# BRITISH CHEMICAL ABSTRACTS

# are provided. Possible applications are in the reduction—feeding and from which that drop to a lower disc of observed zinc or cost and the troduction of nitrogenous—larger districter where also are nixed. On the upper and the transferred based of B.—APPLIED CHEMISTRY Shared T. W. Lors dance to I state of the property of the control of the co

# I.—GENERAL; PLANT; MACHINERY.

Some of the newer uses for silicon carbide. C. McMullen (Trans. Amer. Electrochem. Soc., 1931, 59, 199-204).—Tubes of bonded silicon carbide are now being used instead of metal tubes in heat-regenerator systems in view of their higher heat conductivity and better resistance to high temperatures. Other uses referred to include combustion chambers in "Carboradiant" furnaces and rotary metallurgical furnaces, boiler-furnace walls, ignition baffles of oil burners, resistors for heating elements and wireless apparatus, lighting arresters, and in the protection of powertransmission lines from high-voltage surges. Some of the more important properties of pure recrystallised carborundum and of bonded 80% SiC are tabulated.

H. J. T. Ellingham.

Recent engineering applications of rubber. J. R. HOOVER and F. L. HAUSHALTER (Ind. Eng. Chem., 1931, 23, 462—469).—The importance of rubber as an engineering material is illustrated by its use for bearings, for absorption of noise and vibration, as a chemically resistant covering or lining for tanks, pipes, valves, etc., for elimination of the ice hazard in aviation, and as an abrasionresistant surface layer for ball mills and conveyor belts. Reference is made to the successful development and applications of oil-resisting rubber and to the anode process for the manufacture of rubber articles and the rubber-coating of metals. D. F. Twiss.

Fire extinction.—See II. Measurement of turbidity.—See XVII.

See also A., May, 591, Thermal insulation. Fractionating columns. 592, Hydrometer. Extraction apparatus. 593, Colorimeters, spectrophotometers, and nephelometers.

# PATENTS.

Furnace. R. Walker (U.S.P. 1,781,616, 11.11.30. Appl., 14.11.28).—A furnace, suitable for burning sawdust etc. liable to be carried over to the heat-absorbing surfaces (e.g., boiler tubes), comprises primary and secondary combustion chambers separated by a depending arch and upstanding bridge; a secondary grate is also provided just beyond the bridge. Materials rising out of the primary combustion zone are deflected downwards by gas currents from ports in the arch; air is also admitted through the bridge. B. M. VENABLES.

Thermal treatment of pulverulent material. F. BARTLING (U.S.P. 1,781,659, 11.11.30. Appl., 14.2.29. Ger., 2.2.28).—Powdered material is subjected to heat treatment, e.g., destructive distillation, while

attached to a moving surface by means of static electrical charges. B. M. Venables.

Processing of furnace dust. W. J. McGurty, Assr. to Bartlett Hayward Co. (U.S.P. 1,780,833, 4.11.30. Appl., 24.4.28).—The gases from, e.g., a blast furnace are passed through a primary dust collector where the coarser dust is collected in a dry or damp state and are then passed through a wet scrubber. The fluid from the scrubber is thickened and the primary dust added to the thick pulp in a mixer, after which the sludge is dried and sintered by roasting or other means, the sinter being returned to the blast furnace.

B. M. VENABLES.

Heat removal by mercury. B. L. NEWKIRK, Assr. to Gen. Electric Co. (U.S.P. 1,780,683, 4.11.30. Appl., 4.8.27).—A mercury boiler is constructed so that the heat is removed from the tubes mostly by a mixture of liquid and vapour of mercury having a low proportion (a few %) of liquid by vol. but not by wt., which absorbs heat much better than does mercury vapour. To this end the tubes are made long and of small diameter; they may also be flattened and provided with internal spirals or other means of preventing the mercury mist from settling out until the mixture reaches the desired place of separation, viz., the vapour drum. The tubes are led tangentially into the drum so that the droplets reach the wall by centrifugal force and are caught in a pool of mercury in the bottom, while the vapour leaves B. M. VENABLES. axially.

Heat exchangers. F. B. Dehn. From York HEATING & VENTILATING CORP. (B.P. 346,197, 9.5.30).— An exchanger comprising a number of U-tubes in a casing is described. B. M. VENABLES.

Tubular heat exchangers. E. F. A. D. Beck (B.P. 345,794, 7.3.30. Belg., 9.3.29).—The tubes, preferably carrying a liquid, of an exchanger are embedded in metallic masses providing a large surface for the outer fluid, usually a gas, and leaving only narrow passages for it, the shape being such that lodgment of dust and resistance to flow are minimised. Ribs may be provided parallel to the flow. B. M. VENABLES.

Installations for carrying out strongly endothermic reactions. Soc. D'ETUDES ET RÉALISATION DITE ERÉAL, Assees. of E. CONNERADE (B.P. 345,629, 24.12.29. Belg., 27.12.28).—The reaction zone is placed on the axis within a gas producer in which one of the reacting gases is produced; the gas passes upwards through the producer, downwards through passages in the dividing wall, and upwards through the reaction zone. Other, external, condensing and heat-exchanging devices

are provided. Possible applications are in the reduction of roasted zinc ores and for the production of nitrogenous compounds.

B. M. Venables.

Refrigeration. W. H. CARRIER, ASST. to CARRIER ENG. CORP. (U.S.P. 1,781,051, 11.11.30. Appl., 15.10.26).

—Methylene chloride, preferably refined so that it is free from fractions of different b.p., is utilised at a pressure not much above 1 atm. (preferably under partial vacuum). It is sprayed so as to form a film over unsubmerged evaporating surfaces and the vapour is drawn off, condensed, and re-used together with the unvaporised liquid.

B. M. Venables.

Centrifugal mills. E. Barthelmess (B.P. 346,011, 1.1.30. Ger., 2.5.29).—A centrifugal cup or other rotor which may be unbalanced is permitted to rotate about its centre of gravity by giving the upper bearing (that nearest the cup) restrained freedom in all horizontal directions, by permitting spherical movement of the lower bearing, and providing a flexible coupling to the motor.

B. M. Venables.

Apparatus for grinding and sifting. E. Barthelmess (B.P. 345,996, 30.12.29. Ger., 11.10.29).—In a machine where the grinding is effected by ascending and rotating currents of air produced by a centrifugal rotor, means are provided for adjusting the annular air exit and hence the size of the finished material.

B. M. VENABLES.

Multiple rubbing, grinding, and mixing rolling mill for chocolate, soap, paint, etc. Simon A.-G. (B.P. 346,166, 9.4.30. Ger., 10.4.29).—The mill comprises a number of rolls in series rotating alternately at high and low speeds, but with a progressive increase in speed in the direction of travel of the material, e.g., the odd numbers may rotate at 144—160 and the even at 72—80 r.p.m.

B. M. Venables.

Dry separation of solid materials. A. T. Masterman (B.P. 345,997, 30.12.29).—A device of the kind in which heavy particles fall farther away from an air jet than light particles is supplemented by devices intended to effect separation of the middling containing large light and small heavy particles. The falling particles are caught and rendered nearly stationary by a baffle and then allowed to slide down inclined surfaces which may be roughened to any desired degree, the heavy particles acquiring more momentum being collected in side compartments and the lighter in central compartments.

B. M. Venables.

Separation of granular, powdered, and like materials. W. D. Williams (B.P. 346,002, 1.1.30).— The apparatus is suitable for estimating the amount of "flour" in such materials as Portland cement by elutriation with air. It comprises a \(\Omega\)-tube with suitable branches for connexion to gauges and a suction device. The limb to which the sample is fed is provided with bulges to maintain the air current turbulent.

B. M. VENABLES.

Disc feeder and mixer. L. C. Bonnot, Assr. to Bonnot Co. (U.S.P. 1,781,097, 11.11.30. Appl., 22.7.29).

—A number of constituents are delivered from separate hoppers through separate spouts with adjustable outlets on to a single rotating disc which effects the

feeding and from which they drop to a lower disc of larger diameter where they are mixed. On the upper disc a scraper is provided for each constituent, but on the lower a single scraper suffices. B. M. VENABLES.

Mixing machine [for dough etc.]. W. F. Dehuff (U.S.P. 1,781,321, 11.11.30. Appl., 4.5.29).—A variablespeed gear of the expanding-V pulley type is applied to a mixer. B. M. Venables.

Worm presses for treatment of moist materials. F. Krupp Grusonwerk A.-G. (B.P. 345,820, 29.3.30. Ger., 13.5.29).—A method of restricting, in an adjustable manner, the annular outlet of an archimedean press is described.

B. M. Venables.

Apparatus for treating [laminated] materials under pressure [and while under vacuum]. H. Griffiths (B.P. 345,772, 18.2. and 30.10.30).—Laminated materials such as safety glass are interleaved with flexible chambers in a press which is run into a vacuum chamber. The squeezing is produced by admission to the flexible chambers of a pressure fluid which may, if desired, be hot.

B. M. Venables.

Press for extracting liquids. H. G. Schwarz (U.S.P. 1,781,250, 11.11.30. Appl., 5.4.27).—An archimedean press having perforations for exit of liquid in both the cylindrical casing and the conical hollow shaft of the worm is described. The annular outlet for solids is obstructed more or less by an adjustable tapering collar. Applications cited are the extraction of oils from fish and fish residues, and of juices from various fruits.

B. M. Venables.

Filter presses. Soc. CHEM. IND. IN BASLE (B.P. 346,191, 1.5.30. Ger., 1.5.29).—A filter press is provided with hot plates in each frame on the line of division of the cake.

B. M. VENABLES.

Filtering apparatus. Soc. EN NOM COLLECTIF E. & M. LAMORT FILS (B.P. 345,761, 12.2.30. Fr., 22.2.29).— A filter for large quantities of liquid of the type which is cleaned by reverse flow is described. The filter is in the form of a cylindrical shell which is smaller than the surrounding casing. At opposite ends of a diameter fins, attached to the filter, make fluid-tight joints with the casing; the inlet and dirt-outlet ports are diametrically opposite in the casing and the outlet for filtrate is axial. In normal filtering the fins are placed opposite the middle of the opposing ports, the dirt outlet being closed by a valve; for cleansing purposes the axial outlet is closed, the dirt outlet opened, and the filter rotated so that the fins cause the fluid to enter at one side and leave by the other, carrying the dirt from one B. M. VENABLES.

Filter bed. A. LENDERINK (U.S.P. 1,780,791, 4.11.30. Appl., 8.3.29).—A form of construction of a bed for a filter, but not for the filter medium, suitable for fluids such as sewage, is described.

B. M. VENABLES.

Treating the filter cake produced from continuous filters. B. H. Ryley (U.S.P. 1,781,652, 11.11.30. Appl., 4.5.28).—Each leaf is jarred by a hammer in order to compact the cake. B. M. Venables.

Centrifugal machines for separating milk or the like. Ecremeuses Melotte, Soc. Anon. (B.P.

345,834, 11.4.30. Belg., 11.4.29).—The supply tank and casing for the gearing of a centrifugal separator are cast in one piece with rounded corners, the gearing being placed in one corner of the tank. Means are provided for rapidly removing the casing of the bowl.

B. M. VENABLES.

Centrifugal separator bowls. ARTIEB. SEPARATOR (B.P. 345,849, 23.4.30. Swed., 24.4.29. Addn. to B.P. 316,181; B., 1930, 307).—A centrifuge constructed on the principles described in the prior patent is provided with outlets for the products at opposite ends of the bowl, at least one of the outlets being adjustable, e.g., by a removable weir-plate. B. M. Venables.

Evaporators. G. & J. Weir, Ltd., and J. G. Weir (B.P. 345,810, 21.3.30).—The spray-separating baffle in the vapour space of an evaporator is provided with a gutter and drainpipe to lead the collected liquid to an innocuous place, and in combination with this is placed, below the baffle, a perforated diaphragm, which is substantially horizontal, but is higher in the middle.

B. M. VENABLES.

Steam distillation. E. H. Leslie and E. M. Baker (U.S.P. 1,780,977, 11.11.30. Appl., 11.5.23).—The apparatus comprises a fractionating tower with condenser for total or reflux condensation at the top, filling in the tower, means for admitting the original hot liquid (e.g., petroleum oil) to any desired stage, and means for drawing off separate liquid fractions from a number of stages. Below the tower are means for the flash-vaporisation of the residual liquid, which is readmitted as vapour between two layers of filling, steam being admitted into the lowest pool of all.

B. M. VENABLES.

Apparatus for separating liquids into constituents of low b.p. and those of high b.p. by distillation and rectification. A. Messer (U.S.P. 1,780,563, 4.11.30. Appl., 9.11.25. Ger., 15.11.24).—In a bubbling tower the downflow of liquid is arranged alternately at the centre and circumference of the trays, and the bubbling caps are distributed at a uniform distance apart.

B. M. Venables.

Fractionation apparatus. W. Z. FRIEND, Assr. to BALTIMORE GAS ENG. CORP. (U.S.P. 1,780,818, 4.11.30. Appl., 17.9.28).—A fractionating tower is provided with a manhole on the axis of the casing and with corresponding detachable plates in the centre of each tray so that access may be had for cleaning.

B. M. VENABLES.

Atomisation of liquids. J. A. Reavell (B.P. 345,714, 13.1.30).—The liquid is admitted on to a rotary atomising disc by means of an annular passage surrounding the driving shaft and, if desired, supporting the lower bearing for the latter. The disc is corrugated and the feed passage may be Venturi-shaped; the initial inlets to the disc may be tangential to give the liquid a preliminary spin in the direction of travel of the disc.

B. M. Venables.

Liquid and gas contact apparatus. F. H. WAGNER, Assr. to BARTLETT HAYWARD Co. (U.S.P. 1,780,255, 4.11.30. Appl., 8.3.28).—The apparatus comprises a tower having a number of perforated diaphragms over which liquid is spread by discs (on a common shaft)

running in apertures in the centre of the diaphragms. The pressure of the upflowing gas should be sufficient to prevent any substantial downflow of liquid through the perforations, annular gutters and siphon pipes being provided for that purpose. B. M. Venables.

