol is condensed with anhydroformaldehydeaniline (or a homologue) and aniline excess is removed. According to the temperature employed a reactive or non-reactive (*i.e.*, non-hardening) resin is obtained; the latter may be made reactive by addition of hexamethylenetetramine, or this reagent may be added initially.

#### C. HOLLINS.

Artificial resin compositions. BAKELITE CORP., Assees. of J. H. SCHMIDT (B.P. 275,604, 26.7.27. U.S., 3.8.26).—A high-boiling solvent (b.p. above 170°), e.g., ethyl oxalate, glycol diacetate, etc., is added to a "glyptal" resin that has previously been taken to the stage of incipient gelatinisation in the substantial absence of solvent, and the "curing" is continued in the presence of the incorporated solvent, until gelatinisation once more sets in, when medium-boiling solvent (b.p. 110—160°) may be added. S. S. WOOLF.

Production of a viscous condensation product. SCHIEFERWERKE AUSDAUER A.-G. (B.P. 286,731, 9.3.28. Ger., 10.3.27).—A mixture of phenol, *p*-dichlorobenzene, and formaldehyde is heated to boiling in the presence as catalyst of a salt mixture composed of sodium chloride, ammonium chloride, and hexamethylenetetramine, the water formed is removed, and volatile vapours are expelled from the product, which is then washed with water. The light-coloured syrup thus obtained may be readily hardened at 80°. S. S. WOOLF,

Production of highly glazed surfaces. R. ARNOT, Assee. of ELEKTROCHEM. FABR. KEMPEN-RHEIN, DR. BRANDENBURG & WEYLAND G.M.B.H. (B.P. 288,303, 4.4.28. Ger., 6.4.27).—A film of hardened artificial resin of highly polished surface is applied under pressure at normal or elevated temperatures to articles previously covered with glue solution etc. S. S. WOOLF.

Methods of enamelling and apparatus therefor. W. E. BEATTY. From BELL TELEPHONE LABS. INC. (B.P. 305,611, 8.11.27). Green hydrated chromium oxide (B.P. 304,809). —See VII. Resins from hops (B.P. 305,066).—See XVIII.

# XIV.--INDIA-RUBBER; GUTTA-PERCHA.

**Report of the Raw Rubber Specifications Committee.** E. B. SPEAR, C. R. BOGGS, H. E. SIMMONS, H. L. TRUMBULL, and N. A. SHEFARD (Ind. Eng. Chem. [Anal.], 1929, **1**, 55—56).—As the result of tests in five laboratories using a rubber–sulphur mixing and one containing also zinc oxide and di-o-tolylguanidine, the conclusion is drawn that the physical testing of raw rubber is not in a very satisfactory condition, and further investigations are recommended with a view of securing more definite standardisation and greater uniformity.

D. F. Twiss.

Cold vulcanisation [of rubber] (Parkes' process). P. BOURGOIS (Bull. Féd. Ind. Chim. Belg., 1929, 8, 3-8).—The method of vulcanisation of rubber by immersion in a solution of sulphur chloride is reviewed, with especial reference to certain practical details and difficulties. The vulcanising effect is attributed to nascent sulphur formed from the sulphur chloride by the action of natural moisture in the rubber.

D. F. Twiss.

Vulcanisation and structure of rubber. E. LINDMAYER (Kautschuk, 1929, 5, 35—38).—Vulcanisation of rubber is regarded as comprising a primary stage producing soft rubber and a secondary stage leading to vulcanite; these frequently overlap in practice, but with care can be distinguished. With the aid of the "needle structure" for rubber it is possible to explain the mechanism of vulcanisation and to illustrate it by means of three-dimensional formulæ, D. F. TWISS.

The accelerator [of vulcanisation] "Tuads" and its substitutes. F. Löblein (Kautschuk, 1929, 5, 38—43; cf. Esch, B., 1929, 28).—Contrary to the earlier statement, "Tuads" and "Vulkacit Thiuram" (commercial forms of tetramethylthiuram disulphide) are of comparable quality—the latter, indeed, being somewhat purer. Experimental evidence is given. D. F. Twiss.

Production of lustrous spread rubbered material with the aid of vulcanisation by ultra-violet light. R. DITMAR (Gummi-Ztg., 1929, 43, 1172).—The material is spread in the usual way with a benzene solution of a mixture containing crêpe rubber (100 pts.), colloidal sulphur (3.5 pts.), tetramethylthiuram disulphide (2 pts.), sulzin (ZnSO<sub>4</sub>,5NH<sub>3</sub>, 7 pts.), anhydrous sodium thiosulphate (2 pts.), selenium-red (1.8 pts.), and titanium oxide (2 pts.). It is then led directly under a battery of quartz mercury lamps, the exposure being for 5 min. at a distance of 8 cm.; complete vulcanisation results with production of a non-tacky rubbered surface of high lustre. D. F. TWISS.

New physical test for vulcanised rubber. D. D. WRIGHT (Ind. Eng. Chem. [Anal.], 1929, 1, 17-20).— The test-piece is a strip of rubber which has been trisected for a considerable portion of its length; tension is then applied between the uncut end and the end of the central tongue until rupture occurs. This test subjects the sample to a combination of tensile and shearing stresses, the latter predominating. It is frequently a more sensitive index than the ordinary stress-strain test for over-vulcanisation or depreciation by accelerated ageing.

D. F. Twiss.

See also A., Mar., 305, Polystyrene (STAUDINGER and others). 305-6, Highly polymerised compounds (STAUDINGER and others). 321, Isoprene and caout-chouc (STAUDINGER and others).

#### PATENTS.

Vulcanisation of rubber. Production of accelerators for vulcanising rubber. NAUGATUCK CHEM. Co., Assees. of S. M. CADWELL (B.P. 279,815 and 298,537, [A, B] 5.10.27. U.S., 1.11.26).-(A) Vulcanisation is accelerated by the chlorination derivative of a condensation product of an amine and an aldehyde containing an open chain of 2-7 carbon atoms, such as heptaldehyde-aniline. The reaction products with 1-3 chlorine atoms accelerate well; a pentachloro-product is also obtainable which is not an accelerator, but by hydrolysis with aqueous alkali is converted into a very active tetrachloro-compound. Bromine and iodine derivatives can be used similarly. (B) Heptaldehydeaniline and other condensation products and amines with an aldehyde containing an open chain of several carbon atoms are treated with a halogen or a halogen compound such as hydrogen chloride for the production of new vulcanisation accelerators. D. F. Twiss.

Reclamation of rubber. DUNLOP RUBBER CO., LTD., D. F. TWISS, and F. THOMAS (B.P. 305,826, 17.2.28).—Scrap vulcanised rubber is treated with a solution of caustic alkali in a polyhydroxy-compound such as glycerol, *e.g.*, at 190—200°. D. F. TWISS.

Production of rubber articles from natural rubber latex. ANODE RUBBER CO., LTD. (B.P. 283,871, 23.12.27. Ger., 18.1.27).—In order to modify the properties of articles made directly from rubber latex, the latter is previously mixed with an artificial aqueous dispersion of masticated rubber. D. F. Twiss.

Bonding of articles or parts formed respectively of india-rubber or the like and cellulose derivatives. C. MACINTOSH & Co., LTD., S. A. BRAZIER, and G. F. THOMPSON (B.P. 305,745, 1.12.27).—The india-rubber surface is first treated with a mutual solvent for rubber and cellulose derivatives, *e.g., cyclohexanol.* A varnish or lacquer of cellulose acetate or nitrate, into which a mutual solvent may have been introduced, is then applied to the softened surface. An increased strength of adhesion is thus ensured. D. F. Twiss.

Treatment of fabric with rubber solution or the like. C. MACINTOSH & Co., LTD., S. A. BRAZIER, and J. D. CAMPBELL (B.P. 305,264, 4.11.27).—Rubber in solution or aqueous dispersion is applied to fabric, the treated material being transported in festoons through a drying chamber; the material may pass progressively through another application of rubber and a drying process, and finally to a vulcanising chamber.

D. F. Twiss.

Chemical product [chlorinated rubber] and its manufacture. C. ELLIS, ASST. to CHADELOID CHEM. Co. (U.S.P. 1,695,636, 18.12.28. Appl., 17.6.22).—Solid rubber, plasticised with not more than 20% of benzene or other solvent, is chlorinated under pressure at, e.g., 20-30°. R. BRIGHTMAN.