[Laboratory] gas-producing apparatus. G. R. H. Ford (B.P. 346,421, 31.12.29).—For provision of a supply of gas, such as hydrogen sulphide, a generator and a reservoir are mounted at opposite ends of a platform which can be tilted at any desired angle. The principle may be adapted to gas production by electrolysis.

R. H. GRIFFITH.

Liquefaction of gases. L. B. Strong, Assr. to F. G. Campbell (U.S.P. 1,780,250, 4.11.30. Appl., 27.3.30). —The gas to be liquefied, e.g., air, is compressed, cooled to ordinary temperature, and expanded adiabatically in a heat-insulated engine, e.g., a turbine, the work from which may be used in the original compression. The high pressure is so chosen that the entropy of the gas at high pressure and ordinary temperature is equal to or less than its entropy at the lower pressure after expansion and its b.p. at that pressure, under which conditions the expanded gas will be in the form of saturated vapour. Another gas of lower critical temperature, e.g., hydrogen, is compressed, cooled, and isothermally expanded in a series of turbines or engines alternating with heatexchanging coils upon which the saturated air is condensed, the latent heat of the air now in the hydrogen being recovered by heat exchange between the in- and out-going streams of hydrogen, which is wholly returned to its compressor. The process may be applied to the separation of helium from natural gas.

B. M. VENABLES.

[Controlling the flow of gas through] gas-analysis apparatus. Aktiebolaget Carba, Assees. of Platen-Munters Refrigerating System Aktiebolag (B.P. 345,665, 14.12.29. Swed., 17.12.28).—The actual analysing apparatus is placed in a shorter by-pass of a longer one, the latter permitting an adequate current of gas to be brought close to the apparatus irrespective of the small quantity actually analysed.

B. M. VENABLES.

Viscosimeter. E. M. Symmes, Assr. to Hercules Powder Co. (U.S.P. 1,780,952, 11.11.30. Appl., 27.9.27).

—A viscosimeter of the falling-ball type is adapted to opaque liquids by the use of oscillating radio-valve circuits to detect the fall of the ball. B. M. Venables.

[Wax-resin balls for] determining the sp. gr. of liquids [e.g., battery acid]. Britannia Batteries, Ltd. (B.P. 345,846, 17.4.30. Ger., 23.4.29).—It is stated that gas bubbles do not adhere to density-indicating floats when they are made of artificial wax and resin. Claim is made for compositions containing equal parts of artificial resin and artificial wax with colouring matter and a variable amount of barium sulphate according to the gravity desired. B. M. Venables.

Friction material for brakes, clutches, etc. A. E. White. From Union Asbestos & Rubber Co. (B.P. 345,991, 24.12.29).—The lining comprises an asbestos base with wires or strips of lead or lead alloy embodied in it and a resinous binder. Besides its known function as a lubricant the lead seems to act as

a chemical preventative of the formation of abrasive materials such as slags or carbides ordinarily caused by the friction.

B. M. VENABLES.

Apparatus for producing artificial fog. Hanseatische Apparatebau-Ges. vorm. L. von Bremen & Co. M.B.H. (B.P. 345,215, 17.12.29. Ger., 17.12.28).— An apparatus for the production of acid fog by the method described in B.P. 298,980 (B., 1930, 127) is provided with automatic valves for the emission of spray and admission of reagent controlled by corrugated tube diaphragms, similar devices being used instead of stuffing boxes.

B. M. Venables.

Dialysing membranes.—See V. Furnaces for sulphate production etc.—See VII. Inextensible belting.—See XV.

# II.—FUEL; GAS; TAR; MINERAL OILS.

Composition of coal. Soluble constituents of coal and their degree of coalification. C. COCKRAM and R. V. WHEELER (J.C.S., 1931, 854-860).—Samples of coal from the Pittsburgh seam (80.5-89% C) and the Barnsley seam (80.6-84.5% C) have been subjected to solvent analysis (cf. B., 1927, 401). Whilst the Pittsburgh coals exhibit no progressive variation in the proportion of the complete \( \gamma \) fraction, there is a gradual increase in the ratio of the combined \u03c32 and  $\gamma_3$  fractions (resinic compounds) to the  $\gamma_1$  fraction (free hydrocarbons) with increasing carbon content. Taking the "Festbitumen" and "Ölbitumen" of Fischer, Broche, and Strauch (B., 1925, 233) as corresponding with the combined  $\gamma_2$  and  $\gamma_3$  fractions and the  $\gamma_1$  fraction, respectively, the relative proportions of these in the German coals vary with the carbon content (" rank") in the opposite direction to that observed for the Pittsburgh coals. In the Barnsley coals there appears to be no relationship between their "rank" and their content of free hydrocarbons. The necessity for caution when attempting to compare coals that have been formed under widely different conditions is emphasised.

A. B. Manning. Unit coal studies on some Virginia coals. F. H. FISH and J. A. ADDLESTONE (Ind. Eng. Chem. [Anal.], 1931, 3, 155-158).-The unit B.Th.U. values for the Virginia coals under investigation are in best agreement when calculated by the Fieldner modification of the Parr formula (Trans. Amer. Inst. Min. Met. Eng., 1930, 78, 597). When, however, a correction is applied for the carbon dioxide, which is present in considerable amount in some cases, the original Parr formula gives the better agreement. For 28 samples taken from the same seam the average deviation from the mean is  $\pm 75$  B.Th.U. by the Parr formula and ±86 B.Th.U. by the Fieldner modification. For other coals studied, the average difference between the untreated, float, and sink portions of each coal is 82 B.Th.U. (Parr) and 96 B.Th.U. (Field-E. S. HEDGES.

Determination of the hygroscopic properties of coal. R. Vondráček and L. Mosendz (Coll. Czech. Chem. Comm., 1931, 3, 81—92).—The static and dynamic methods of determining the hygroscopicity of coal are described. By consideration of previous results obtained by the static method with different types of

coal the equation  $A^n - (A + a)^n = nkp$ , where A is the initial humidity and a the humidity at vapour pressure p, and n and k are constants characteristic of the type of coal, has been derived. Experiments on the rate of dehydration of lignite at the ordinary temperature over sulphuric acid in stationary and moving air and in vacuo are described. The results accord with the theoretical equation  $(H - x)^{-m} - H^{-m} = mkt$ , where H is the total moisture content, x the loss during time t, and m and k are constants. The value of m is independent of the method of desiccation and increases from about  $1\cdot 1$  at  $25^\circ$  to  $1\cdot 9$  at  $60^\circ$ , falling again to  $0\cdot 75$  at  $100^\circ$ ; k increases continuously with rise of temperature and varies with the method of desiccation.

Physics of coal carbonisation. S. P. BURKE, T. E. W. SCHUMANN, and V. F. PARRY (Fuel, 1931, 10, 148—171).—The temperature distributions in (a)] a flat slab, (b) a cylinder, (c) a cylindrical annulus, and (d) a sphere, the outer surfaces of which are suddenly heated to, and maintained at, a fixed higher temperature, have been calculated as functions of the time and the thermal diffusivity of the material. The times required to attain a definite distribution of temperature in two geometrically similar bodies are proportional to the squares of their corresponding linear dimensions. It is deduced theoretically that this "law of squares" still holds even when the thermal constants vary with the temperature, and when thermal reactions involving the liberation or absorption of heat occur in the material, provided that such reactions may be regarded as taking place instantaneously. Experiments on crushed coal and coke in cylindrical retorts of different sizes have shown that the laws of conduction for crushed materials deviate by less than 2-3% from those for solid materials. The thermal diffusivity of crushed coal (approx. 16mesh) was 0.010 sq. in./min. (15-340°), and of monolithic coke 0.037 sq. in./min. (15-540°). The initial part of the curve showing the variation of the temperature at the centre of a charge of coal with time is modified by the presence of moisture, heat being transferred through the charge by convection of water vapour as well as by conduction. Endothermic reactions probably occur during the decomposition of coal in the plastic range, and both endothermic and exothermic reactions subsequently occur on heating the coke. These reactions alter the shape of the heating curve and thus affect to some degree the time of carbonisation. Despite the effect of convection and the influence of the time element on the chemical reactions occurring in coal, the "law of squares" is found to hold good for the coking process. If F is the carbonising time (in min.) for a cylindrical retort of unit radius for any particular wall temperature, then the carbonising time for a cylindrical retort of radius R (in.) is  $FR^2$ , that for a flat retort of semi-width R is  $2FR^2$ , and that for a cylindrical annular retort is  $2FR^2$ , where 2R is the difference of the outer and inner radii. The coking time of Pittsburgh coal as a function of the retort wall temperature, initial temperature of the coal, and dimensions of the retort has been studied, experiments having been carried out in cylindrical retorts ranging from < 2 in. to > 13 in. in diam. The curve showing the coking time as a function of the temperature lies between the theoretical curves for coal and coke, respectively, approaching the former asymptotically at the lower temperatures and the latter at the higher temperatures.

A. B. Manning.

Process of combustion of powdered coal. A. Greel (Compt. rend., 1931, 192, 567—569).—The ignition of coal etc. in presence of a trace of moisture is due to ignition of hydrogen evolved through the action of heat on the carbon and the moisture. An outline of the subsequent process of combustion follows, and is illustrated by a series of curves based on Fink's results.

C. A. Silberrad.

Behaviour of carbonised fuels in an open fire grate. G. Milner, J. H. Dyde, and H. J. Hodsman-(J.S.C.I., 1931, 50, 113—120 T).—The laboratory properties of carbonised fuels have been correlated with their behaviour in an open, vertical-fronted fire grate. The radiation depends jointly on the area radiating,  $A_{\perp}$ its emissivity, e, and its temperature, T, i.e., on the product  $eAT^4$ . The area factor depends on grate design and geometrical configuration of the fuel. Dense compact fuels, tending to pack closely, do not give so high an output of heat as spongy, bulky fuels of irregular form. The emissivity factor is lessened by the presence of flame, resulting either from the combustion of the volatile matter of the fuel or the reduction of carbon dioxide in the upper part of the fire. The latter also lowers the temperature of the fuel bed, thus tending to reduce the output of heat. A similar chilling effect is produced by moisture, particularly in the early stages of lighting. Ash reduces the rate of output of heat, but has only a slight influence on thermal efficiency. Ignition temperatures serve as a useful guide as to ease of lighting. Under the experimental conditions the radiant efficiencies lie between 20% and 37%, but when the fire reaches a steady state about 40% may be emitted. It seems possible to predict these figures approximately from a knowledge of the reactivity and bulk density of a coke. It is concluded that the desirable properties of a solid fuel are sometimes mutually incompatible, and therefore an ideal fuel cannot be realised.

"Melting" of coal during coke formation. E. Audibert (Fuel, 1931, 10, 189—190).—A reply to Davies and Wheeler (B., 1931, 428). The question as to whether coal melts completely or only partly during coke formation is regarded as still open.

A. B. Manning.
Application of antioxygenic action to fire extinction: negative catalysis of the ignition of coal.
C. Dufraisse and R. Horclois (Compt. rend., 1931, 192, 564—566).—Addition of 5% of carbon tetrachloride vapour or of 1% of phosphoryl chloride vapour to air used for combustion of charcoal has practically the same effect as total deprivation of air. Several other halogencontaining compounds, diethylamine, and sulphur dioxide act more or less similarly. It is concluded that such substances act catalytically. C. A. Silberrad.

Activity of incrusted coke and the production of malleable pig iron low in carbon. E. Pivovarsky [with F. Kramer] (Giesserei, 1930, 17, 1149—1152; Chem. Zentr., 1931, i, 843).—The reactivity of coke towards carbon dioxide at 900—1100° is increased by

treatment with milk of lime. The production in the cupola furnace of iron containing less than 1.6% C is described.

A. A. Eldridge.

Sodium thiocyanate as a medium for drying [coal] gas. K. Keller and H. Nord (Ber. Ges. Kohlentech., 1931, 3, 460—464).—The procedure of gas-drying with sodium thiocyanate is similar to that of methods using other hygroscopic salts, but the material is one which can be manufactured at a cokeoven plant. If gas saturated with water vapour at 20° is washed at this temperature with a saturated solution containing 508 g. NaCNS per litre, the dewpoint falls to 7.3°. If gas-washing is carried on at 25°, using the solution saturated at this temperature, the dewpoint of the dried gas is 8.4°. A series of determinations of dewpoints, using more dilute solutions, is given.

C. Irwin.

Utility of solutions of nickel and copper salts for the removal of hydrogen cyanide from cokeoven gas. W. Gluud and W. Riese (Ber. Ges. Kohlentech., 1931, 3, 437—451).—It was desired to ascertain whether hydrogen cyanide could be separated from coke-oven gas efficiently as complex copper or nickel cyanides and the cyanide conveniently recovered. The reaction is complicated by the simultaneous formation of thiocyanate, depending on the oxygen content of the gas and on other factors. The principal reactions of crude coke-oven gas with a solution of a nickel salt are the initial formation of nickel sulphide followed by  $NiS + 2NH_4CN = (NH_4)_2S + Ni(CN)_2$ ;  $Ni(CN)_2 + 2(NH_4)CN = (NH_4)_2[Ni(CN)_4]$ . This last salt is not decomposed by carbon dioxide or hydrogen sulphide. The simultaneous formation of thiocyanate is greatly promoted by ammonia. It is therefore necessary to use gas free from ammonia and to replace this by another alkali. The soda or potash double salts can be brought up to concentrations suitable for crystallisation without ill effect on the cyanide removal. There are several methods of decomposing the double salts, but none appears likely to be economical, and this is the principal objection to the process. A double copper salt K<sub>3</sub>[Cu(CN)<sub>4</sub>] can be prepared in a similar way, but is not so stable to carbon dioxide as the nickel salt. With increasing concentration the washing efficiency on coke-oven gas deteriorates and its use does not appear practical. The recovery of hydrogen cyanide from the copper salt offers the same difficulties as with nickel.