Production of an elastic composition. W. T. GALBRAITH and A. E. MILLS (B.P. 305,249, 29.2.28).-A mixture of paper pulp (50 lb.) or its equivalent of rubber (synthetic, natural, or reclaimed), heated vegetable oil (50 lb.), "synothin" (5 lb.), gluten (20 lb.), water-glass (3 quarts), glue (20 lb.), gum (40 lb.), casein (2 lb.), and asbestos (5 lb.) is immersed for a few seconds in sulphuric acid, diluted with water or paraffin, washed repeatedly with dilute ammonia, digested with alcohol and sal-ammoniac, and, after the addition of 2% of copper oxide, dried by treatment with superheated air for 5 hrs. Any tendency to perish is overcome by treating with sulphur fumes and shellac. The elasticity is increased by incorporating tannic acid, the sheeted material being finally immersed in aniline water. The dried product is elastic and fire-proof.

#### D. F. Twiss.

Manufacture of rubber articles. W. B. WESCOTT, Assr. to RUBBER LATEX RES. CORP. (U.S.P. 1,702,225, 12.2.29. Appl., 1.6.26).—See B.P. 272,187; B., 1928, 101.

Mixing machines for rubber compositions etc. F. H. BANBURY (B.P. 306,371, 1.9.28. U.S., 3.3.28)

Manufacture of articles [rubber tubes] containing material at one stage plastic, and particularly extrusion of the plastic content. DUNLOP RUBBER Co., LTD., and F. FELLOWES (B.P. 305,152, 31.10.27).

[Embossed] rubber goods and their manufacture. BROADHURST & Co., LTD., and W. J. DEXTER (B.P. 306,276, 11.2.28).

Coating compositions (U.S.P. 1,695,637—1,695,642). —See XIII.

# XV.-LEATHER; GLUE.

Removal of chromium salts from leather. F. C. HEMMINGS and M. C. LAMB (J. Soc. Leather Trades' Chem., 1928, 12, 599—602).—The chromium compounds are removed from wet or dry chrome-leather shavings most rapidly by solutions of oxalic acid, which is more effective than ammonium oxalate alone or with acid or ammonia. Dry shavings are stripped of their chromium more readily than wet shavings. The liquor obtained is acid to litmus, but the chromium is present in an anionic form. 15% of oxalic acid on the weight of the dry shavings is necessary to remove the chromium salts, and the stripped product should be washed with dilute ammonia because ammonium chromoxalate is more soluble than chromoxalic acid. Oxalic acid is also useful for " wetting back" dry chrome leather.

# D. WOODROFFE.

Effect of heat on wetted vegetable-tanned leathers. II. W. J. CHATERS (J. Soc. Leather Trades' Chem., 1928, 12, 608—614; cf. B., 1929, 257).—Shrinkage curves have been derived in the author's apparatus for wet-salted calf pelt after treatment with water, acid and alkaline solutions, respectively, and for oak bark-tanned leathers containing different amounts of free mineral acid. The irregularities in the curves are indicative of the physical and chemical conditions in the leathers, and could be used for control purposes in manufacture. D. WOODROFFE.

Determination of the strength of a bate. A. BOIDIN (J. Soc. Leather Trades' Chem., 1928, 12, 614-626).-Experiments have been made on casein substrates using two bacterial bates and two pancreatic bates. Complete precipitation of casein is obtained only at  $p_{\rm H}$  5. The use of buffer salts to dissolve casein diminishes the hydrolytic power of the enzymes. In the absence of buffer salts, the methods of weighing the casein and of determining the soluble nitrogen yield identical results for the casein digested. The casein is sufficiently buffered when dissolved in a small quantity of sodium hydroxide to prevent variations > 0.2 in the  $p_{\rm H}$ value during the digestion. If buffer salts are present, the "soluble nitrogen" method yields more accurate results for the amount of digested casein than are obtained by weighing the unchanged casein, since the latter will absorb salts when precipitated. Pancreatic enzymes and bates are more reactive the nearer the  $p_{\rm H}$  value is to 8, but bacterial products are reactive over a wider range of pH values. The results are not affected by dissolving the casein at 40° or on a boiling water-bath. The soluble nitrogenous matter is a constant for any particular casein, but varies with different products. The digestion of the casein is not proportional to the time of digestion; 1 hr. is the best period at 40°, and 35-40° is the best temperature range. The unchanged casein cannot be precipitated if more than 84% of the total casein originally present has been hydrolysed.

## D. WOODROFFE.

Specifications for standard gelatin. Report of Committee on Standard Gelatin appointed by Division of Leather and Gelatin Chemistry. C. E. DAVIS, S. E. SHEPPARD, and M. BRIEFER (Ind. Eng. Chem. [Anal.], 1929, 1, 56).—For standard gelatin for physico-chemical work the following features are tentatively specified :—calfskin gelatin from green stock extracted at  $54 \cdot 4^{\circ}$  and dried at not less than 5% concentration; ash, max.  $0 \cdot 05\%$ ; constant isoelectric point; definite viscosity and jelly strength under standardised conditions; clear, colourless, and free from preservatives; fat, probable max.  $0 \cdot 1\%$ ; heatcoagulable protein probable max.  $0 \cdot 2\%$ ; arsenic, max.  $0 \cdot 5$  p.p.m. (as trioxide); zinc, max. 20 p.p.m.; copper, max. 10 p.p.m.; other heavy metals, max. 50 p.p.m.

D. F. Twiss.

See also A., Mar., 353, Specific action of proteolytic enzymes (WALDSCHMIDT-LEITZ).

# PATENTS.

Tanning of hides and skins. E. G. STIASNY and B. JALOWZER (B.P. 305,562, 7.11.27).—The materials are tanned with, or treated prior to or after treatment with other materials with, a compound of iron and (a) an aliphatic organic acid containing more than one carbon atom in its molecule, *e.g.*, ferric lactate, or (b) a sulphonic acid of an aromatic hydrocarbon, *e.g.*, naphthalene, anthracene, or mixtures forming these. Watersoluble carbohydrates, *e.g.*, sulphite-cellulose extract, may be added to the solution of the iron compound.

D. WOODROFFE. . Employment of highly sulphonated Turkey-red oils in tanning. M. FLESCH (B.P. 282,710, 4.8.27, Ger., 27.12.26).—An acid-resisting, highly sulphonated Tur-

key-red oil, containing over 6% of organically combined sulphuric acid, is used, either alone or with organic acids, in the pretreatment and first stages of tanning of hides and skins. D. WOODROFFE.

Hydrolysis of casein or casein-containing bodies. R. ARNOT (B.P. 306,168, 12.11.27).—Casein or material containing it, *e.g.*, horse-chestnuts, soya beans, lupin seeds, etc., is treated under pressure and at 135° or below with water in the presence of alkali carbonates, alkaline-earth or magnesium hydroxides, or organic bases. The products may be used for the preparation of foodstuffs or of adhesives. L. A. COLES.

# XVI.—AGRICULTURE.

Soil-sampling tube. E. J. VEIIMEYER (Soil Sci., 1929, 27, 147—152).—The tube is characterised by a detachable cutting point shaped to minimise compression of the soil beneath it and of the core sample within. A specially constructed head withstands hammering and allows of easy withdrawal. Nearly dry sand and gravelly soils may be sampled without difficulty.

A. G. POLLARD.

Measurement of "suction forces" in soil. C. F. SHAW (Soil Sci., 1929, 27, 111—115).—Apparatus involving the suction of water by soil through porous tubes etc. cannot give true values for the "suction forces" of soils. The porous materials themselves have high "suction forces," and soil enclosed within them does not exert any greater suction than in taking up water from a free surface. A. G. POLLARD.

Usefulness of capillary potential to soil-moisture and plant investigators. L. A. RICHARDS (J. Agric. Res., 1928, **37**, 719—742).—Capillary potential in soil is explained and utilised as a basis of a general con-sideration of soil-water relationships. The vertical and horizontal flow of water in soil can be mathematically expressed in a manner analogous to that for the flow of heat and electricity, viz., as capillary conductivity  $\times$  potential gradient. Experimental results illustrating the application of the potential theory are recorded, and apparatus for the determination of the factors concerned is described. The availability of the soil water to plants depends on the tenacity with which the water is held by the soil and the ease of its transmission towards the plant root. The capillary potential is a direct measure of the first factor. A porous clay apparatus for the control of soil moisture in pot experiments is described. A. G. POLLARD.