Production of water-gas in horizontal chamber ovens. A. Steding (Gas- u. Wasserfach, 1931, 74, 357—361; cf. Gill, B., 1930, 848).—Three series of experiments are described in which steam was passed through the charge in a suitably modified Otto coke-oven setting. The steam was introduced (a) through the floor of the oven, (b) through the walls, and (c) through tubes projecting into the charge from above, respectively. With each of these arrangements a good-quality water-gas could be produced. Moreover, the introduction of steam lowered the nitrogen content of the gas by diminishing the in-leakage of flue gases. The temperature of the coke when discharged was considerably lower with steaming than without.

A. B. Manning.

Kinetics of the thermal decomposition of methane. T. S. Wheeler (Fuel, 1931, 10, 175—181. Cf. Bone and Coward, B., 1908, 886; Wheeler and Wood, B., 1929, 88; 1931, 193; Holliday and Exell, A., 1929, 773).—The available experimental data have been critically examined. They indicate that the decomposition of methane in contact with silica, porcelain, and chrome iron is heterogeneous, and is retarded by the hydrogen produced. The experimental results can be reproduced by an equation which indicates that the retardation is a function of both the hydrogen and the methane pressures.

A. B. Manning.

Nature and properties of coal tar. E. V. Evans and H. Pickard (S. Metropolitan Gas Co., Chem. Dept., 1931, 41 pp.).—The consistency of a tar (seconds Hutchinson or time of flow in the Redwood No. 2 viscosimeter) is related to the temperature (° F.) by the formula  $CT^n = a$ , where n and a are constants. The addition of naphthalene to a "synthetic" tar markedly lowered the consistency, the addition of 5%, e.g., changing the values of n and a from 5.7 and  $712 \times$  $10^{11}$  to  $5 \cdot 1$  and  $213 \times 10^{10}$ , respectively. A similar lowering of the consistency was produced by the addition of naphthylamine, benzene, or aniline, whereas the effect of adding naphthol or phenol was much less marked. By plotting log temperature (° F.) against log consistency for these mixtures, and for mixtures of pitch with the tar, a series of straight lines was obtained which when produced passed approximately through a common point (focus). A viscosimeter suitable for determining the consistency of pitch at raised temperatures has been designed, based on the measurement of the time of flow of the viscous liquid down an inclined cylinder. The logarithmic consistency curves of mixtures of tar and pitch obtained by using this instrument again took the form of a series of straight lines emanating from a common focus. Asphaltic bitumens differed from coal-tar products in the slope of the corresponding lines, the temperature coefficient of consistency being less for bitumens than for tar products, and in the position of the focus. Addition of a filler, e.g., lampblack, to the tar or pitch increased the consistency, the new log consistency curve lying parallel to the original and passing through a different focus. Removal of the "free carbon" insoluble in pyridine had a negligible effect on the consistency of a pitch, whereas removal of the "free carbon" soluble in pyridine but insoluble in benzene produced a pronounced lowering in the consistency. Finely-divided coal has been shown to be a suitable filler for modifying the consistency of a tar (cf. B.P. 316,897; B., 1929, 803). The consistency of pitch at ordinary temperatures has been measured by means of a modified penetrometer, in which the needle is replaced by a rod having a rounded end. Within practical limits of accuracy, pitch behaved as a true fluid at these temperatures, the velocity-load curves being straight lines passing through the origin. Asphaltic bitumen, on the other hand, exhibited anomalous consistency effects in its velocity-load relationship. Dispersions of coal in tar exhibited corresponding anomalous consistency effects, and in this respect, therefore, the physical properties of the asphaltic bitumen have A. B. MANNING. been reproduced in a tar.

Low-temperature tars. G. T. Morgan (Fuel, 1931, 10, 183—189; cf. Morgan, Pratt, and Ross, B., 1929, 156).—The methods and results of examining some low-temperature tars are described. The presence of the following substances in the fractions and/or the aqueous liquor of a "primary" tar produced by the carbonisation of Dalton Main coal in a Pehrson retort at 450° has been demonstrated: benzene, toluene, m- and p-xylenes, naphthalene, 2-methylnaphthalene, phenol, o- and p-cresols, pyrocatechol, resorcinol, and quinol. No pyridine was found. The "primary" tar had a lower sp. gr. (1·008) than that of a tar produced from the same coal at 625° (1·02), and contained more neutral oil, less bases, less phenols, and less paraffin wax than the latter.

A. B. Manning.

Phenol content of tars and oils derived from coal. J. L. Wiltshire (J.S.C.I., 1931, 50, 125—128 t).

—The methods available for the determination of phenol in tars are discussed, and one of them is shown to give satisfactory results without undue elaboration. This method is applied to a series of products derived from coal: low-temperature tar from Fuel Research Board vertical retorts, 1.0% of phenol (Warwick coal) and 1.5% of phenol (Shafton coal); hydrogenated Shafton low-temperature tar, 4.9% of phenol; liquid product from hydrogenation of Shafton coal, 2.6% of phenol.

Ageing phenomena in creosote oils. W. Grund-Mann (Chem.-Ztg., 1931, 55, 267).—The viscosities of creosote oils and of their alcoholic solutions increase on keeping, the change being accelerated by repeated cooling at —30° to —40°. Similar changes occur also in their electrical conductivities, so that creosotes of different age and from different distillations rarely have identical physical properties. F. R. Ennos.

Chemical aspect of [oil-well] drilling muds. A. Duckham (J. Inst. Petroleum Tech., 1931, 17, 153— 182).—The mud, used as a lubricant in rotary drilling and as a vehicle for removing rock cuttings, may either be prepared from clays found near the well or imported and may require "doctoring" to give it the required weight, viscosity, and colloidity. A heavy mud may be necessary to prevent "blow outs," and for this purpose barytes or hæmatite may be added, weights up to 120 lb./cub. ft. being occasionally used, although 80 lb./ cub. ft. is the more usual quantity. A chart which enables the amount of barytes required to be added to a given mud to produce the desired weight is given. Viscosity is determined in the Stormer viscosimeter by rotating a cylinder in the mud. No ready means of determining colloidity has been elaborated. Muds are freed from cuttings by straining or by such apparatus as the Dorr classifier. Colloidity is controlled by the addition of such materials as bentonite, "aquagel," or soda. The addition of soda gives a suitable viscosity to heavy muds, but excess causes "ropiness" and separation. Before adding soda the alkalinity of the mud and its water content should be determined. The ingress of salt water may spoil a mud. Mud which has become gassed" may sometimes be freed from gas by pumping it against a plate. T. A. SMITH.

Desulphurisation of shale oils. T. Koern (Tehn. Ajakiri, 1930, 9, 166—168; Chem. Zentr., 1931, i, 1046).—When passed over a heated catalyst (bog-iron ore, or various metallic oxides deposited on carriers as nitrates and subsequently reduced with hydrogen or water-gas) the hydrocarbon (1.06% S) gave a distillate of which the first fractions were low in sulphur. The catalyst was rapidly inactivated and regeneration was difficult. When the oil is heated under pressure with hydrogen in presence of the powdered catalyst the sulphur content may be reduced to 0.06%. Regeneration is easily effected.

A. A. Eldridge.

Hydrogenation and desulphurisation of Esthonian shale oils. J. Hüsse (Tehn. Ajakiri, 1930, 9, 162—165; Chem. Zentr., 1931, i, 1046).—Desulphurisation is effected in the liquid phase by heating under pressure with hydrogen in presence of a catalyst (bogiron ore, minette, or nickel-chromium). The oil so treated gives lower values for density, viscosity, phenol content, and solubility in sulphuric acid.

A. A. ELDRIDGE.

Reactions of olefines with sulphuric acid. W. R. Ormandy and E. C. Craven (J. Inst. Petroleum Tech., 1931, 17, 185—187).—The reaction of a number of olefines in "aromatic-free" heavy petroleum spirit (1 pt. of olefine to 9 pts. of spirit) with sulphuric acid of various concentrations (81—99·5%) has been determined, the bromine values (Hanus) before and after treatment being determined. The olefines become resistant to sulphuric acid as their mol. wt. increases, thus agreeing with the behaviour of these substances towards permanganate as stated by Howes and Nash (B., 1930, 596). The difference in behaviour, however, is marked only at the lower concentrations (up to 90%) of acid. At higher concentrations the bromine values are reduced to very low figures with all the olefines examined.

T. A. Smith.

Effect of light on determination of ethylene. J. L. Oberseider and J. H. Boyd, Jun. (Ind. Eng. Chem. [Anal.], 1931, 3, 123).—Inaccuracies occur in the determination of ethylene in gaseous mixtures of paraffin and olefine hydrocarbons separated from cracked oils, when the absorption in bromine water is carried out in daylight or artificial light. Correct results are obtained when a blackened absorption pipette is used. E. S. Hedges.

Removal of hydrogen sulphide and hydrogen cyanide from benzol. K. Keller (Ber. Ges. Kohlentech., 1931, 3, 429-436).—The claim in G.P. 76,348, that benzol used for the dephenolisation of ammoniacal liquor and containing 1% of phenol was quickly freed from hydrogen sulphide by keeping even in the dark and in absence of air, was investigated. It was found that the presence of ammonia rather than that of phenol is the most likely explanation. Benzol either pure or crude is quickly freed from hydrogen sulphide by air treatment in presence of ammonia and moisture, the sulphide being oxidised to thiosulphate. At the same time hydrogen cyanide is also removed. Such a treatment could usefully be given to benzol intended for phenol extraction before use, in a closed washer with agitation, and troublesome corrosion thereby be avoided. Crude benzol containing 1.5 g. H<sub>2</sub>S and 0.3 g. HCN per litre will require 35 litres of 5% ammonia solution per in.<sup>3</sup> The ammonia can be recovered. Experimental work is reported in detail. C. IRWIN.

Silicon carbide. Applications of rubber.—See I. Ammonium sulphate crystals. Conversion of cyanide into ferricyanide.—See VII. Tar oils for timber.—See IX. Fruit tree carbolineums.—See XVI.

See also A., May, 574, Combustion of pulverised fuel. 576, Reaction  $\rm H_2 + \rm CO_2 \rightarrow \rm CO + \rm H_2O.$  630, Pyridine bases from tar. 638, Detection of carbon disulphide.

PATENTS.

Treatment of [anthracite] coal. E. V. Collins, Jun., Assr. to Delaware, Lackawanna, & Western Coal Co. (U.S.P. 1,781,102—3, 11.11.30. Appl., 14.2.30).—(a) The appearance of anthracite which deteriorates on storage is restored by treatment with an aqueous solution containing 20% of ammonium persulphate; this may be applied by spraying or by agitating the coal in the liquid. (b) The same result is obtained by washing anthracite with a solution containing 1.5% of titanium trichloride in 10% hydrochloric acid.

R. H. Griffith.

Heating of low-temperature carbonisation retorts. J. Y. Johnson. From I. G. Farbenind. A.-G. (B.P. 346,533, 28.2.30).—A plant for the low-temperature distillation of coal, shale, etc. is built into a boiler setting so that heating gases pass first over one part of the boiler, then over the surface of the carbonisation retort, and finally over another portion of the boiler. The position of the retort is chosen so that it is heated to any desired temperature. High thermal efficiency is claimed.

R. H. Griffith.

Treatment of coal and other carbonaceous material. A. E. White. From Thermolized Coal Corp. (B.P. 346,383, 7.12.29).—Crushed coal, or similar carbonaceous material, is treated with a stream of waste gas, and then subjected to low-temperature carbonisation. The resulting semi-coke is cooled in an inert atmosphere and finely pulverised so as to be suitable for furnace firing. The powdered material thus obtained is preferable to coal dust in that it is less liable to spontaneous ignition and can be burned with a shorter and more uniform flame. [Stat. ref.]

R. H. GRIFFITH.

Distillation of coals. W. E. TRENT, Assr. to TRENT
PROCESS CORP. (U.S.P. 1,781,614, 11.11.30. Appl.,
21.12.25).—Carbonisation of finely-divided coal is effected
by the combustion of part of the material. The process
is carried out in a stream of air which moves at such a
speed that a selected proportion of the carbonised
particles is carried away together with gases and
vapours which are simultaneously produced. The
calorific value of the gas-solid mixture is appreciably
higher than that of the gas alone, and provides a useful
fuel from coals very low in volatile matter.

R. H. GRIFFITH.

Production of coke. A. H. White, Assr. to Regents of the Univ. of Michigan (U.S.P. 1,782,556, 25.11.30. Appl., 26.10.25).—Broken coal is allowed to fall freely

through an empty vertical retort, and is carbonised by heat radiated from the walls, which are at 700° or above. The time of treatment is very short, but the coke obtained has appreciably less volatile matter than the original coal, and can be pulverised to give an excellent dust fuel. The properties of the coke depend on the size of the raw material, the length of the retort, and the temperature to which it is heated; particles too large to pass 12-mesh are not suitable for treatment. Steam or hydrogen may be added to the retort, and the gas produced is of very uniform quality.

R. H. GRIFFITH. Improving explosive mixtures for internalcombustion engines etc. A. Ducloux (U.S.P. 1,777,554, 7.10.30. Appl., 3.5.27. Fr., 7.5.26).—A carburetted fuel is passed through a confined space containing a heate mixture of approx. 10% of a mineral containing thorium, a relatively small percentage of an oxide of a radioactive metal, and a relatively large percentage of an inert substance. The heating may be done conveniently by the exhaust gases. A preferred mixture consists of monazite or thorite 10%, thoria 2%, asbestos 40%, and tale or chlorite (which may include powdered iron, nickel, or manganese) 48%.

H. S. GARLICK. Gas producer. H. F. Smith, Assr. to Gas Research Co. (U.S.P. 1,781,767, 18.11.30. Appl., 10.12.23).—In a small gas producer (< 1 ft. in diam.), special precautions are introduced to prevent heat losses from the relatively large surface available. Moist air is drawn into the system in such a way that it passes three times over cylindrical jackets which surround the fuel bed. The grate can be reached only by such indirect passage, so that radiant heat lost from the generator is largely returned to the apparatus. R. H. GRIFFITH.