Determination of the fineness of marl. E. P. DEATRICK and C. DORMAN (Soil Sci., 1929, 27, 121-122). -Current methods for the examination of marl involve screening the dried but not deflocculated material. More satisfactory results are obtained by passing an alkaline aqueous suspension of marl through a battery of fine sieves. A. G. POLLARD.

Condition of formation and constitution of the humic-clay complex of soils. A. DEMOLON and G. BARBIER (Compt. rend., 1929, 188, 654-656).—The fixation of humic acid by brick clay decreases as the  $p_{\rm H}$  value of the medium increases. Experiments on the influence of the concentrations of humate and calcium

ions indicate that colloidal clay is a factor in the fixation of humic colloids in soils, the properties of the complex depending on the cations adsorbed by the former.

J. GRANT.

Colorimetric determination of nitrates in soils and waters. L. U. DE NARDO (Compt. rend., 1929, 188, 563-565).-A 2.5% solution of pyrogallolsulphonic acid (0.5 c.c.) is added to 10 c.c. of a solution containing 0.0005-0.1 mg. of nitrate (as KNO<sub>3</sub>) and 20 c.c. of sulphuric acid, and the brown-red to olive-green coloration matched after 1 hr. against that produced with a standard nitrate solution. For greater amounts 0.5 c.c. of a 2.5% solution of pyrogallol preserved with 0.1%of sodium bisulphite is added to 5 c.c. of solution and 25 c.c. of sulphuric acid. Ferric salts and iodides give a violet colour, chromates yellow, and organic matter brown, and extracts of soils should therefore be treated in succession with solutions of baryta (saturated and boiling) and basic lead acetate (50%), the excess of which is removed by means of a saturated solution of sodium sulphate, and the solution filtered. Nitrites, which react as nitrates, are eliminated by shaking with 1 c.c. of sulphuric acid and a drop of a concentrated solution of urea for 10 min. J. GRANT.

Influence of manganiferous soils on the accuracy of the quinhydrone electrode. W. T. McGEORGE (Soil Sci., 1929, 27, 83–88).—The quinhydrone electrode gives abnormally high  $p_{\rm H}$  values in soils containing appreciable quantities of manganese. The potentiometer drift during the experiment is sufficient indication of the doubtful nature of the results. A. G. POLLARD.

Significance of hydrogen-ion concentration in soil nitrification studies. H. HUMFELD and L. W. ERDMAN (Proc. Iowa Acad. Sci., 1927, 34, 63-66).— Nitrification proceeds when  $p_{\rm H}$  4·4—4·8 is reached; thereafter it is slow. Whenever the final  $p_{\rm H}$  is below 4·2 the amount of ammonium sulphate nitrified is small. Nitrification is considerably increased by addition of calcium carbonate. The final reaction of the soil is somewhat more acid than the initial reaction.

# CHEMICAL ABSTRACTS.

Determination of the nutrient value of soils by pot experiments. H. WIESSMANN (Z. Pflanz. Düng., 1929, 8B, 76–89).—The criticisms of Maiwald (B., 1928, 764) are replied to and details of the author's methods explained. A. G. POLLARD.

Application of nitrogenous fertilisers. O. ENGELS (Z. Pflanz. Düng, 1929, 8B, 49-61).—An explanatory survey of the usually accepted tenets of nitrogenous fertilisation is given. The use of chemical fertilisers is discussed from the point of view of their effects on soil reaction, physical properties, and cropping.

## A. G. POLLARD.

Effects of different nitrogenous fertilisers. GER-LACH (Z. Pflanz. Düng., 1929, 8B, 89-91).—Average figures from numerous field trials indicate calcium nitrate to be as efficient as Chile saltpetre and superior to ammonium sulphate. No increase in crop yields is attributable to the iodine in Chile saltpetre.

A. G. POLLARD. Influence of organic matter and lime on soil moisture and on the percentage of carbon and nitrogen in field soils. J. F. MULLER (Soil Sci., 1929, 27, 137-141).—Generally speaking, soils treated annually with organic manures had higher moisture contents than when minerals alone were used. Liming decreased the moisture content of the soils to an extent approximating to one half the increase resulting from the use of dung. Both the nitrogen and carbon contents of soils treated with organic manure increased during the period of examination. In all other cases the proportions of these constituents decreased.

A. G. POLLARD.

Physico-chemical effects of organic soil colloids. W. F. LOEHWING (Proc. Iowa Acad. Sci., 1927, 34, 149—152).—Liming of certain acid soils is injurious; decreased potash assimilation is associated with the change from acidity to alkalinity or with release of some toxic substance, *e.g.*, iron. Administration of potash following lime injury does not restore the yield on organic soils. CHEMICAL ABSTRACTS.

Effect of crop growth on replaceable bases in some Californian soils. J. C. MARTIN (Soil Sci., 1929, 27, 123-136).-Variations in the amounts of replaceable bases in soils lie within narrower limits among soils of similar classification than among soils of different origin. Clays and silty clay loams have much larger proportions of replaceable bases than sandy loams. Soils cropped annually for a lengthy period showed no marked difference in the content of replaceable bases from those cropped twice only with a long fallow interval. Continuous cropping decreased the proportion of replaceable potassium in soils without significant change in the total bases present, since the replaceable calcium and magnesium, comprising 90% of the total, remained unaltered. The replaceable potassium removed by twelve annual barley crops represented 32% of the amount originally present and 82% of the total potassium contained in the crops. Replaceable bases in soil as measured by extraction with 0.05N-acid are appreciably greater than when ammonium acetate extracts are utilised. In the latter case results agree more closely with the amounts of ammonia adsorbed by the soil. Acid extraction probably results in some decomposition of the adsorbing complex. A. G. POLLARD.

Effect of sodium chloride and carbonate on the growth of asparagus. B. L. HARTWELL, J. B. SMITH, and L. C. DAMON (Rhode Is. Agric. Exp. Sta. Bull., 1928, No. 213, 16 pp.).—Potassium chloride or alkali carbonate decreased, whilst sodium chloride increased, soil acidity. Heavy rainfall over two months reduced active acidity and active alumina. Omission of sodium decreased the yield; in plots deficient in potassium, sodium chloride and carbonate increased the yield.

CHEMICAL ABSTRACTS.

See also A., Mar., 262, Dual emulsions for spraying of trees (WOODMAN). 289, Geology and mineralogy of soils (HART). 337, Determination of iodine in soil (SCHWAIBOLD). 360, Forcing of plants by warm baths (BORESCH). Influence of radiations on germination of seeds and growth of plants (MEZZADROLI and VARETON). 362, Chlorosis of fruit trees (WALLACE).

#### PATENTS.

Manufacture of fertilisers. R. E. SLADE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 305,760, 13.12.27).—A mixture of, e.g., 90% ammonium nitrate solution containing 43 pts. of ammonium nitrate with 57 pts. of calcium carbonate obtained as a by-product in the manufacture of ammonium sulphate from ammonium carbonate and calcium sulphate, is evaporated to dryness by heating at 50—100° with constant stirring in a vacuum evaporator. When dry, the mixture is sieved and oversize particles and fines are mixed with a subsequent charge. The product contains 15% N. L. A. COLES.

Cold frames and like transparent roofings for use especially in horticulture. J. Y. JOHNSON. From I. G. FARBENIND, A.-G. (B.P. 306,242, 2.1.28).— Sheets of transparent material comprise cellulose ethers, urea-formaldehyde condensation products, etc. strengthened by an embedded network. L. A. COLES.

Caking of crystals (B.P. 305,721).—See I. Fertiliser salts (B.P. 304,872).—See VII.

# XVII.—SUGARS; STARCHES; GUMS.

Analysis of maple products. X. Modification of the Canadian lead method. XI. Composition of the Canadian lead precipitate. D. E. FOWLER and J. F. SNELL (Ind. Eng. Chem. [Anal.], 1929, 1, 8-13).-X. In order to eliminate errors due chiefly to formation of fissures in the precipitate during washing, the following method is proposed. The prepared syrup containing 25 g. of dry matter is weighed, made up to 100 c.c. at 20°, and 20 c.c. are pipetted into a large test tube; to this 2 c.c. of basic lead acetate solution are added, the tube is corked, and, after 2 hrs., the contents are filtered with suction through an asbestos mat at least 3 mm. thick. Four washings with cold water are made, care being taken to keep the precipitate covered and that filtration is not quicker than 40 drops per min. The precipitate is dried at 100° and weighed. Somewhat higher results are thereby obtained.

XI. Analysis of the lead precipitates from three maple syrups showed that the total lead content is greater  $(72 \cdot 64 - 67 \cdot 58\%)$  and the basic lead content slightly higher the lower are the Canadian lead values. The difference between total and basic lead exceeds considerably the lead equivalent of the acids freed by hydrogen sulphide, and 97% of these latter acids was malic acid as determined polarimetrically in a composite sample of more than 20 syrups. Substances more carbonaceous than lead malate are present in the lead precipitate since they contained  $11 \cdot 1 - 12 \cdot 2\%$  C and  $1 \cdot 46 - 1 \cdot 15\%$  H. Flavouring and colouring matters appeared to be precipitated. D. G. HEWER.

Determination of moisture in sugar syrups. E. W. RICE (Ind. Eng. Chem. [Anal.], 1929, 1, 31—32).— Half of a 10 g. roughly weighed portion of dried Filter-Cel is put in a flask followed by 15 g. of the syrup (weighed in a capsule of thin waxed paper), and the rest of the Filter-Cel is added. Distillation is then carried out as described by Bidwell and Sterling (B., 1925, 268). The whole operation occupies only 4 hrs.,

no blackening or decomposition of the residue occurs, and the same rate of distillation of the toluene can be maintained with the oil bath at 127° as at 143° without Filter-Cel. D. G. HEWER.

Determination of the adhesive power of starch by Saare's method. W. EKHARD (Z. Spiritusind., 1929, 52, 70—71).—A metal plate 22 mm. in diam., having a rod attached perpendicularly to its centre, is suspended 3 cm. below the surface of 200 g. of a carefully prepared solution, containing 9 g. of starch, by means of a small transverse rod which rests in a slot in an adjustable metal bridge fixed to the rim of the 250 c.c. beaker. After allowing the starch solution to set for 24 hrs., the vertical rod is connected by means of a hook at the end to one beam of a balance, and sand is gradually added through a funnel to the pan attached to the other beam until the metal plate is pulled out of the starch paste. The weight of sand required is taken as a measure of the adhesive power of the starch.

F. R. ENNOS.

Quantitative separation of dextrins and gum arabic. A. HAMY (Ann. Falsif., 1929, 21, 24-26).-To 20 c.c. of the gum arabic syrup, previously diluted to twice its volume, are added 23 c.c. of 95% alcohol and the solution is made up to 55 c.c. with water; after a few hours the insoluble dextrins are deposited, no gum being precipitated in alcohol of this concentration. 50 c.c. of the solution are filtered and 90 c.c. of water containing 5 g. of dextrose and 8 g. of sucrose are added, followed by 42 c.c. of 95% alcohol and 15 c.c. of lead subacetate, and, after shaking, is left to settle overnight. The supernatant liquid is decanted, and the residue centrifuged in 2 tubes, the precipitate and tubes being washed with 50 c.c. of 50% lead acetate. The precipitate in each tube is mixed with 4.3 c.c. of a 75% sugar solution, which effects partial dissolution, and 35 c.c. of a mixture of 120 c.c. of 95% alcohol, 27 c.c. of lead subacetate, and 160 c.c. of water are added. The precipitate, after being centriguged, is dissolved in 4% acetic acid, and the gum precipitated by alcohol. When precipitation is complete, the gum is filtered, dried at 110°, and weighed, then calcined and weighed again. D. G. HEWER.

See also A., Mar., 267, Saturation of sugar-lime solutions (ATEN and others). 272, Mutarotation of galactose (Lowry and SMITH). 297, Precipitation of carbohydrates by alkaloids (ROSENTHALER). 298, Colorimetric determination of dextrose (SCHACKEL-DIAN). Distillation of sucrose (HIDAKA). 299, Starch acetate (BRIGL and SCHINLE). Disruption of maize starch granules (TAYLOR and BECKMANN). 341, Colour reactions of the carbohydrates (DISCHE). 352, Enzymic fission of sucrose (WEIDENHAGEN). 354, Conversion of dihydroxyacetone into hexose by fermentation (NEUBERG and KOBEL; IWASAKI). 360, Nature of waxy starch of maize (BRINX).

# XVIII.—FERMENTATION INDUSTRIES.

Maintenance and regeneration of warm-stored bottom- and top-fermentation yeasts. F. WINDISCH (Woch. Brau., 1929, 46, 71-74, 81-86).—Yeasts the reproductive and fermentative powers of which had been weakened by storage under beer for three days at  $18-20^{\circ}$  maintained their weakened character without further deterioration when similarly stored between four successive fermentations. The lower reproduction and fermentation rates as compared with yeasts stored at  $2-6^{\circ}$  are not due to the slightly higher proportion of dead cells. After four warm-storage periods the yeasts regained their original reproductive and fermentative activity when kept for 3 days under beer at  $2-6^{\circ}$ .

F. E. DAY.

Drying of hops in vacuo. K. BEHRENDT (Woch. Brau., 1929, 46, 86-87).-Four samples of hops were dried under partial vacuum (100 mm.) in a current of partly dried air. The air entered the drying vessel at 50-52° and left at 20-25° at the start, rising to 30-36° at the end of drying, which occupied 5-8 hrs. The hop moisture was reduced from 64.4-70.2% to 6.13-14.85%. The dried samples were compared as regards humulone and resin content with portions of the same samples dried on trays in air for 2-3 days, and with samples from the kiln-dried bulks. In all cases the vacuum- and air-dried samples contained similar amounts of humulone, which in three cases exceeded that in the kiln-dried material by 3-30%. The exceptional sample had been damaged in transit, and the results indicate that vacuum-drying causes as little harm as air-drying and less than kilning. F. E. DAY.

Importance of hydrogen-ion concentration for the distillery. II. W. DIEMAIR and K. SICHERT (Biochem. Z., 1929, 204, 414-430; cf. B., 1928, 797).-During the whole period of fermentation maize mashes differ considerably as regards acidity and state of combination of the nitrogen from potato mashes, the latter having a lower  $p_{\rm H}$  maximum and producing more titratable acid than the former The difference in amount of titratable acid is due to alterations in the composition of the yeast which, in the maize mashes, cannot obtain sufficient nitrogen for growth and so decomposes, leaving much protein and protein decomposition products in the substrate. Hence, in this case, the buffering power is at a maximum. No relation could be found between the forms of the curves of acidity and buffering power. The results of fermentation are best when the maxima of the two curves do not coincide. W. McCARTNEY.

Estimating heat consumed in batch rectification of alcohol. G. S. HOUGHLAND (Chem. & Met. Eng., 1929, 36, 89-91).- A graphical method is described for determining the average theoretical heat consumption during intermittent rectification, and is applied to the concentration of ethyl alcohol from aqueous solutions of 1-20%, producing distillate containing 92.46% of alcohol and exhausting the residue to 0.1% of alcohol, in columns containing 9-16 perfect plates. The composition of the residue in the kettle and of the vapours arising therefrom being known, the theoretical heat quantities evolved at the instant when a given weight of distillate has passed over may be calculated, and these values may be plotted against the weight of distillate to give a curve the area under which represents the H. S. GARLICK. desired total heat consumption.

See also A., Mar., 352, Separation of enzymes of barley malt (PRINGSHEIM and THILO). Action of light on takadiastase in presence of sensitisers (KAM- BAYASHI). Enzymic fission of sucrose (WEIDEN-HAGEN). Liberation of invertase from yeast (WILLSTÄTTER and GRASSMANN). 353, Specific action of proteolytic enzymes (WALDSCHMIDT-LEITZ). Specific action of erepsin, trypsin, and trypsinkinase (AbderHalden and others). 354, Conversion of dihydroxyacetone into hexose (Neuberg and KOBEL; IWASAKI). 355, Lactic acid fermentation (VIRTANEN).

Power alcohol. THAYSEN and GALLOWAY.-See II.