Gas producer. A. K. Bradley, Assr. to Morgan Construction Co. (U.S.P. 1,782,677, 25.11.30. Appl., 7.6.28).—The shell of a gas producer is mounted to rotate at a constant speed, the vessel being closed by a fixed water-sealed cover and by a water-sealed grate which rotates independently of the producer. The speed of the lower part is variable, and within it is an ash plough which can either be fixed so as to move with the shell, or be carried round solely by the weight R. H. GRIFFITH. of ashes and fuel pressing on it.

Generation of combustible gas and coke. H. F. SMITH, Assr. to Gas Research Co. (U.S.P. 1,781,766, 18.11.30. Appl., 9.9.22).—A vertical vessel for the carbonisation of coal narrows rapidly below the coal inlet, and then widens to a grate, the centre of which rotates. Below the fire bars is a gastight chamber (where ash and clinker collect) provided with a steam inlet to a hollow standard carrying the rotary section. At the narrowest portion of the retort, and also towards the base, are numerous openings from which gas can pass to, or air be introduced from, a heat exchanger. Producer gas, water-gas, and low-temperature coal gas can be obtained by different methods of operation, and the required mixture is collected from a common top offtake. R. H. GRIFFITH.

Water-gas generators. D. Tyrer, and Imperial CHEM. INDUSTRIES, LTD. (B.P. 345,411, 3.2.30).—Air for the "blow" period is supplied by tuyères in different amounts and at different levels to a deep bed of fuel contained in the generator, so that the maximum air supply is at the point nearest to that of the exit of the "blow" gases (which is at the bottom of the generator). During the "run" period steam is passed in at the bottom, the resulting water-gas being passed out at the top. Regenerators, to recover the heat of the "blow" gases, for preheating the steam are supplied. C. B. MARSON.

Use of heavy oil in the manufacture of carburetted water-gas. Humphreys & Glasgow, Ltd., Assees. of J. A. Perry (B.P. 345,577, 6.6.30).—A watergas set comprises a generator, a carburetter unobstructed by heat-absorbing material such as chequer brick, a superheater, and a wash-box. The fuel bed in the generator is blasted with air, the resulting "blow" gases passing to the carburetter where, after being burned with secondary air, thereby storing heat in the walls, they are passed through the superheater to the stack. At the same time a shallow fuel bed in the carburetter is blasted with air, the blow gases also being passed through the superheater to the stack. When sufficient heat has been stored in the generator fuel bed, the carburetter, and the superheater, the air blast is terminated and steam is admitted either to the base or top of the regenerator, the resulting watergas passing to the top of the carburetter, where it is carburetted by heavy oil, also admitted at the top. The oil is vaporised by the heat from the wall and from the fuel bed. The coke from the oil vaporised above the fuel bed is deposited thereon by the downward passage of the water-gas through the carburetter. From the carburetter the water-gas and oil vapour pass to the superheater, where further cracking takes place, and then through the wash-box to storage. C. B. Marson.

Production of gas from garbage. W. C. RATH (U.S.P. 1,777,449, 7.10.30. Appl., 19.5.23).—The waste materials are heated in a retort to about 400°, and the moisture and other volatile products driven off are passed through a bed of incandescent carbon, whereby an enriched water-gas is obtained. After cooling and scrubbing, a gas of high calorific value and free from sulphur is obtained; the residue in the retort is suitable as fertiliser. C. Jepson.

Removal of sulphur from industrial gases by a cyclic process with simultaneous extraction of pure sulphur. S. Hunyady and K. Koller (B.P. 345,594, 2.7.30).—The gases to be purified are brought into intimate contact with an aqueous suspension of finely-divided manganic oxide. The sulphur-manganese sulphide mixture is collected on a filter and the manganese sulphide converted into manganic hydroxide and sulphur by the action of atmospheric oxygen. The manganic hydroxide-sulphur mixture is treated with dry air, after which the sulphur is removed by a solvent such as trichloroethylene; the residual manganic oxide is employed for further gas purification. C. B. Marson.

Removing ammonia and hydrogen sulphide from gases. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 346,452, 13.1.30).—The process for removal by means of a solution of a polythionate is carried out in apparatus constructed of aluminium or an aluminium alloy. If other metals are employed they must be coated with aluminium.

R. H. GRIFFITH.

Tar separator. G. W. Watts, Assr. to Standard Oil Co. of Indiana (U.S.P. 1,781,618, 11.11.30. Appl., 24.6.25).—A centrifugal separator, for treating tars containing water, is constructed so that the material is fed tangentially to the top of an annular space between two vertical, cylindrical shells. The process is operated under reduced pressure, and the liquids draining from the inner and outer walls are separately collected.

R. H. GRIFFITH. Refining of tars. Comp. Technique des Pétroles (B.P. 346,415, 11.12.29. U.S., 11.12.28).—The higherboiling fractions of low-temperature tars are worked up, particularly for the recovery of lubricating oils and of waxes, by non-destructive distillation with steam or under reduced pressure. A pretreatment to remove acids and bases is advantageous, and crystalline waxes are separated from the distillates by filter-pressing, preferably after dilution with a low-boiling oil. melting amorphous waxes are subsequently obtained by dilution and chilling, and the residue is divided into fractions of varying viscosity. Final purification may be carried out by acid and alkali washing, and by further distillation. R. H. GRIFFITH.

Manufacture of asphaltic material. G. Abson, Assr. to Chicago Paving Laboratory, Inc. (U.S.P. 1,782,186, 18.11.30. Appl., 25.10.29).—The preparation of asphalt by air-blowing of petroleum still residues is greatly accelerated by the presence of a small quantity of a metallic salt which is not volatile at the temperature of operation and does not cause cracking of the material. The product has a higher penetration number than that obtained under the same conditions without the addition of the inorganic compound.

R. H. Griffith.

Oil still. W. A. Jones, Assr. to Babcock & Wilcox Co. (U.S.P. 1,776,437, 23.9.30. Appl., 23.5.25).—The still consists of longitudinal drums connected at either end to lower transverse drums, these, in turn, being joined by a steeply inclined bank of tubes which is traversed by the furnace gases. Oil circulates along the longitudinal drums, through a pipe of wide cross-section, to the lower of the transverse drums. It then passes by means of the heated bank of tubes to the upper transverse drum and returns to the longitudinal drum. The last-named drum is connected by means of curved pipes (to permit of differential expansion) to an upper longitudinal drum where vapours can be disengaged. One end of the apparatus is fixed, whilst the other is free to permit of expansion. A number of such stills may be arranged in series. T. A. SMITH.

Apparatus for distilling liquid hydrocarbons. N. E. MERRILL (U.S.P. 1,778,269, 14.10.30. Appl., 30.1.26).—Oil is led through a preheater into a heating chamber mounted in a furnace and consisting of a conical wall closed at the bottom with a removable plate directly exposed to the heat. This heating chamber forms the bottom section of a vertical shell still and communicates with a tube up which the vapours and liquid pass. The vapours are led off from the top of the shell, while

the liquid flows from the top of the tube and downwardly over superimposed baffle plates between the inner wall of the shell and the tube, the residue being drawn off from the bottom.

H. S. GARLICK.

Distillation of hydrocarbon oils. W. H. Bahlke, Assr. to Standard Oil Co. (U.S.P. 1,778,445, 14.10.30. Appl., 9.8.28).—Oil is passed through a heating coil wherein it is superheated to a predetermined temperature. The inlet of the coil is provided with a pipe through which a regulated amount of steam is admitted. The outlet of the coil leads into an intermediate point of a fractionating column provided above and below the point of entry with suitable fractionating devices. A perforated steam coil enters the lower end of the column where is situated an outlet for the withdrawal of the heavy stripped oil. From its upper end a vapour outlet leads to a partial condenser followed by a water condenser.

H. S. Garlick.

Apparatus for cracking oil. G. Egloff, Assr. to Universal Oil Products Co. (U.S.P. 1,766,986, 24.6.30. Appl., 23.5.21. Renewed 17.12.29).—Oil is passed under pressure through a heating coil in a furnace, into an enlarged vapour chamber provided with a vertical partition which forms a relatively small compartment at one side. Vapours leave the chamber and pass up through the bottom of a dephlegmator and condensing system. Reflux condensate is returned to the small compartment and thence to the heating coil. The residue is continually drawn off from the vapour chamber to maintain its level below the upper portion of the partition, thereby maintaining the reflux condensate separate from the residuum, but heated prior to its introduction into the heating coil. H. S. GARLICK.

Distillation of mineral oil under high vacuum.

A. E. Pew, Jun., Assr. to Sun Oil Co. (U.S.P. 1,778,565, 14.10.30. Appl., 27.2.26).—A pump with throttling device, for preventing the admission of air while oil is supplied to a still operating under high vacuum, is described.

H. S. Garlick.

Distillate petroleum liquid. J. B. RATHER and L. C. BEARD, JUN., ASSIS. to STANDARD OIL CO. OF NEW YORK (U.S.P. 1,776,598, 23.9.30. Appl., 26.11.26).—
The development of colour, odour, or gum in light petroleum distillates, e.g., kerosene and gasoline, is prevented by passing a finely-divided stream thereof downwardly through a container, filled with baffle pieces, up which is passed a copious stream of inert gas, thereby removing the air content of the distillate, which is drawn off from the bottom of the container and stored in hermetically sealed vessels. H. S. GARLICK.

Treatment of hydrocarbons. L. B. Cherry, Assr. to C. & C. Developing Co. (U.S.P. 1,779,356, 21.10.30. Appl., 10.3.22).—Cracking stock is introduced into a low-pressure, furnace-heated, horizontal still through perforations in a coil arranged therein. Vapours pass from the still through one or more vertical reaction tubes where they are subjected to the action of high-frequency oscillatory discharges sufficient to maintain a violet glow in each tube, which in addition is heated by an individual suitable furnace. The vapour outlet from the cracking tubes delivers the hot gaseous products to a fractionating condenser from which the condensate is returned to the

still by gravity, and the uncondensed vapours pass on to a final condenser. Suitable pressure-equalising means are provided for the apparatus. H. S. GARLICK.

Treatment of hydrocarbon oils. W. S. Hadaway, Jun., Assr. to Texas Co. (U.S.P. 1,776,023, 16.9.30. Appl., 16.5.27).—In a cracking process, the bulk of the heat necessary to raise the oil to cracking temperature is supplied during passage through a furnace-heated high-pressure coil, and a further relatively small amount of heat is supplied by passing the hot oil through an electrically-heated tube in which the electric heat input is regulated to maintain a predetermined outlet temperature.

H. S. Garlick.

Apparatus for heat-treating hydrocarbon oils. H. C. Wade, Assr. to Hydrogenating Process Corp. (U.S.P. 1,777,708, 7.10.30. Appl., 2.5.28).—Economy of fuel and increased capacity of heat-treating apparatus are obtained by providing a heating kiln of normal dimensions with several series of coils, the coils of each series lying contiguously and having the same axis and diameter. One series of coils embraces a second series so that both have substantially the same axis, the lineal capacity of both being substantially the same. Each coil has a discharge outlet at its base. H. S. Garlick.

Decomposition of relatively high-b.p. oils into relatively low-b.p. oils. A. L. Strout, Assr. to Standard Oil Co. of California (U.S.P. 1,776,985, 30.9.30. Appl., 25.4.27).—Oil is heated to cracking temperature in a tube furnace and passed to a reaction and vaporising chamber. The vapours from this chamber are taken to a dephlegmator and the residue passes to a further vaporising chamber in which the pressure is reduced and part of the oil is vaporised. These vapours are scrubbed with the incoming stock, which is then used as refluxing material in the dephlegmator before passing to the cracking coil. T. A. Smith.

Neutralisation of washed light oil. J. J. Lawton, Assr. to Semet-Solvay Co. (U.S.P. 1,779,944, 28.10.30. Appl., 31.12.27).—Crude light oil from the destructive distillation of coal is washed with sulphuric acid and the resultant sludge separated. The washed oil is treated with ammonia and subjected to steam distillation in the presence of the neutralising liquid to separate low-boiling oils, thereby breaking any emulsions and preventing decomposition and volatilisation of any sulphur compounds present. H. S. Garlick.

Manufacture of hydrocarbons of high b.p., in particular viscous hydrocarbons from hydrocarbons of low b.p. J. Y. Johnson. From I. G. Farbenind. A.-G. (B.P. 345,334, 18.12.29).—Low-b.p. hydrocarbons are condensed by heat treatment in the presence of a previously prepared double compound of ethylene and aluminium chloride dissolved in a hydrocarbon solvent, while passing gaseous olefines or mixtures containing them into the reaction mixture. The double compound may be made by passing, at 40—60°, ethylene alone or mixed with inert gases into a solvent, free from unsaturated compounds (e.g., paraffin oil), in which the aluminium chloride is suspended.

H. S. Garlick.

Manufacture of conversion products of higher paraffin hydrocarbons [lubricants]. A. Carpmael.

From I. G. Farbenind. A.-G. (B.P. 343,948, 11.9.29).—Di- and poly-halogenated paraffins above C<sub>8</sub> are heated (e.g., at 190—220°) with aqueous caustic alkalis or alkalic carbonates (excluding hydrogen carbonates) or alkaline earths, preferably under pressure, to give viscous light-coloured oils, which are probably unsaturated monohydric alcohols and may still contain halogen.

Reclamation of lubricating oils. L. D. Grisbaum, Assr. to Railway Service & Supply Corp. (U.S.P. 1,777,722, 7.10.30. Appl., 30.4.27).—The used oil is mixed with a chemical treating solution (e.g., caustic soda or potash) and heated in a sealed container to approx. 115°. The developed pressure is further increased by a superimposed air pressure sufficient to prevent ebullition. Heating is stopped and the pressure maintained during cooling. H. S. Garlick.

[Lubricating or insulating] oil purification process. F. M. Clark and A. T. Harding, Assis. to Gen. Electric Co. (U.S.P. 1,776,550, 23.9.30. Appl., 18.12.29).—Unsaturated substances are extracted in the preparation or recovery of such oils, by means of a polyhydric alcohol (glycerol or ethylene glycol); after separation the oil is distilled to remove traces of alcohol. T. A. Smith.