#### PATENT.

Extraction of oils and resins from hops. Drying of hops. L. WEBER (B.P. 305,066 and 305,076, 27.10.27). -(A) Hops are drawn by means of a vacuum into a cylindrical still with a vertical axis furnished with rakes and so arranged that the hops are confined by two perforated plates between top and bottom dome-shaped jacketed covers. After drying the hops by the method described under (B), steam is passed under reduced pressure through the hops and the volatile oils are recovered from the condensed vapour. When the hops have again been dried as before, concentrated alcohol, preferably 96%, is circulated through the hops to extract the resins. When extraction is complete, the alcoholic solution is concentrated and run off, and the alcohol and resins are separated by distillation under as high a vacuum as possible. Following the passage of warm air which removes and permits the recovery of the alcohol remaining in the hops, warm water is circulated through the hops to extract the remainder of the resins, tannins, etc. (B) Using the still charged with hops as in (A), air or alcoholised air at 21-32° is drawn in through an inlet in the bottom cover of the still and allowed to emerge by an outlet in the top cover, the stirring gear being maintained in motion during the process. The dried hops are removed from the still by suction and baled for the market. The oil and vapour collected during the passage of the warm air are condensed and collected. C. RANKEN.

XIX.—FOODS.

Improvement of flour. M. VUK and P. SPANYAR (Z. Unters. Lebensm., 1928, 56, 292-308).—The effects on the baking qualities of flour of addition of wheat germ, sugar, dextrin, potassium bromate, ammonium persulphate, sodium borate, "novadelox," and of treatment with nitrogen peroxide and "gologas" (chlorine containing 0.5% of nitrosyl chloride) were studied. Wheat germ improves the baking qualities, but cannot be used owing to its effect on colour and taste of the bread. Sugar or, to a smaller extent, dextrin is beneficial, but addition of the above salts gave no definite improvement. Novadelox resulted in improved texture and bleaching, nitrogen peroxide in bleaching only, but gologas had a very favourable effect especially on bread volume and texture. Gologas is a flour improver rather than a maturing agent, as flour matured by ageing can be further improved thereby. Its action appears to be due to the production of acidic protein substances in the flour. W. J. BOYD.

Semi-microchemical method for the determination of gluten in flour. A. C. RÖTTINGER (Mikrochem., 1929, 7, 106—109).—The flour (1 g.) is moistened with two drops of water in a bag of suitable material and, after 30 min., the paste is kneaded until no further starch is removed. The small ball of moist gluten obtained is removed from the bag and dried in an evacuated tube immersed in a water-bath. The weight of dried material obtained multiplied by 3 gives approximately the weight of moist gluten obtained in the usual macro-test using 30—40 g. of flour. A. R. POWELL.

Heat-resistant and heat-loving bacteria in their relation to the pasteurisation of milk. R. S. BREED (New York State Agric. Exp. Stat., Bull. No. 559, 1928, 19 pp.) .- A review of recent investigations on the subject. Under certain conditions milk may contain more living bacteria after pasteurisation than before, owing to the introduction into the raw milk of both heat-resistant and heat-loving bacteria. The former, which survive pasteurisation temperatures, tend to grow well at 38-45° and produce the so-called "pin-point" colonies when incubated at 37° on agar plates ; they are probably derived from insufficiently sterilised dairy utensils. The latter, which are not only not killed, but actually multiply rapidly at the pasteurisation temperature, do not grow on agar plates at 37° as the temperature is too low; their presence may therefore be overlooked in ordinary routine work, but they may be detected by incubation at 63° or as rod-like bacilli by microscopical examination of the pasteurised milk. The source of these heat-loving bacteria is the dust, hay, silage, etc. in proximity with the milk, and their development is favoured by faulty procedures during pasteurisation, such as holding the milk at pasteurisation temperature for longer than 30 min., repasteurisation of left-over milk, and the incomplete removal of one batch of milk before introduction of the next. There is no evidence to show that the presence of these bacteria in milk is dangerous to human health, but in excess they cause a noticeable change in the acidity of the milk and also disagreeable flavours. F. R. ENNOS.

Types of bacteria which produce a "caramel" flavour in milk. C. D. KELLY (Trans. Roy. Soc. Canada, 1928, [iii], 22, V, 227-232).—The four strains of organisms studied are classified (on fermentative properties) as variants of *Streptococcus lactis* (Lister) according to Orla-Jensen's classification.

F. C. HAPPOLD. Sireptococcus lactis strain that produces "caramel" odour and flavour in dairy products. W. SADLER (Trans. Roy. Soc. Canada, 1928, [iii], 22, V, 243-248).—The fermentative action of the organism on different carbohydrates has been quantitatively studied. The acid produced was titrated against 0.25N-caustic soda and recorded as lactic acid.

F. C. HAPPOLD.

Casein-splitting properties of starters [in ripening of cheese]. C. BARTHEL and W. SADLER (Trans. Roy. Soc. Canada, 1928, [iii], 22, V, 233— 235).—The amounts of soluble nitrogen produced by pure cultures of lactic acid bacteria of the *Streptococcus* group is comparable with that produced by "starters," but the amount of amino-nitrogen produced is much

less with the former. This indicates that the hydrolysing action of the different *Streptococci* present in cheese is of a symbiotic nature. F. C. HAPPOLD.

Proportion of the citrates of milk incorporated in the curd during cheese-making. N. S. GOLDING (Trans. Roy. Soc. Canada, 1928, [iii], 22, V, 237— 242).—Only small amounts of the milk citrate are held by the curd ; these amounts cannot affect significantly the ripening of cheese. F. C. HAPPOLD.

Action of rennin. W. GRIMMER and E. HINKEL-MANN (Milch. Forsch., 1928, 6, 274–301; Chem. Zentr., 1928, ii, 1399).—Rennin, freed from pepsin by treatment with a small quantity of alumina, gave a productcurve which rose after a preliminary fall, and corresponded with the equation  $J_1 = p/2 (c_1^x + c_2^x)$ ; "peptolab," after purification, gave a falling logarithmic curve. In presence of phosphate "peptolab" gave almost constant product-values. A purified rennin preparation behaves like an unpurified preparation towards souring milk; the coagulation time decreases with increasing acidity. A. A. ELDRIDGE.

Lipolysis of worked butter several days after preparation. O. GRATZ (World's Butter Rev., 1928, 2, No. 7-8, 21-22).—Bacteria develop most rapidly on the outer layer of butter; mixed butters have a greater acid content when the outer layer is not removed, and lipolysis in the butter may be greater after 14 days. CHEMICAL ABSTRACTS.

Cacao beans and cocoa products. VI. H. FINCKE (Z. Unters. Lebensm., 1928, 56, 312-334; cf. B., 1928, 911).-Observations on the calculation of the composition of chocolate preparations are made in view of the specifications drawn up by the Verbande Deutscher Schokolade-Fabrikanten. Addition of coffee to chocolate does not necessitate modification of methods or calculations. The effect of fermentation on the appearance, taste, odour, and chemical composition of cacao beans has been studied. The fat of immature beans does not differ from that of riper beans. Fermentation does not change the properties of the fat. A case of an abnormal cacao butter from a consignment of Accra beans derived from only a few trees is reported. The analytical data indicate an unusually high content of unsaturated fatty acid of low m.p., probably oleic acid. Similar divergence from the normal could not be found in consignments from many different sources nor in the fat of individual beans. It is probable that the theobromine in cacao exists partly free and partly in loose combination with tannin, from which it can be liberated by moistening the material with addition of magnesium oxide as well as by treatment with dilute sulphuric acid. W. J. BOYD.

Comparison between complete and incomplete digestion of sprayed apple foliage in determining arsenic by the Gutzeit method. J. M. GINSBURG (J. Econ. Entomol., 1928, 21, 588-592).—When the material is boiled for 30 min. with 10% nitric acid or 20% hydrochloric acid, the amount of arsenic recovered is equal to that obtained after complete digestion with nitric and sulphuric acids. CHEMICAL ABSTRACTS. Use of 2:6-dichlorophenol-indophenol as reduction indicator in the examination of foodstuffs. J. TILLMANS, P. HIRSCH, and E. REINSHAGEN (Z. Unters. Lebensm., 1928, 56, 272—292).—The behaviour of the indicator in the presence of milk, meat extract, and lemon juice was studied. Milk showed no welldefinied action on it, but the other products exhibited definite reduction. In no case was it found possible to determine the reduction—oxidation potential colorimetrically thereby. The reducing effect of lemon juice on 2:6-dichlorophenol-indophenol provides a method of distinguishing the natural from the artificial product.

W. J. BOYD.

See also A., Mar., 337, Determination of iodine in milk (Schwalbold): 358, Vitamin content of honey (Hoyle). 362, Chemical changes in stored apples (Haynes and Archbold). Composition of juices of some American apples (Caldwell). Boron compounds in fruits and vegetable products (Dodd).