Purification of [insulating] oils. Brit. Thomson-Houston Co., Ltd., Assees. of F. M. Clark (B.P. 345,708, 9.1.30. U.S., 10.1.29).—Glycerin or other equivalent polyhydric alcohol is maintained in contact with the oil, but out of contact with the insulating and electrical members of transformers, switches, etc.

J. S. G. Thomas.

Oil-purifying process. L. D. Jones, Assr. to Sharples Specialty Co. (U.S.P. 1,778,831, 21.10.30. Appl., 29.4.24).—Oil is continuously withdrawn from a steam turbine or similar circulating system and treated with cooled water to bring the temperature of the resulting mixture to that at which precipitation of sludge and impurities soluble in hot oil occurs. The water and impurities are separated from the oil by centrifugal means, and then the oil is heated and any traces of water remaining are removed by a further centrifugal treatment.

H. S. Garlick.

Refining of hydrocarbons. N.V. DE BATAAFSCHE PETROLEUM MAATS., Assees. of R. I. Lewis (B.P. 345,596, 7.7.30. U.S., 15.7.29).—Pressure distillate containing sulphur is treated first with dilute acid, e.g., sulphuric acid (d 1·23), at atmospheric temperature, and thereafter with acid of d 1·84 at a temperature substantially below atmospheric. If desirable, a further treatment with acid (d 1·84) at a raised temperature may be given. H. S. GARLICK.

Purification of hydrocarbons. W. H. Low, Assr. to Richfield Oil Co. of California (U.S.P. 1,777,619, 7.10.30. Appl., 6.9.27).—A substantially dry cracked naphtha or gasoline stock containing mercaptans or other sulphur compounds is rendered "sweet" to the "doctor test" by filtration through a filter-bed containing sufficient comminuted dry lead sulphide The lead sulphide may be intermittently revivified by treatment with a solution of an alkaline sulphide.

H. S. GARLICK.

Purification of hydrocarbons. M. Mercurio, Assee. of J. F. A. Bruzac (B.P. 346,152—3, 2.4.30. Fr., [A] 30.10.29, [B] 22.6.29).—(A) Crude hydrocarbons, containing sulphur compounds which are not separable by distillation, are purified by treatment with oxygen or ozone, preferably at ordinary temperatures, followed by washing with a solution of alkali. (B) Distillates obtained from heavy hydrocarbons by heating them in contact with inert surfaces at temperatures below 500° are purified as described above. [Stat. refs.]

R. H. GRIFFITH.

Refined viscous hydrocarbon oil. T. H. ROGERS,
Assr. to STANDARD OIL Co. (U.S.P. 1,774,845, 2.9.30.

Appl., 19.8.26).—Acid formation and increase in emulsibility in use of highly refined viscous hydrocarbon oils is prevented by adding thereto up to 0·1% of an aromatic compound in which an amino- or hydroxyl group is attached to the ring. Suitable additions are: diphenylamine 0·4—0·1%, gallic acid 0·002%, dibutyl-resorcinol 0·05%.

H. S. GARLICK.

Testing the purity of solid or liquid hydrocarbons. A. Esau (B.P. 345,464, 5.3.30. Ger., 13.3.29).—The material to be tested is subjected to the action of short electric waves of frequency of the order of 10<sup>8</sup> Hertz and the resultant changes of temperature produced are measured. H. S. Garlick.

[Anti-knock] motor fuels. H. D. ELKINGTON. From Benzol-Verband Ges.m.b.H. (B.P. 345,587, 20.6.30).—The mixture of aromatic amines obtained by the direct nitration and reduction of crude hydrocarbon mixtures containing benzene and its homologues, obtained in the dry distillation of coal, is added to a benzine motor fuel.

H. S. Garlick.

Absorbent oil treater. J. L. Looney (U.S.P. 1,777,005, 30.9.30. Appl., 13.8.28).—An apparatus for the chemical treatment of oils to remove sulphur and sludge consists of a horizontal cylinder fitted with intake and offtake manifolds. The former delivers into a number of pipes which convey oil to the lower part of the cylinder. The offtake is connected to the upper part of the cylinder. Openings for running off sludge are fitted at the oil-chemical interface. T. A. SMITH.

Burners for pulverulent materials. H. Nielsen and B. Laing (B.P. 346,826, 16.1.30).

Quenching and conveying apparatus for coke or charred substances. South Metropolitan Gas Co., and C. C. Carpenter (B.P. 347,424, 25.3.30).

Means for controlling or operating water-gas apparatus. A. Breisig (B.P. 346,711, 15.1.30. Austr., 15.1.29).

Furnace. Thermal treatment of material. Steam distillation.—See I. Antioxidants [for oils].—See III. Wax-impregnated fibres.—See V. Water-proof fibres. Mine-ventilating tubing.—See VI. Hydrogen.—See VII. Road coverings. Paving compositions.—See IX.

#### III.—ORGANIC INTERMEDIATES.

Determination of ethylene.—See II. [Furan derivatives from] agricultural wastes.—See XVI. Tests for methyl alcohol.—See XVIII. Cresol

soap solutions.—See XX. Alkyl p-hydroxybenzoates as disinfectants.—See XXIII.

See also A., May, 574, Furfuraldehyde. 578, Reduction of carboxylic acids. 612, Anthracene derivatives. 623, p-2-Carboxybenzoylbenzene-sulphonic acids. 630, Pyridine bases from tar. 638, Detection of carbon disulphide. 656, Acetic fermentation. Propionic group of bacteria.

PATENTS.

Production of organic compounds containing oxygen [acetic acid from methyl alcohol and carbon monoxide]. Brit. Celanese, Ltd., and H. F. Oxley (B.P. 343,947, 22.8.29).—There is added to the catalyst (e.g., phosphoric acids or acid phosphates) as promoter iron, manganese, vanadium, chromium, cobalt, or nickel, or a compound thereof; e.g., 0.5—1% of iron or manganese formate is added to pyrophosphoric acid to increase the yield of acetic acid from methyl alcohol and carbon monoxide at 290—315°/120—160 atm.

C. Hollins.

Manufacture of oily polymerisation products from butadiene or a homologue thereof. I. G. FARBENIND. A.-G. (B.P. 343,116, 16.10.29. Ger., 16.10.28).—Vapour of butadiene (etc.) is passed through a tube at  $400-500^{\circ}$ , in presence, if desired, of a catalyst, e.g., iron. Butadiene gives vinylcyclohexenes, b.p.  $110-135^{\circ}$ ; isoprene yields mainly dipentene, b.p.  $165-180^{\circ}$ ; from  $\alpha\gamma$ -dimethylbutadiene there is obtained an oil, b.p.  $195-210^{\circ}$ /atm.,  $90-100^{\circ}/13$  mm.

C. HOLLINS.

Antioxidant or age-resister [for rubber, oils, etc.]. A. M. CLIFFORD, Assr. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,779,390, 21.10.30. Appl., 27.7.29).

—On heating piperidine and α- or β-naphthol for several hours at 260—290° under pressure, products are obtained, viz., 1- and 2-piperidylnaphthalenes, respectively, which are effective antioxidants for rubber and for other oxidisable organic substances such as transformer oils. For the production of this type of antioxidant substituted naphthols also may be used in the reaction with piperidine.

D. F. Twiss.

Manufacture of wetting, cleansing, and dispersing agents. J. Y. Johnson. From I. G. Farbenind. A.-G. (B.P. 343,098, 7.8.29).—Open-chain or monocyclic aliphatic ketones above C<sub>7</sub>, free from aryl and carboxyl groups, are sulphonated. Oleone, obtained by heating oleic acid and iron filings at 340°, is treated with 23% oleum until the product is water-soluble and is not saponified by heating with 10% hydrochloric acid; or is treated with chlorosulphonic acid in carbon tetrachloride at 20—25°. The ketone from palmitic acid or from tall oil gives a similar product. C. Hollins.

Manufacture of mercaptobenzthiazoles. Goodyear Tire & Rubber Co., Assees. of J. Teppema and L. B. Sebrell (B.P. 343,013, 8.8.29. U.S., 26.11.28).—A mixture of carbon disulphide and an alkali salt of an o-aminothiophenol is treated with hydrochloric or sulphuric acid while heating under reflux. C. Hollins.

Manufacture of (A) 1:4:4'-trihydroxy-2:2'-dianthraquinonyl3:1'-oxide [2:6:9-trihydroxy-3:4:7:8-diphthaloyldiphenylene oxide] and (B) 1:4:1':4'-tetrahydroxy-2:2'-dianthraquinonyl.

A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 343,090—1, 13.11.29).—Substance A of B.P. 10,074 of 1903 (B., 1904, 367) is 1:4:1':4'-tetrahydroxy-2:2'-dianthraquinonyl, whilst substance B of that specification is the corresponding furan derivative formed by loss of 2 hydrogen atoms. (B) Substance A is conveniently obtained free from substance B by warming quinizarin with piperidine in alcohol at 60—70°. (A) This product is converted smoothly into the furan derivative by heating in nitronaphthalene or other high-boiling nitro-compound. C. Hollins.

Lubricants.—See II. [Acetic acid from] black liquor.—See V. Elastic-plastic body.—See XIV.

#### IV.—DYESTUFFS.

Determining dyes in foods.—See XIX.

See also A., May, 563, Action of electrolytes on substantive dyes. 618, Combined dye radicals. 627, Condensation of ketones with resorcinol. Fluoran derivatives. 632, Triphenylmethane dyes. Dyes from acenaphthaquinone. 636, Azo dyes from arsanilic acids. 644, Colouring matter of milk.

#### PATENTS.

Manufacture of derivatives of pyranthrone [vat dyes]. J. Y. Johnson. From I. G. Farbenind. A.-G. (B.P. 343,100, 9.8.29).—The dihvdroxypyranthrone

(annexed formula) or the monohydroxy - compound formed by its reduction, prepared by acid condensation of 2:2'-dialdehydo- or 2:2'-tetra-halogenodimethyl-1:1'-dianthraquinonyl in presence of a reducing agent, is alkylated and, if desired, halogenated. Dimethoxy-(golden-orange), diethoxy-, and monomethoxy-

(red-brown) derivatives are described. C. Hollins.

Manufacture of [acid] dyes of the anthraquinone series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 342,667 and 342,706, 30.9.29).—4-Halogeno-1aminoanthraquinone-2-sulphonic acids are condensed (A) with aromatic diamines (except o-arylenediamines) which may carry a single N-alkyl, -aralkyl, or -cycloalkyl substituent (p-phenylenediamine itself and its nuclear derivatives being excluded), or (B) with aromatic diamines (except o-arylenediamines), the product being subsequently acylated, especially by means of ω-halogenoacyl halides. Green-blue to blue wool dyes of good levelling power are obtained. Examples are the condensation of 4-bromo-1-aminoanthraquinone-2-sulphonic acid with m-phenylenediamine (green-blue), acetylated (pure blue), chloroacetylated (reddish-blue), dichloroacetylated (blue), or \(\beta\)-chloropropionylated (blue); with benzidine (green-blue), acetylated (green-blue); with tolidine (blue); with 1:5- or 1:8-naphthylenediamine (blue or grey-blue); with 4:4'-diaminodiphenyl sulphide (blue); with p-aminoethylaniline (greenish-blue), acetylated (reddish-blue); with p-amino-N-cyclohexylaniline (greenish-blue); with p-phenylenediamine, acetylated (green-blue), chloroacetylated (blue), or con-

densed with ethyl chloroformate (blue); with p-phenylenediaminethiosulphonic acid, acetylated (blue); with m-aminoethylaniline, chloroacetylated (blue); with p-aminomethylaniline, chloroacetylated (blue).

C. Hollins.

Manufacture of [mono-]azo dyes and their application. Soc. Chem. Ind. in Basle (B.P. 343,016, 8.10.29. Switz., 8.10.28).—Diazo compounds are coupled in the 3-position with 4-hydroxy-1-naphthoic esters to give, according to the first component used, light-fast acid, mordant, lake, or cotton dyes, or pigments for acetate silk or lacquers. Examples of diazo components are: aniline (red), p-aminoacetanilide (bluish-red), m-aminobenzyl alcohol (red-orange), sulphanilic acid (red-orange), 4-chloro-o-aminophenol-6-sulphonic acid (chrome-red-violet), and β-naphthylamine-1-sulphonic acid (red barium lake). C. Hollins.

Manufacture of [mono-]azo dyes [for acetate silk, lacquer pigments, etc.]. Soc. Chem. Ind. in Basle (B.P. 343,006, 2.11.29. Switz., 2.11.28).—A nonsulphonated o-aminophenol o-arylsulphonate is diazotised and coupled with a non-sulphonated phenol or arylamine of the benzene series or an aniline-formaldehyde-bisulphite compound, the dye is hydrolysed and then alkylated or acylated, to give pigments suitable for acetate silk dyeing and for colouring zapon varnishes. Examples are: o-aminophenyl toluene-p-sulphonate > aniline, hydrolysed, and acetylated (greenish-yellow), or > phenol, hydrolysed, and ethylated (greenish-yellow).

C. Hollins.

Manufacture of azo dyes [ice colours and pigments]. W. W. Groves. From I. G. Farbenind. A.-G. (B.P. 343,419, 10.4.30. Addn. to B.P. 336,938 and 339,620; B., 1931, 151, 339).—2:3-Hydroxynaphthoic 3-chloro-4:6-dimethoxyanilide and 2-chloro-4:5-dimethoxyanilide are used as coupling components for pigments or ice colours, especially with diazotised m-xylidine, 4:6-dichloro-m-toluidine, or o-nitroaniline. C. Hollins.

Manufacture of azo dyes insoluble in water [ice colours and pigments]. I. G. Farbenind. A.-G. (B.P. 343,164, 15.11.29. Ger., 16.11.28).—Arylamides of 3-hydroxycarbazole-2-carboxylic acid are coupled in substance or on the fibre with diazo components free from solubilising groups. Examples are: anilide with diazotised 5-nitro-o-anisidine (brown), 3-aminocarbazole (red-brown), or tetrazotised dianisidine (blue-black);  $\beta$ -naphthylamide with tetrazotised dianisidine (dark blue), diazotised o-phenetidine  $\rightarrow$   $\alpha$ -naphthylamine (deep black);  $\beta$ -naphthylamide with diazotised 4-nitro-o-anisidine (brown);  $\rho$ -anisidide with diazotised 4-nitro-o-anisidine (black-brown);  $\sigma$ -anisidide with diazotised 5-nitro-o-anisidine (brown). C. Hollins.