# PATENTS.

Making wheat-meal bread. D. FRAME (B.P. 305,845, 7.3.28).—The yeast is grown in an emulsion consisting of sweet milk, yolk of eggs, malt extract, and treacle, and then mixed with wheat meal, butter, and salt. The dough is divided, then placed in a chamber at 30° for 15 min., and baked at about 205°. W. J. BOYD.

Egg product and its production. A. K. EPSTEIN (B.P. 305,844, 6.3.28).—Egg yolk, before being frozen, is treated with an amount of a neutral edible salt sufficient to produce in the product, when thawed, a viscosity greater than that of untreated yolk before freezing, and less than that of untreated yolk after being frozen and thawed. The addition of 2—10% of sodium chloride gives good results, but other neutral salts or neutral mixtures of salts may be used.

#### W. J. BOYD.

Food product. M. NILSSON, ASST. to FLEISCHMANN Co. (U.S.P. 1,701,081, 5.2.29. Appl., 2.3.26).—Compressed yeast, dried at about 30° to a moisture content of 55%, is mixed with dry sugar material, so that the resulting product contains about 20% of moisture and about 60% of sugar. H. ROYAL-DAWSON.

Chewing gum. CANADIAN ELECTRO PRODUCTS Co., LTD., Assees. of H. W. MATHESON (B.P. 280,247, 8.11.27. U.S., 8.11.26).—A gummy product is prepared by reaction between an aliphatic vinyl ester (e.g., vinyl acetate) and a saturated aliphatic aldehyde (e.g., acetaldehyde); sugar, flavouring ingredients, or gum chicle may be added. W. J. BOYD.

Manufacture of ordinary bread. J. MATTI, Assr. to AUGMENTINE HOLDING Soc. ANON. (U.S.P. 1,701,580, 12.2.29. Appl., 25.6.27. U.K., 25.6.26).— See B.P. 266,967; B., 1927, 376.

Preservation of perishable [tinned] food products. D. ROBERTSON (B.P. 276,646, 4.8.27. N.Z., 30.3.27).

Mills for grinding etc (B.P. 303,227).—See I. Hydrolysed casein (B.P. 306,168).—See XV. Sterilisation (B.P. 274,909).—See XXIII.

# XX.-MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Determination of free iodine and potassium iodide in tincture of iodine (D.A.B. VI). K. WINTER-FELD and H. SIECKE (Apoth.-Ztg., 1928, 43, 852—854; Chem. Zentr., 1928, ii, 1363).—The tincture (1-1.5 g.)is mixed with aqueous potassium bromide (3 g. in 10 c.c.) and diluted to 100 c.c. The iodine is determined by titration of a portion with thiosulphate. For the determination of potassium iodide, 10 c.c. are mixed with 25 c.c. of potassium hypobromite solution; after 15 min. phenol is added, and then hydrochloric acid to acidity (methyl-orange). Potassium iodide and phosphoric acid are then added, and after 20 min. the iodine is titrated with thiosulphate. A. A. ELDRIDGE.

Determination of chloral in chloral syrup. C. LORMAND (J. Pharm. Chim., 1929, [viii], 9, 151-153; cf. Fleury and Malmy, B., 1929, 188).—10 g. of chloral syrup containing about 0.5 g. of chloral are added to a mixture of ammonia solution (d 0.925) 50 c.c., silver nitrate 4 g., and potassium hydroxide 5 g. The mixture is kept for 24 hrs., boiled on a water-bath to remove excess of ammonia, and then acidified with a slight excess of nitric acid. After diluting to 100 c.c. with water, the liquid is warmed to coagulate the silver chloride, which, after cooling, is collected and weighed. It is claimed that this process gives more accurate results than the alkalimetric titration process.

E. H. SHARPLES.

Liquor aluminii acetico-tartarici, D.A.B. VI. H. MATTHES and P. SCHÜTZ (Apoth.-Ztg., 1928, 43, 1023-1024; Chem. Zentr., 1928, ii, 1469).—The (German) official method of analysis is criticised.

A. A. ELDRIDGE.

Determination of cresol in liquor cresoli saponatus. K. FEIST (Apoth.-Ztg., 1928, 43, 1024–1025; Chem. Zentr., 1928, ii, 1469).—5% of the light petroleum extract (D.A.B. VI) is treated with formaldehyde solution (5 c.c.) and 25% hydrochloric acid (4 c.c.), and the mixture dried to constant weight  $(1\cdot0-1\cdot1 \text{ g.})$  at 100° (cf. B., 1929, 188). A. A. ELDRIDGE.

Nicotine dusts. W. R. HARLAN and R. M. HIXON (Iowa State Coll. J. Sci., 1928, 2, 313—316).—Losses of nicotine on adsorbents are due to adsorption, and not to chemical reaction. Nicotined bentonite, when extracted with ether, retains 88% (3% dust), 45% (6% dust), or 20% (14% dust) of nicotine, but less in presence of calcium hydroxide. The adsorbed nicotine can be recovered by steam-distillation. CHEMICAL ABSTRACTS.

Determination of pilocarpine. P. BOURCET (Ann-Falsif., 1929, 21, 23-24).—Jaborandi leaves (25 g.) are bruised, passed through a no. 30 brass sieve, moistened with 200 c.c. of 10% sodium carbonate, extracted for 3 hrs. with warm benzene, and the cooled solution is immediately extracted 4 times with 1% sulphuric acid, with shaking. The alkaloids dissolve, and the filtered solution, neutralised to Congo-red with ammonia, is oxidised by 10% potassium permanganate, rendered alkaline with ammonia, and repeatedly extracted with chloroform. The chloroform solution, filtered over sodium carbonate, is neutralised with nitric acid (1:50). evaporated to dryness, and the residue extracted with acetone, which dissolves impurities, leaving the pilocarpine nitrate. After filtering and drying, the latter is weighed; the m.p. should be 174—175° and not less than 165°. In certain samples if the benzene solution of alkaloids is converted into sulphate and left for 24—48 hrs., particularly in light, the proportion of pilocarpine is found to have diminished by half, but if a benzene or light petroleum extract of the leaves themselves is treated with sodium carbonate and then with benzene this loss does not occur. D. G. HEWER.

Use of the refractometric method in determination of oil in avocados. B. E. LESLEY and A. W. CHRISTIE (Ind. Eng. Chem. [Anal.], 1929, 1, 24).-The oil content of avocados may be rapidly determined within an error of 0.3% by transferring 5 g. of finelyground and well-mixed avocado paste into a warm mortar, pipetting in 5 c.c. of Halowax oil (a commercial substituted monochloronaphthalene), grinding for 5 min., determining the refractive index on one drop of the oil mixture, which readily separates, and repeating the process after further intervals of grinding until a constant value is obtained, which is then corrected to 25° by adding or subtracting 0.0004 for every degree above or below 25°, respectively. The percentage (c) of oil present is [(a - 0.0032) - b]/d, where a and b are values of  $n_{\rm p}^{25}$  for Halowax oil and the oil mixture, and d is the change in  $n_{\rm D}$  per percentage of oil in the sample. The value obtained for several samples is 0.001555.

## D. G. HEWER.

See also A., Mar., 297, Catalytic reduction of geraniol and citronellal (SUZUKI). 297, Precipitation of carbohydrates and glucosides and by alkaloids (ROSENTHALER). 313, Resolution of dl-thyroxine (HARINGTON). Derivatives of thyroxine (AshLEY and HARINGTON). 315, Products with an odour of musk 323, Transformation of pinocarveol (RUZICKA). and sabinol (SCHMIDT). Essential oil and alcohol from Camphorosma Monspeliacum (MAZZA and SPAGNOLO). 335, Amine oxides of hydrastine and narcotine (POLONOVSKI). Dehydrogenation of yohimbine (MENDLIK). 336, Organo-antimony compounds (NIYOGI). 337, Determination of iodine in thyroid extract (SCHWAIBOLD). 351, Lipo-soluble bismuth compounds (LEVADITI). 357, Standardisation of insulin (FREUDENBERG and DIRSCHERL). 361, Constancy of essential oil of seedlings of aniseed fruit (IVANOV and GRIGOROJEVA). Total alkaloids of Datura fastuosa and alba (MARANON).

# PATENTS.

Blood coagulant. M. BYE, Assr. to W. S. MERRELL Co. (U.S.P. 1,697,162, 1.1.29. Appl., 11.2.27).—A brine extract of lung or other tissue is treated with excess of acid, and the resulting precipitate is dissolved in caustic alkali. The water-white, neutral solution obtained may be evaporated to dryness. F. G. CLARKE.

Therapeutic agent for treatment of bodily lesions. H. L. JOHNSON (U.S.P. 1,696,958, 1.1.29. Appl., 2.3.27).—Amniotic fluid of cows and other mammals, after removal of impurities and micro-

organisms by filtration, is applied to lesions of the human body during or after operations.

# F. G. CLARKE.

Manufacture of solutions of the active principle of organs or glands with internal secretion. I. G. FARBENIND. A.-G. (B.P. 283,493, 7.12.27. Ger., 11.1.27. Addn. to B.P. 279,123; B., 1929, 226).—The process of the prior patent is applied to preparation of active principles from other organs, especially male germglands or the anterior lobe of the hypophysis.

C. HOLLINS.

Manufacture of optically active phenylpropanolmethylamines [ $\beta$ -methylamino- $\alpha$ -phenyl-n-propyl alcohols ; *l*-ephedrine]. I. G. FARBENIND, A.-G. (B.P. 297,385, 11.9.28. Ger., 20.9.27).—By resolution of dl- $\beta$ -methylamino- $\alpha$ -phenyl-n-propyl alcohol (ephedrine) with *l*- instead of with *d*-tartaric acid the *l*-tartrate of *l*-ephedrine, m.p. 148° (hydrochloride, m.p. 215—216°, [ $\alpha$ ] —34°), crystallises before the *d*-ephedrine *l*-tartrate (*d*-base, m.p. 39—40°; hydrochloride, m.p. 215°, [ $\alpha$ ] +34°). The *l*-base is therapeutically more valuable than its enantiomorph. C. HOLLINS.

Manufacture of urethanes [carbamates] of secondary alcohols. I. G. FARBENIND. A.-G. (B.P. 300,177, 6.11.28. Ger., 7.11.27).—Carbamates of secondary alcohols containing an aryl group  $\gamma$  to the hydroxyl group are powerful antipyretics. The alcohol is treated first with carbonyl chloride and then with ammonia. Examples are  $\delta$ -phenyl- $\beta$ -n-butyl carbamate, CH<sub>2</sub>Ph·CH<sub>2</sub>·CHMe·O·CO·NH<sub>2</sub>, m.p. 63°,  $\varepsilon$ -phenyl- $\gamma$ -namyl carbamate, m.p. 88°,  $\Delta^{\alpha}$ - $\delta$ -benzyl- $\varepsilon$ -n-hexenyl carbamate, CH<sub>2</sub>: CH·CH<sub>2</sub>·CH(CH<sub>2</sub>Ph)·CHMe·O·CO·NH<sub>2</sub>, m.p. 42°, and  $\delta$ -p-tolyl- $\beta$ -n-butyl carbamate, m.p. 77°. C. HOLLINS.

Preparation of monocyclic lactones with 14 to 18 ring members. Soc. ANON. M. NAEF & CIE. (B.P. 306,352, 26.6.28. Addn. to B.P. 294,602; B., 1929, 235).—Cyclic ketones,  $C_{13}$  to  $C_{17}$ , when warmed at 50° with Caro's acid yield lactones of the corresponding  $\omega$ -hydroxy-acids; cyclopentadecanone is disclaimed. The lactones described are :  $C_{13}$  (lactone of  $\omega$ -hydroxytridecoic acid), m.p. 26—27°, b.p. 148°/15 mm.,  $d_{3}^{33}$ 0·9590;  $C_{14}$ , m.p. 29—30°, b.p. 165°/15 mm.,  $d_{3}^{33}$ 0·9528;  $C_{16}$ , m.p. 35—36°, b.p. 188°/15 mm.,  $d_{3}^{33}$  0·9326;  $C_{17}$ , m.p. 41—42°, b.p. 194°/15 mm.,  $d_{3}^{33}$  0·9326;  $\xi$ hydroxy- $\gamma$ -methylpentadecoic lactone, b.p. 180°/15 mm.,  $d_{3}^{34}$  0·9305, from muscone;  $\xi$ -hydroxy- $\alpha$ -methylpentadecoic lactone, b.p. 178°/15 mm.,  $d_{3}^{34}$  0·9301, from 2-methylexaltone. C. Hollins.

Manufacture of metal mercaptocarboxylic acid esters. SCHERING-KAHLBAUM A.-G. (B.P. 282,427, 15.12.27. Ger., 16.12.26).—o-Thiolbenzoic esters, best obtained from the acid chloride of 2:2'-dicarboxydiphenyl disulphide by interaction with alcohols and subsequent reduction, are heated with oxides or salts of gold, silver, arsenic, antimony, or bismuth to yield metalthiol derivatives soluble in olive oil, sesamé oil, glycerol triethyl ether, and other solvents suitable for injection. n-Butyl o-thiolbenzoate ["thiosalicylate"], b.p. 163°/20 mm., gives a bismuth compound, m.p. 72°; the methyl ester, b.p. 130°/20 mm., yields an antimony compound, m.p. 88°. Aliphatic thiol-substituted acids give similar compounds. C. HOLLINS.

Manufacture of arsenobenziminazolones. I. G. FARBENIND. A.-G. (B.P. 281,690, 1.12.27. Ger., 2.12.26). —Benziminazolonearsinic acids carrying N-substituents (cf. B.P. 256,243; B., 1927, 670) give on reduction arseno-compounds more stable and effective than known arsenicals. Arsenobenziminazolones from 1methyl-, 1-propyl-, and 1-allyl-benziminazolone-5-arsinic acids are described. C. HOLLINS.

Manufacture of a therapeutically active iron preparation having yeast as its basic substance. G. M. CLARK. From MATRO GES.M.B.H. (B.P. 304,895, 24.12.27. Cf. B.P. 300,039; B., 1929, 73).—To ordinary or acetonised yeast suspended in 10 pts. of water is added a solution of a colloidal basic iron salt (liquor ferri chloroxidi, or liquor ferri chloroxidi dialysati). The liquid is shaken overnight, and the solid filtered off, treated with alcohol and ether, and dried at low temperature. B. FULLMAN.

Manufacture of tobaccos. Soc. ZIHNA (B.P. 298,151, 26.6.28. Fr., 4.10.27).—Low-grade tobacco is, preferably after fermentation of the leaves, treated by immersion, spraying, etc. with a solution obtained by diluting 1 pt. of the extract of leaves, waste, or stalks of high-grade tobacco with 10 pts. of alcohol or other volatile solvent. Finished cigarettes or cigars may be treated by injection. The product is allowed to dry.

B. FULLMAN.

Improvement of perfumes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 305,555, 29.10.27).— Substances such as ionones, geraniol, citral, clove oil, etc. are subjected to partial hydrogenation with or without the presence of a catalyst; e.g., 500 g. of methylionone are treated at 60—90° with 30 litres of hydrogen in presence of 5 g. of catalytic nickel for 1 hr.; after distilling in a vacuum, a colourless or nearly colourless methylionone free from the usual empyreumatic odour is obtained. E. H. SHARPLES.

Complex antimony compound. H. HAHL, Assr. to WINTHROP CHEM. Co., INC. (U.S.P. 1,701,234, 5.2.29. Appl., 10.2.27. Ger., 22.4.26).—See B.P. 288,370; B., 1928, 427.

Derivatives of cinchona alkaloids. E. BURCK-HARDT, F. MÜLLER, and E. ROTHLIN, ASSIS. to CHEM. WORKS FORMERLY SANDOZ (U.S.P. 1,701,138, 5.2.29. Appl., 30.11.27. Switz., 17.12.26).—See B.P. 282,356; B., 1928, 769.

# XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Effect of environment on photographic sensitivity. II. Effect of certain salts. S. E. SHEPPARD and E. P. WIGHTMAN (Phot. J., 1929, 69, 134—141; cf. B., 1929, 151).—Potassium citrate, whether present during or immediately after exposure, has no effect on the formation of the latent image, but potassium chloride under the same conditions produces a slight decrease in sensitivity which, however, is attributed to a development effect. Potassium bromide, when present during exposure, markedly inhibits the formation of the latent image, but when present immediately after exposure is almost without action. Although acid silver nitrate causes a strong acceleration of the rate of print-out of the visible image, it has no such effect on the formation of the latent image. The results are discussed in terms of the transfer of electrons from bromide ions to silver ions of the lattice, and it is suggested that the electrostatic field of the adsorbed double layer is a modifying factor of differing importance for visible- and latent-image formation.