Manufacture of [azo] dyes containing metal and the application thereof [in varnishes]. Soc. Chem. Ind. in Basle (B.P. 343,014, 7.10.29. Switz., 5.10.28).— Non-sulphonated, non-carboxylated monoazo dyes of the type: an o-aminophenol  $\rightarrow$  a phenol or an arylamine, are treated in presence of alkali but in absence of hyposulphites with hydroxides of one or more metals (preferably mordant metals) or with the products of the interaction of such hydroxides with alcohols and alkali.

Examples are: 2-amino- $\alpha$ -naphthol  $\rightarrow$   $\beta$ -naphthol with chromium hydroxide and aqueous potassium hydroxide to give a violet pigment soluble in varnish media; 4-chloro-o-aminophenol  $\rightarrow$  resorcinol with chromium hydroxide, alkali, and glycerol (brown-red varnish pigment); etc. The pigments are specially suitable for zapon varnishes. C. Hollins.

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

Factors affecting the development of mildew in wool. R. Burgess (J. Soc. Dyers and Col., 1931, 47, 96-99).—Mainly a review of previous investigations (B., 1930, 96, 1062) from a technical viewpoint. Commercially scoured worsted cloth containing about 0.5% of alkali (calc. as sodium carbonate) will not "mildew at 23° unless its moisture regain is about 24%, which corresponds to the regain in an atmosphere of 94% R.H. Unscoured yarn, raw wool in the grease, and sized wools mildew at a lower R.H., whereas well washed wool mildews only slowly even in a moisture-saturated atmosphere. The presence of vegetable oils, but not mineral oils, in wool promotes mildew growth. Disintegration of wool by micro-organisms can be followed by means of the Rimington-Pauly test (cf. Rimington, B., 1930, 1021). Sodium silicofluoride is the most suitable mildew preventitive for wool. The stoving of wool gives temporary protection, but over-chlorination (in the non-shrink finish) favours the growth of mildew. A. J. HALL.

Identification of fungi causing mildew in cotton goods. The genus Aspergillus. II. G. SMITH (J. Text. Inst., 1931, 22, T 110—116).—An analysis is made of the whole series of strains belonging to the above group isolated from mildewed cloths and yarns.

B. P. RIDGE.

Permeability of fabrics to air. M. C. Marsh (J. Text. Inst., 1931, 22, T56—63).—The apparatus and methods used are fully described, and comparisons are made between the permeabilities of knitted and woven cotton, woollen, artificial silk, linen, and silk fabrics. Large variations from point to point are found for most fabrics.

B. P. Ridge.

Production of cellulose by means of chlorine. III. Chlorination. II. J. KAWAMURA (J. Soc. Chem. Ind., Japan, 1931, 34, 62—64 B; cf. B., 1931, 478).— Samples of bagasse were chlorinated for various times over the temperature range 22—27°, using different proportions of chlorine to material, and the ratio of chlorine present as hydrochloric acid to combined chlorine and the yield of cellulose were determined. When the temperature and time of reaction are increased, the amount of chlorine taking part is increased, whilst, as previously observed by Waentig, the ratio of hydrochloric acid formed to combined chlorine remains practically constant. When a much larger amount of chlorine is used for a long time, this ratio is increased and probably oxidation of the non-lignin or of the lignin substance is promoted.

B. P. Ridge.

Cellulose acetate. H. Reedy (Kunstseide, 1930, 12, 420—422; Chem. Zentr., 1931, i, 596).—Acetylsulphuric acid is considered to be formed first, and

then from it sulphoacetic acid. The formation of sulphoacetates is unlikely to be prevented by acetylation with acetylsulphuric acid, in which free sulphuric acid and sulphoacetic acid are in equilibrium.

A. A. ELDRIDGE.

Production of viscose silk of high tensile strength. H. Schmidt (Chem.-Ztg., 1931, 55, 265—267, 286—288).—The latest processes as described in patent literature are outlined, with special reference to the composition of the spinning baths and methods of stretching the thread.

F. R. Ennos.

Chemical analysis of rayons. I. Chemical properties of some commercial rayon yarns. B. P. Ridge, H. L. Parsons, and M. Corner (J. Text. Inst., 1931, 22, T 117—140).—The following properties have been measured for standard (first quality) varieties of viscose, Lilienfeld, cuprammonium, acetate, and nitrocellulose rayons of recent manufacture, mostly of British origin: copper number, loss of weight on boiling with alkali, methylene-blue absorption, fluidity in cuprammonium hydroxide solution, ash content and ash alkalinity, material extractable by solvents, sulphur, copper, and iron contents, and, for acetate rayons, acetic acid content. The first four are dependent mainly on the extent of chemical modification of the cellulose itself, whilst the remainder are indicative of the nature and amount of the non-cellulose impurities. The chemical properties of these materials resemble those of chemically attacked cotton cellulose rather than those of the purified but otherwise unchanged material. None of the chemical properties measured can be used to characterise all the varieties examined. Copper number and loss of weight fail to distinguish between viscose, Lilienfeld, and cuprammonium yarns made from wood pulp; methylene-blue absorption is lowest for acetate rayon, but is otherwise very variable; and fluidity values are low and approximately the same for Lilienfeld and cuprammonium rayons made from cotton linters and high for acetate varieties, though here they are of little significance because these are not composed wholly of cellulose. All the tests applied show the chemical inferiority of nitrocellulose rayon.

B. P. RIDGE.

Durability of purified wood fibres. Accelerated ageing tests of various types of papermaking fibres. G. A. RICHTER (Ind. Eng. Chem., 1931, 23, 371—380).—Papers made from rag stock, sulphite wood pulp, and purified wood pulp of high α-cellulose content, respectively, have been tested. Exposure to a stream of air at 100° for 72 hrs. causes appreciable reduction of physical strength of sulphite papers, rag and purified wood retaining a high percentage of their original strength. Oxygen accelerates the decomposition of paper, exposure at 60° and 200 lb./in.2 giving similar results to the ageing at 100° in air. Decomposition is, in general, accelerated by the presence of moisture. Highly-hydrated pulps prepared from the purified wood fibres do not deteriorate to any greater extent than those beaten normally, whereas highlybeaten sulphite pulps decompose readily. Rag and purified wood papers are no more resistant to the action of acid vapours than sulphite papers. Extensive

tables showing the effect of ageing on the  $\alpha$ -cellulose content, tensile strength, tearing strength, and folding endurance are given. T. T. Potts.

Correlation of bursting strength and tensile strength of paper. J. Strachan (Proc. Tech. Sect. Paper Makers' Assoc., 1930, 11, 86—100).—The formulæ advanced by Dawe, Bergmann, Carson, and Dalén for the connexion between bursting strength and tensile strength are discussed and criticised. Deviation of calculated tensile strength from observed values is shown to be an indication of the degree of homogeneity of the sheet, and a factor of uniformity, v = Br/2T, where r is the radius of the test disc used for the burst, is claimed to yield satisfactory results. The factor v is shown to depend on fibre length and cohesion.

Variability in results in paper strength testing. C. V. OLIVER (Proc. Tech. Sect. Paper Makers' Assoc., 1930, 11, 53—74).—Numerical examples of the variations occurring in the testing of paper for bursting, tearing, and tensile strengths are given. It is shown that the errors vary according to the type of paper being examined, and that different instruments designed for a given test, e.g., the Mullen and Ashcroft bursting testers, do not yield results which bear a constant relation to one another. The effects of varying conditions on the accuracy of results are discussed.

Colouring of paper. N. L. Mathews (Proc. Tech. Sect. Paper Makers' Assoc., 1930, 11, 144—156).—A lecture, giving an account of the various natural and synthetic colouring matters used in the paper industry, with notes on their application. T. T. Potts.

Soda recovery [in the paper mill]. J. A. WALKER (Proc. Tech. Sect. Paper Makers' Assoc., 1930, 11, 187—198).—An account is given of practical experience in the conduct of plant for the recovery of soda from the spent liquors of esparto digestion. T. T. Potts.

Sampling of cotton for determination of fibre properties. III. Size and reliability of a satisfactory sample. R. S. Koshal and A. J. Turner (J. Text. Inst., 1931, 22, T 17—55).

Fabric wear testing. I. H. Crawshaw, W. E. Morton, and K. C. Brown (J. Text. Inst., 1931, 22, T 64—76).

Random and systematic selections of warp specimens in cloth sampling. A. J. Turner (J. Text. Inst., 1931, 22, T 77—97).

See also A., May, 606, Cellulose butyrate, and nitrate. Red beech. 655, Cellulose-fermenting organism.

PATENTS.

Manufacture of soluble ethers from vegetable materials. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 343,147, 14.11.29).—Straw, sea-weed, esparto grass, etc., containing fibres which are encrusted but not turned into wood, are alkylated, and especially benzylated, by the methods used for cellulose ethers.

C. HOLLINS.

Manufacture of cellulose acetate. Kodak, Ltd., Assees. of C. J. Staud and C. S. Webber (B.P. 345,235,

10.12.29. U.S., 22.12.28).—Cellulosic material is treated at 0° with a saturated solution of nitrogen peroxide in a lower aliphatic acid, preferably acetic acid, before being acetylated in the usual manner. F. R. Ennos.

Treatment of cellulose derivatives. Brit. Celanese, Ltd. (B.P. 345,408, 29.1.30. U.S., 7.2.29).—Cellulose esters are fractionally precipitated from solution, e.g., by addition of a non-solvent or by cooling, yielding fractions differing in physical properties.

F. R. Ennos.

Production of artificial filaments, films, and like products from viscose. H. Dreyfus (B.P. 344,474, 5.11.29).—Viscose is coagulated in the required form as a cellulose metal xanthate in a bath free from mineral acid and composed of either an organic liquid (alcohol) or an aqueous solution of a metallic salt; the product is continuously reconverted into cellulose by means of mineral acids, acid esters of mineral acids, or organomineral acids in low or high concentration, together with glycerin, sugars, sulphonated oils, etc., if desired.

F. R. Ennos.

Manufacture of artificial filaments, threads, ribbons, films, and other artificial products. H. Dreyfus (B.P. 344,510, 7.12.29).—Materials of modified lustre are produced by spinning solutions of cellulose or of cellulose derivatives containing insoluble organic fibrous material (cotton, linen, straw, wheat husks, wool, or artificial fibrous materials) in a finely-divided state.

F. R. Ennos.

Spinning of artificial threads or filaments. Cellulose Acetate Silk Co., Ltd., and H. C. Curtis (B.P. 344,591, 10.1.30).—To obtain a temporary increase of pressure at the jet when starting the spinning operation, communication between the jet and the supply pipe is closed, and the solution pumped into a pressure chamber in or connected with the supply pipe; when a sufficiently high pressure is reached the pressure chamber is connected with the jet.

F. R. Ennos.

Dry-spinning of artificial silk. "CHATILLON" Soc. Anon. Ital. Per la Seta Artificiale, and E. Orioli (B.P. 344,385, 25.11.29).—The spinning cell is provided with a pipe open at each end, which is heated by either an internal pipe or an external jacket. The rising current of gases in the pipe is caused to impinge on the filament from one side only of the cell through the upper aperture, which has its axis at right angles to the path of the filament.

F. R. Ennos.

Treatment of artificial fibres. L. A. Paley (U.S.P. 1,779,103, 21.10.30. Appl., 17.12.26).—Freshly spun fibre cakes, after insertion of a specially constructed holder, are grouped end to end in the form of a closed cylinder on a tube having a number of openings through which water, hot dilute sodium carbonate, or air is passed under pressure to the interior and through the walls of the cakes, whereby the latter are washed, freed from impurities, and dried; the holder also facilitates unwinding of the fibre cake therefrom after treatment.

F. R. Ennos.

Digestors for treating fibrous materials. J. J. DE LA ROZA (B.P. 346,119, 6.3.30).—The digestor is constructed so that the material may be charged.

constructed so that the material may be charged, digested, and discharged while contained in baskets or

other containers; the manhole is of about the same diameter as the cylindrical body portion.

B. M. VENABLES.

Manufacture of pulp. J. Neumann (U.S.P. 1,778,199, 14.10.30. Appl., 14.6.26. Renewed 4.1.30). —Cellulosic raw material is digested with a solution of sodium silicate, either hot or cold, to which a solution of sodium sulphide is afterwards added. F. R. Ennos.

Manufacture of cellulose pulp. Verein. Glanz-stoff-Fabr. A.-G. (B.P. 345,208, 12.12.29. Ger., 13.12.28).—At any desired stage before working up into cellulose solutions or compounds, the pulp is broken down by intensive chemical treatment such as boiling with acids, strong bleaching, or oxidation in the presence of alkali, so that it has a copper viscosity (method of determination described) of less than 6. It is then freed from hemicellulose by dilute alkali until the α-cellulose content is at least 90% (preferably >93%).

Manufacture of transparent sheet material.

S. D. Warren Co., and W. M. Driesen (B.P. 344,471, 29.10.29).—A paper web composed of transparent fibres is saturated with a solution of a cellulosic film material (nitrocellulose) in a mixture of a volatile solvent (alcohol and volatile esters) and a stable solvent (lanoline, amyl phthalate), and the volatile solvent is removed by evaporation.

F. R. Ennos.

Stencil sheet. E. E. Novotny (U.S.P. 1,776,368, 23.9.30. Appl., 11.7.28).—A porous base (Yoshino paper) is coated with a cellulose ester solution (pyroxylin lacquer) containing ethyl oleate and carnauba wax.

Manufacture of impregnated sheets. H. C. Cheetham, Assr. to Bakelite Corp. (U.S.P. 1,776,885, 30.9.30. Appl., 16.11.26).—A reactive resin, e.g., of the phenol-formaldehyde type, together with a suitable plasticiser (ethyl tartrate) is heated until it loses its initial fusibility and solubility; after fine grinding, the plasticised resinoid is mixed with fibre in the presence of water and laid into sheets, which are subsequently dried and hot-pressed.

F. R. Ennos.

Hektographic materials and blankets. W. W. Triggs. From Ditto Inc. (B.P. 345,062, 25.2.30).—The coating materials comprise a glue and glycerin composition, containing approx. 0.5% of titania.

H. ROYAL-DAWSON.

Manufacture of dialysing and pervaporating membranes. P. A. Kober (U.S.P. 1,779,942, 28.10.30. Appl., 26.11.26).—A cellulose derivative (nitrocellulose) and a protein substance (gelatin) are dissolved in a common solvent (acetic or other aliphatic carboxylic acid) and the membrane, which is formed in the required shape by removal of the solvent, is treated with water. (Cf. B., 1917, 1038.)

F. R. Ennos.

[Wax-]impregnated fibrous materials. H. FRIED-LANDER, and SPICERS, LTD. (B.P. 345,453, 26.2.30. Addn. to B.P. 236,224; B., 1926, 986).—Instead of hard wax the impregnating material used consists of montan wax pitch, brown-coal wax pitch, or similar pitchy residues derived from wax-containing materials.

F. R. Ennos.

Production of designs [on paper, cloth, etc.]. C. Maier and W. Swaysland (U.S.P. 1,778,397, 14.10.30. Appl., 18.8.28).—A visible design is formed on white paper etc., the surface of which has been impregnated with a series of tiny spots of lead sulphate, by treating certain of the spots with a solution of a soluble sulphide; the paper (etc.) may be used again by reconverting the lead sulphide spots into lead sulphate by means of hydrogen peroxide. Half-tones are thus obtainable, and spots of other colours produced with different solutions. F. R. Ennos.

Device for evaporation of the solvents in the manufacture of papers and films from cellulose esters and ethers. A. GIEBMANNS (B.P. 344,645, 24.2.30. Fr., 23.2.29).—The casting cylinder or other device on which the films etc. are formed is enclosed in a chamber subdivided into several compartments, preferably four, each containing one or more heating devices such as radiators; as the amount of solvent to be evaporated in the successive compartments decreases, the radiators are arranged nearer to the casting surface of the cylinder, which finally passes through a compartment containing water, heated or not, to remove the last traces of solvents and provide the film surface with water vapour, to avoid formation of static elec-F. R. Ennos. tricity.

Treatment of black liquor [from soda-pulp process]. L. Bradley and E. P. McKeefe, Assrs. to Bradley-McKeefe Corp. (U.S.P. 1,779,226, 21.10.30. Appl., 3.1.21. Renewed 5.7.28).—Black liquor is treated with sufficient acid or acid salt yielding a causticisable sodium salt to precipitate most of the organic matter or in addition to liberate the acetic and other volatile acids. After removal of the organic matter, the liquor is causticised before or after concentration and is returned to the digestor for re-use; the acetic acid in the liquor is recovered either as sodium acetate during concentration, or as free acid by distillation of the acid liquor. F. R. Ennos.

[Apparatus for] stretching of artificial threads. Verein. Glanzstoff-Fabr. A.-G. (B.P. 347,454, 7.4.30. Ger., 29.4.30).

Waterproofing of paper.—See VI. Spent wood liquors.—See XII. Rubber compositions.—See XIV.

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

Detection of traces of active chlorine in [bleached] textiles. A. Schmidt (Z. angew. Chem., 1931, 44, 278). — The production of a blue colour when bleached textile fibres are brought into contact with an iodide-starch mixture is not proof of the presence of hypochlorite or free chlorine (cf. Schwarze, B., 1931, 291), neither of which is likely to exist for any period in contact with organic materials. It may, however, result from the presence of relatively stable compounds of the chloro-amine type, which are frequently produced during the bleaching of linen and cotton; they may be removed most readily by treatment of the material with bisulphite or thiosulphate. H. F. Gillee.

Measurement of the colour of textile fabrics. VII. Relations between concentration of dyestuff and colour of unexposed and exposed fabrics. P. W. CUNLIFFE and (MISS) P. N. LAMBERT (J. Soc. Dyers and Col., 1931, 47, 73—78).—A continuation of previous work (B., 1930, 657), with special reference to the relations between "concentration" and "colour quality," between "concentration" and "brightness factor" (luminosity), and between the "brightness factor" of exposed and unexposed dyeings, these relations being illustrated by means of numerous graphs. The "colour quality" curves of Acetyl Rose 2GL and Coomassie Violet R dyed on worsted indicate that in dyeings of concentration exceeding 4% and 2%, respectively the colour becomes darker or less pure. The linear relationship discovered previously (J. Soc. Dyers and Col., 1929, 45, 306) between luminosity ("brightness factor") of a dyed material and the logarithm of the concentration of the dye has been examined and confirmed over a much wider range of concentrations with a number of dyes; the earlier results have also been confirmed for concentrations less than the opti-Owing to the change in "brightness factor" observed during the fading of several dyes it is concluded that no method is permissible for calculating the degree of fading which assumes that a dyeing will fade to a colour equal in brightness to that of the original undyed material (cf. Haller and Ziersch, B., 1930, 369). A. J. HALL.

Fur dyes: their oxidation and identification on the fibre. R. B. Forster and C. Soyka (J. Soc. Dyers and Col., 1931, 47, 99—109).—Unoxidised bases may be extracted by means of ligroin or ether, the evaporated extract being further extracted with 0.1N-hydrochloric acid for separation of fats from the bases, or by means of that acid after first degreasing the fur; these methods are satisfactory for rabbit, skunk, sable, beaver, and sheep skins. Full details are given of a number of colorimetric methods for identifying the bases in the resulting extract, and the reactions of a number of bases are tabulated. Bandrowski's base, formed by oxidation of p-phenylenediamine, reacts very similarly to p-phenylenediamine in the Indamine and hypochlorite tests, but it can be distinguished by reducing with zinc and hydrochloric acid and then exposing a drop of the resulting colourless solution on filter paper to ammonia; the spot becomes brownish and then brownish-violet with a pink rim. A satisfactory method is described for determining p-phenylenediamine in dilute solution by titration with nitrous acid; in this reaction only one amino-group is diazotised, the second group forming an inner diazoamino-compound. p-Phenylenediamine oxidises with hydrogen peroxide more easily than the m-compound in the absence of a mordant, but the reverse is true when a mordant is present. p-Phenylenediamine has been identified in the reduction products of Bandrowski's base. A. J. HALL.

Mildew in wool. Colouring of paper.—See V. Determining dyes in foods.—See XIX.

See also A., May, 572-3, Kinetics of chlorine bleaching.

PATENTS.

Finishing [bleaching or dyeing] of yarns or fabrics. F. J. Stokes (U.S.P. 1,779,831, 28.10.30. Appl.,

22.6.25).—The usual treatments for preparing yarns and fabrics for dyeing are avoided by placing the textile material (in the standard package or form in which it is manufactured) in a centrifuge situated within a closed tank, creating a vacuum, admitting dye liquor until the material is completely immersed, applying air pressure to assist penetration of dye liquor into the textile material, and removing excess of dye liquor by means of the centrifuge.

A. J. Hall.

Printing of cotton [with vat dyes]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 343,102, 14.8.29).— Relatively stable printing preparations are made by reducing a vat dye, in the absence of caustic alkali, with a reducing agent (hyposulphite, titanous chloride, etc.) and a weak alkali (ammonia, potassium carbonate) in presence of water and glycerin or other water-soluble alcohol (e.g., glycol, diglycol, thiodiglycol, thiodiglycerol, glycerol hydroxyethyl ether). The leuco-compound is present in insoluble form in stable suspension, which may be concentrated by evaporation. Hydrotropic substances, e.g., urea, sodium benzenesulphonate, phthalic anhydride, sodium dimethylaniline-m-sulphonate or naphthalene-2-sulphonate, may be added. The preparations are printed in the usual way with thickening agent and formaldehydesulphoxylate, steamed damp, and developed with dichromate and acetic acid.

C. HOLLINS.

Dry cleaning. L. E. Jackson (U.S.P. 1,778,948, 21.10.30. Appl., 8.4.29).—The development of rancidity in textile materials which have been dry-cleaned (due to their retention of free fatty acids from the dry-cleaning liquor) may be avoided by adding periodically to the liquor sufficient of an alkali to neutralise acid substances present in the materials being cleaned.

1. J. HALL.

Laundering methods. R. A. Phair, Assr. to H. Kohnstamm & Co., Inc. (U.S.P. 1,777,857—8, 7.10.30. Appl., [A] 11.6.23, [B] 7.10.24).—In a laundering process which comprises treatment of the goods with a liquor containing an alkali, the removal of this alkali in the final rinsing is facilitated by adding (A) ammonium salts, e.g., ammonium chloride and sulphate, or (B) ammonium sulphite, to the rinsing water.

A. J. HALL.

Treatment [delustring] of textile [cellulose ester and ether] materials. Brit. Celanese, Ltd. (B.P. 345,509, 31.3.30. U.S., 12.4.29. Addn. to B.P. 343,698; B., 1931, 486).—The fabrics etc. are treated as described in the prior patent except that they are suspended in the delustring liquor in the form of hanks in such a way that they are completely immersed.

A. J. HALL.

Manufacture of spangled, diamanté, and like fabrics, leather, and like flexible materials. M. I. Keller (B.P. 344,585, 7.1.30).—Fabric is coated with a caoutchouc adhesive, then dusted or sprayed with spangles, pressed, and excess of spangles removed, e.g., by suction.

A. J. Hall.

Treatment of textile materials. Brit. Celanese, Ltd. (B.P. 345,022, 28.1.30. U.S., 6.2.29).—Fabric having a cockled appearance is produced by weaving

with mixed yarns containing fibres of cellulose acetate or other organic derivative of cellulose and fibres of another material and then treating the fabric with a reagent which causes unequal shrinkage of the two types of fibres. Alternatively, the fabric is woven from similar mixed yarns already treated with the shrinking agent. When the mixed yarn consists of cotton and cellulose acetate silk and caustic soda is used for effecting shrinkage, the alkali should be used at about 0° so that hydrolysis of the silk is retarded. A. J. Hall.

Treatment [with ozone] of wool, fur, and other animal fibres, or textile fabrics containing them. S. W. Wilkinson (B.P. 345,406, 27.1.30).—To prevent over-ozonisation with consequent deterioration of the fibres when treated as in B.P. 317,133 and 242,027 (B., 1929, 848; 1926, 11), they are pretreated with gaseous or a solution of formaldehyde, with or without the presence of alkaline or acid solutions or gases.

H. ROYAL-DAWSON.

Treatment [sizing] of textile materials. Brit. Celanese, Ltd., H. Dreyfus, W. A. Dickie, and P. F. C. Sowter (B.P. 344,528, 27.8.29).—The materials are sized with aqueous solutions of water-soluble hydroxyor carboxy-alkyl ethers of cellulose or degraded cellulose. F. R. Ennos.

Waterproofing of fibrous products. F. Queck (U.S.P. 1,777,447, 7.10.30. Appl., 3.9.29).—Wood pulp is mixed with an emulsion of unsaponifiable substances such as asphalt or a blown asphalt (e.g., Parolite and Stanolite), the emulsion being prepared with the aid of a soap, particularly a rosin soap, and then an acid or acid salt is added to render the asphalt insoluble; fillers such as starch, lampblack, or calcium sulphate may also be added.

A. J. Hall.

Waterproofing compositions [for paper]. C. S. Shelton (U.S.P. 1,778,964, 21.10.30. Appl., 18.4.25).—Compositions consisting of a mixture of one or more metal oxides (zinc oxide and/or lead oxide), benzene, light petroleum, and dammar gum or sodium silicate solution are claimed.

A. J. Hall.

Impregnated and coated fabric. G. BAEKELAND, Assr. to BAKELITE CORP. (U.S.P. 1,776,879, 30.9.30. Appl., 28.1.26).—Rubber-coated fabrics such as are used in the manufacture of motor tyres, pressure hose, belting, etc. are manufactured from fabrics which are initially impregnated with a varnish which yields a flexible film of the phenol-methylene resinoid type; the bond between the subsequently applied coat of rubber and the fabric is thereby improved and the resulting products have increased tensile strength.

A. J. Hall.

(A) Aircraft covering. (B) Flexible impermeable fabric. I. M. Jacobsohn and S. Truscott (U.S.P. 1,779,846—7, 28.10.30. Appl., 5.3.26).—The base fabric (cotton cloth) is coated (A) with a mixture of dried rubber with carbohydrates (glucose), polyhydric alcohols or their derivatives (glycerol, triacetin), or gelatin, which has been milled and dissolved in an organic solvent (benzene), the rubber being afterwards vulcanised; or (B) with cuprammonium cellulose and, after coagulation of the latter with acid, is washed and plasticised by immersion in glycerin.

F. R. Ennos,

Coating of [balloon] fabrics. W. C. CALVERT, Assr. to Goodyear Tire & Rubber Co. (U.S.P. 1,779,388, 21.10.30. Appl., 2.12.27).—Flexible and durable fabric which is non-permeable to gases is prepared by coating a fabric successively with solutions consisting of 1 litre of water and (1) 60% rubber latex 200 c.c.; (2) 60% rubber latex 100 c.c., gelatin 30 g., and glycerin 60 g.; (3) 60% rubber latex 50 c.c., gelatin 60 g., and glycerin 120 g.; (4) gelatin 40 g., glycerin 80 g., and alum 2·4 g., drying between each coat, then sponging with water and applying a layer of gas-resistant Cellophane.

A. J. Hall.

[Fire- and mildew-]resistant [textile] material
[for mine-ventilating tubing]. F. A. McDermott and F. J. Funk, Assrs. to E. I. Du Pont de Nemours & Co. (U.S.P. 1,779,258, 21.10.30. Appl., 3.4.25).—The fabric is impregnated with a solution of zinc salicylate (or similar water-soluble antiseptic), dried, and then coated with a rubber composition containing paraffin, pine oil, zinc salicylate, and barytes; the whole is then vulcanised.