## J. W. GLASSETT.

See also A., Mar., 277, Photolysis of silver bromide (MUTTER). Sensitometry of desensitised films (ARENS and EGGERT). 359, Action of the cholesterol of cod-liver oil on the photographic plate (HUGOU-NENQ and COUTURE).

#### PATENTS.

Colour photography. S. DE PROCOUDINE-GORSKY (B.P. 293,038, 25.6.28. Switz., 30.6.27).—A positive is made on sensitised celluloid film from each of the three colour record images, using a hardening developer such as pyrogallol. The positives are fixed, washed, dried, and then immersed in a saturated solution of iodine in 1% potassium iodide, which converts the reduced silver into silver iodide. Each image is then treated in a separate bath containing sodium thiosulphate and a basic dye of suitable colour (e.g., methylene-blue, Auramine, red-magenta), which fixes the dye in the images and slowly removes the silver iodide.

J. W. GLASSETT.

Colour photography. O. E. WHEELER (B.P. 305,775, 28.12.27).—Bromide prints are made from the three negatives representing the blue, red, and yellow impressions. These are squeegeed in contact with an uncoloured, gelatin-coated tissue sensitised as in the "Carbro" process and, after separation, the tissues are transferred to a celluloid support and developed in warm water. The relief images so obtained are used, by means of a dye imbibition method of printing, to obtain prints on any suitable support.

J. W. GLASSETT.

# XXII.—EXPLOSIVES ; MATCHES.

Thermodynamic treatment of explosive reactions. I. A. SCHMIDT (Z. ges. Schiess- u. Sprengstoffw., 1929, 24, 41—46).—Methods are given for calculating for a given explosive its heat of explosion, explosion temperature, the composition and volume of the explosion gases at that temperature, and the explosion pressure when the heat of formation and the stoicheiometric composition of the explosive in question are known. Nitroglycerin, nitrocellulose, and trinitrotoluene are taken as examples of explosives with excess, sufficiency, and deficiency of oxygen, respectively. S. BINNING.

Electrical conductivity of the boiled [washing] water [in the purification] of cellulose nitrate. K. ATSUKI and N. HONDA (J. Cellulose Inst., Tokyo, 1929, 5, 3-5).—In the stablilisation of nitrocellulose by repeatedly boiling with water, the electrical conductivity of the washing water gives useful indications of the degree of purification of the nitrocellulose. When the specific electrical conductivity of the wash water reaches a constant minimum value, which, however, is still higher than that of the water used, the ignition point of the nitrocellulose becomes constant, though the Abel heat test becomes higher with further boiling, presumably due to the decomposition of the nitrates of degraded celluloses. D. J. NORMAN.

See also A., Mar., 260, Behaviour of cellulose nitrate gels in polarised light (PHILLIPS).

# PATENTS.

**Explosives.** A. C. Scorr (B.P. 305,722, 11.11.27).— Crystalline oxygen carriers, *e.g.*, potassium chlorate, potassium nitrate, or sodium nitrate, are ground with asbestos, and the mixture is then impregnated with a combustible substance, *e.g.*, kerosene, nitrobenzene, or nitrotoluene. Previous to impregnation the mixture may be moistened and then pressed with subsequent granulation. S. BINNING.

Separation and after-separation of nitroglycerin, or nitroglycol, and similar explosives from the acids used in their preparation. A. SCHMID and J. MEISSNER (B.P. 284,701, 28.1.28. Ger., 5.2.27).— Nitroglycerin and nitroglycol are separated continuously from their emulsions in the waste acid by passing the emulsion into a vessel containing inclined plates between which the small drops of nitroglycerin etc. separate out easily and collect on the under-surface of a plate.

S. BINNING.

Nitration of cellulose. HERCULES POWDER Co., Assees. of M. G. MILLIKEN (B.P. 291,082, 11.4.28. U.S., 28.5.27).—Cellulose is nitrated continuously by feeding it through a vertical hopper shoot on to a horizontal worm working in an enclosed perforated cylinder. Excess of nitrating acid is poured on to the cellulose when it enters the cylinder, and more is added through the perforations. The length of the cylinder and speed of the worm are adjusted so that complete nitration has taken place by the time that the cellulose reaches the end of the cylinder. The nitrocellulose passes through a perforated head box on to a conveyor, which passes it under a roller to remove excess acid, and then through another roller through which air passes and removes more waste acid. The nitrocellulose then passes to a submerging vessel, and thence through a flume to any suitable washing plant. S. BINNING.

# XXIII.-SANITATION; WATER PURIFICATION.

Toxicity of hydrogen cyanide. J. B. ALLISON (Iowa State Coll. J. Sci., 1928, 2, 243–252).—Data for cockroaches, rice weevils, and albino rats are recorded. The equation, toxicity = concentration  $\times$  time, is not universally applicable. CHEMICAL ABSTRACTS.

Mosquito breeding [in water] and malaria in relation to the nitrogen cycle. K. B. WILLIAMSON (Bull. Entomol. Res., 1928, 18, 433–439).—The following conditions, but not the  $p_{\rm H}$  of the water, are important: efficient oxidation of the products of protein degradation in the water, the ratio of oxidised to ammoniacal nitrogen, the amount of dissolved oxygen. and the presence of micro-organisms.

CHEMICAL ABSTRACTS.

Olszewski-Keyl water-sampling apparatus. W. OLSZEWSKI (Chem.-Ztg., 1929, 53, 149).—The apparatus serves for taking a water sample for bacteriological testing from a determined depth. A falling weight causes the frame carrying the bottles to rotate on its base plate. This allows the water to enter and a 1.5 litre sample bottle is filled in 1 min. At the same time the temperature is recorded. C. IRWIN.

Determination of borate in natural waters. M. D. FOSTER (Ind. Eng. Chem. [Anal.], 1929, 1, 27-28).-1 c.c. of hydrochloric acid (4 c.c. of acid, d 1.178-1.183, to 96 c.c. of water) and 1 c.c. of turmeric solution (5 g. of powdered turmeric repeatedly washed in water, dissolved in 500 c.c. of alcohol, and filtered before use) are added to 10 c.c. of sample and slowly evaporated to dryness. A golden residue indicates absence and a pink colour presence of borate. The quantity present is found by comparison with standards which may range from 0.0 to 0.5 mg. of BO3, a difference of 0.0025 mg. of BO<sub>3</sub> showing an appreciable difference in tint, but above 0.01 mg. the standards must have a wider range, 0.01, 0.03, 0.06, 0.10, 0.25, and 0.5 mg. of BO3 being suitable. If a larger amount of sample is necessary it is more satisfactory to evaporate the whole bulk rather than instalments. D. G. HEWER.

Waters of Argyle lagoon. I. P. BLALOCK and T. G. THOMPSON. II. G. H. HITCHINGS, S. P. TODD, and T. G. THOMPSON (Pub. Puget Sound Biol. Sta., 1928, 5, 325-332, 341-353).

Carbon dioxide tension of the Fraser River and its lower tributaries and of certain tributaries of the Columbia River. E. B. POWERS and T. A. HICK-MAN (Pub. Puget Sound Biol. Sta., 1928, 5, 373-380).

Manganese in Iowa City waters. E. BARTOW and W. T. BAILEY (Proc. Iowa Acad. Sci., 1927, 34, 191-195).

See also A., Mar., 284, Distinguishing between tap water and distilled water and detection of alkali in water (FEIGL and others).

Nitrates in water. DE NARDO .- See XVI.

## PATENTS.

Method and apparatus for sterilisation. G. A. KRAUSE (B.P. 274,909, 25.7.27. Ger., 26.7.26).— Foodstuffs etc. liable to undergo bacterial decomposition are preserved by storage in or impregnation with water, salt solutions, etc. which have been oligo-dynamically activated by contact with silver or copper or their alloys. The activated water may be concentrated by evaporation, and may be stirred, *e.g.*, by agitation with bubbles of air etc. during use. L. A. COLES.

Sewage treatment by activated sludge combined with a preliminary tank treatment. K. IMHOFF (U.S.P. 1,700,722, 29.1.29. Appl., 25.8.24. Ger., 15.7.24).—The sewage is successively passed through a settling tank, an aeration tank, and a sedimentation and separation tank, and from there is returned to the settling tank. H. ROYAL-DAWSON.

Separating tarry substances from water (B.P. 279,465).—See II.