A. J. Hall.

Treatment of sacking and the like to prevent rotting of the same. F. H. Cotton, and Wakeley Brothers & Co., Ltd. (B.P. 345,468, 6.3.30).—Sacking material is impregnated with a 2% solution of copper sulphate tinted with a basic dye, particularly Brilliant Green Crystals Y, and then treated with a solution containing sodium silicate and common yellow soap, whereby copper stearate and silicate are precipitated as protective agents within the fibres. A. J. Hall.

Liquid treatment of yarn in hank or skein form. W. Gerber (B.P. 347,284, 27.1.30).

Treatment [packaging] of textile strands [to facilitate bleaching, dyeing, etc.]. E. J. Abbott (B.P. 346,895, 6.2.30).

Jigger machines for the dyeing or like treatment of fabrics. Maschinenfabr. Benninger A.-G. (B.P. 347,059, 26.6.30. Ger., 20.7.29).

Machines for treating textile fabrics [to produce moiré effects]. A. MILHOMME (B.P. 347,034, 23.5.30. U.S., 4.4.30).

[Shrinkage-preventing] treatment of textile fabrics [during laundering]. Tootal Broadhurst Lee Co., Ltd., and K. S. Laurie (B.P. 346,782, 16.1.30).

Wetting etc. agents.—See III. Designs on paper etc.—See V. Coloured rubber.—See XIV. Protecting leathers etc.—See XV. Photographic bleaching-out process.—See XXI.

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

Theory of the lead-chamber [sulphuric acid] process. "Blue acid" [SO<sub>5</sub>NH<sub>2</sub>]. E. Berl and H. H. Saenger (Z. angew. Chem., 1931, 44, 291).—Blue acid, formed by reduction of nitrosylsulphuric acid by sulphur dioxide, plays a part in most theories of the lead-chamber process. It can be produced as a blue coloration by cathodic reduction of concentrated sulphuric acid containing SO<sub>5</sub>NH<sub>2</sub> at an E.M.F. of 3—4 volts, and by passing nitrogen peroxide and water

vapour into liquid sulphur dioxide at  $-70^{\circ}$ . At ordinary temperatures it rapidly decomposes:  $SO_5NH_2 = H_2SO_4 + NO$ . This latter reaction has been shown experimentally to be reversed at pressures of the order of 250 atm., when up to 77% combination as blue acid has been obtained. C. IRWIN.

Preparation of hydrogen cyanide by oxidation of ammonium thiocyanate or thiocyanic acid with nitric acid. W. GLUUD and K. KELLER (Ber. Ges. Kohlentech., 1931, 3, 395-419).-The principal reaction is RCNS +  $2HNO_3 = HCN + 2NO + RHSO_4$ . A 20% solution of thiocyanate is run into a stirring vessel with nitric acid, the latter being kept in excess; the temperature must be approx. 100°. The gases are scrubbed with water at 80°, which produces nitrous acid, and are then passed into an alkaline absorbing solution. The remaining nitric oxide is regenerated for further use. This process was originally worked out in England, using sodium or calcium thiocyanate. The authors in applying it to the ammonium salt find that for satisfactory yields in this case a minimum concentration of 1.5 g. of acid ( $d \cdot 1.14$ ) per 100 c.c. must be maintained in the liquor. Losses of nitric oxide occurred and formation of ammonium nitrite with subsequent decomposition was frequent. On this account the oxidation of thiocyanic acid formed by decomposition of the ammonium salt with the sulphuric acid produced was The solution of the free thiocyanic investigated. acid decomposes in a few hours. Decomposition is retarded by dilution and by cooling with ice. It must therefore be oxidised as soon as it has been prepared. The process proposed is to treat a 25% ammonium thiocyanate solution with 78% sulphuric acid and oxidise with excess of nitric acid (d 1.20). After saturation of the residual liquor with ammonia "Leuna saltpetre" is obtained. The decomposition is carried out in a stoneware or ebonite-lined apparatus. Alternatively, a 17% solution of thiocyanic acid is to be oxidised with nitric acid in a similar apparatus. Laboratory experiments are described in detail and yields and water, steam, and power consumptions calculated.

C. IRWIN.

Preparation of ammonium sulphate in coarse tabular crystals. W. GLUUD, W. KLEMPT, and H. RITTER (Ber. Ges. Kohlentech., 1931, 3, 371-384).-It has been shown that the presence of iron as an impurity is responsible for the production of ammonium sulphate in the fine acicular form usually met with in the coke-oven product. The variation of crystalline form with variation of temperature, method of evaporation, and speed of agitation was studied in the laboratory. Little difference in the product was observed except that with very low speeds of agitation a powdery crystal was obtained as from stationary crystallisation. Further tests with mother-liquor from a coke-oven ammonium sulphate plant showed that the crystal form was not due to the presence of tar or tar products or of arsenic. Iron, however, which is present in the ferrous state, and is not precipitated by excess of ammonia, is important. If it was oxidised with hydrogen peroxide and then removed by precipitation the salt was obtained as coarse tabular crystals. On

adding ferrous ammonium sulphate to the purified liquor, crystals of the fine needle type were obtained. The iron may equally be removed as sulphide. Ferric iron (which is reduced under coke-oven conditions), chromium, and aluminium are all harmful, giving a paste of fine crystals. The action of ferrous iron appears to be due to adsorption which prevents crystal growth except in one direction. A process was worked out in the laboratory and on a semi-works' scale for the elimination of iron by atmospheric oxidation, keeping the saturator alkaline. On the larger scale the use of air was afterwards supplemented by hydrogen sulphide treatment, using untreated gas. The precipitated iron was removed by a coke filter or centrifuge, and the liquor passed to a vacuum evaporator. It was found possible to work without the use of air-blowing, and the iron sulphide was easily separated. It was essential to maintain 0.09% free  $NH_3$  in the liquor. Air oxidation requires 0.3—0.4% free  $NH_3$  and a temperature of 60-80°, and the ferric hydroxide is not so easy to separate. The crystals of salt obtained, though of the desired form, were fine owing to the rate of evaporation exceeding the rate of growth of the crystals. This was altered by changing the evaporator design, after which about 80% was obtained above 0.5 mm. in diam. The application of this process to the direct, indirect, and semi-direct sulphate processes is described and costs are discussed. C. IRWIN.

Production of superphosphate from Khibinsk apatite. S. I. Volfkovich, L. E. Berlin, and L. B. Grinshpan (Udobr. Urozhai, 1930, 2, 300—312).— Enriched apatite obtained from nephaline apatite rock by fine grinding and sieving or flotation is used. When 88% of the rock is milled to less than 0.1 mm. the material (40%  $P_2O_5$ ) gives an acid phosphate with 18% of soluble  $P_2O_5$ . Chemical Abstracts.

Conversion of cyanide solution into potassium ferrocyanide and the oxidation of the latter to ferricyanide by compressed air and electrolysis. W. GLUUD [with W. KLEMPT, K. KELLER, F. BRODKORB, and C. DIECKMANN] (Ber. Ges. Kohlentech., 1931, 3, 385-394).—The problem investigated was the utilisation of hydrogen cyanide present in a gas in large quantities. This compound was first converted into calcium cyanide and the slightly alkaline solution at 80° stirred, with gradual addition of concentrated ferrous sulphate slightly in excess of the theoretical. It was filtered from calcium sulphate and the filtrate treated with potassium carbonate solution with heating. After again filtering, the filtrate was evaporated in vacuo and the calcium carbonate used for further cyanide absorption. The yield of ferrocyanide in the laboratory was 97%. The oxidation of ferrocyanide is reversed by the potassium hydroxide liberated in the reaction, and means must be used to prevent alkalinity developing. Chlorine is not suitable owing to the difficulty of separating the potassium ferricyanide from chloride. Other reagents are expensive, and the authors propose the use of air with carbon dioxide at high pressure, which process is, however, not quantitative unless supplemented by other methods. Experiments conducted in a small autoclave at 50 atm. showed that

sulphuric acid was less effective than weaker acids or carbon dioxide. The optimum temperature is 100°, and little variation in results occurred between 20 atm. and 50 atm. pressure. Using oxygen and carbon dioxide, 10 atm. is sufficient, and the theoretical quantity only of carbon dioxide is necessary for 90-95% oxidation. A 20% calcium ferrocyanide solution is most convenient for treatment, and good agitation is necessary. In order to complete oxidation, the treated solution is electrolysed at 50° between nickel electrodes at a current density of 0.005-0.1 amp./cm.<sup>3</sup>, and an E.M.F. of 7-12 volts. Solid calcium oxide is added to the anode solution to neutralise the free acid formed. The current efficiency is about 50%. The solution is then treated with the equivalent of potassium carbonate, filtered, and evaporated in vacuo. The whole process can be carried out with a yield of 90-92%. C. IRWIN.

Complex cyanides of nickel, copper, and chromium. W. Gluud and W. Riese (Ber. Ges. Kohlentech., 1931, 3, 452—459).—Titration with potassium iodide and silver nitrate indicates that a ferrocyanide is decomposed by ammonia with formation of

cyanide and complex cyanides containing ammonia. A similar action occurs with potassium nickel cyanide. In this case the final products are KCN and [Ni(NH<sub>3</sub>)<sub>4</sub>](CN)<sub>2</sub>, all the cyanide being capable of titration with silver nitrate. The nickel salt does not, however, react with ferrous cyanide to form ferrocyanide. Copper acts similarly. With ammonium polysulphide, on the other hand, no cyanide is formed, but the final products are nickelous sulphide and ammonium thiocyanate. Copper and chromium react similarly, but less readily. With ferrous sulphate a white compound, Fe[Ni(CN)<sub>4</sub>] is produced, or, in alkaline solution, ferrous hydroxide.

The copper and especially the chromium complex cyan-

ides appear to produce some ferrocyanide with ferrous

sulphate.

Determination of polysulphide and preparation of calcium sulfuratum solutum (Vlemingkx' solution). J. Bücht (Pharm. Acta Helv., 1930, 5, 124—132; Chem. Zentr., 1931, i, 978).—A solution of boric acid (1 g.) in water (60 c.c.) is boiled, 0·2 g. of potassium cyanide and 1 c.c. of the solution being successively added; after 10 min. the liquid is cooled and diluted to 100 c.c. A portion (50 c.c.) is treated with concentrated hydrochloric acid (1—2 c.c.) and excess of bromine water, the excess being removed with phenol. After 10 min. potassium iodide (1 g.) is added, and after a further 15 min. the iodine is titrated with thiosulphate.

A. A. ELDRIDGE.

C. IRWIN.

Solubility of Bordeaux mixture. G. L. Hockenvos (Phytopath., 1931, 21, 231—234).—The method of
Callan and Henderson (A., 1930, 53) is applied to the
examination of Bordeaux mixtures. Any turbidity
produced during the colour test by the presence of calcium salts is avoided by acidifying the test solution with
hydrochloric acid, making alkaline with ammonia, and
adding saponin solution to peptise colloidal matter, prior
to the addition of the diethyldithiocarbamate reagent.
More metallic copper dissolves in boiled-out distilled
water than in aerated water. The proportion of dis-

solved copper present in a number of Bordeaux mixtures is determined. The minimum solubility (0.00009%) is reached in a 4:1.08:50 preparation, i.e., a Cu: Ca(OH)<sub>2</sub> ratio of 1:0.27. Addition of 0.5% of sugar to the Bordeaux mixture raised the copper solubility to 0.078%.

A. G. POLLARD.

Determination of sulphate in chrome liquors.

O. Macchia (Ind. chimica, 1930, 5, 1346—1351; Chem. Zentr., 1931, i, 818—819).—The liquid (20 c.c.) is gently warmed with acetic acid (20 c.c.) and tartaric acid (3·2—3·5 g.), and then treated with hydrazine or hydroxylamine hydrochloride until effervescence is no longer produced. After dilution with water (150 c.c.) the solution is boiled and the sulphate is precipitated with barium chloride.

A. A. Eldridge.

Chlorometry. Definition of chlorometric value.
H. Lestra (Bull. Sci. pharmacol., 1930, 37, 300—308;
Chem. Zentr., 1931, i, 973).—The chlorometric value is
unchanged when hydrochloric acid or chlorides are
present. The chlorometric value (English) expresses in
g. of chlorine the oxidising power of 100 g. of hypochlorite.

A. A. Eldridge.

Removal of hydrogen sulphide and cyanide from benzol, and of cyanide from coke-oven gas. See II. Soda recovery in paper mills.—See V. Basic slag.—See XVI.

See also A., May, 555, System AlCl<sub>3</sub>-NaCl. 562, Silica gel. 577, Sodium and lead arsenates. 581, Aluminium hydroxide. 582, Cerium sulphide. 583, Rhenium. 588, Micro-determination of calcium and magnesium. Tartar emetic. 638, Detection of carbon disulphide.

PATENTS.

Manufacture of nitric acid. Lodge-Cottrell, Ltd. From Metalles. A.-G. (B.P. 345,591, 27.6.30).—The vapours containing the last traces of nitric acid are passed through irrigating absorption towers and then subjected to electrical precipitation in a single- or multistage apparatus arranged between these towers.

H. ROYAL-DAWSON.

Preparation of concentrated nitric acid. N. Caro and A. R. Frank (B.P. 344,826, 6.12.29. Cf. B.P. 342,068; B., 1931, 392).—Aluminium or an alloy containing it is used in the construction of pressure vessels etc. for use in the process of the prior patent. Such vessels may be (a) provided with a ceramic lining, (b) packed with aluminium rings etc., and (c) applied direct to supporting walls or have an intervening gas space filled with a compressed non-reacting gas.

L. A. Coles.

Treatment of [residual] ammoniacal liquors [from the ammonia-soda process]. D. R. Means, Assr. to Pittsburgh Plate Glass Co. (U.S.P. 1,781,987, 18.11.30. Appl., 20.9.26).—The liquor, after decomposition of any ammonium carbonate present by treatment with steam in the upper section of a tower, is conducted from an intermediate point in the tower to the lower end of a cylindrical tank in which it is treated with unground quicklime containing, e.g., 5—12% of unburnt limestone. The lime, which is fed in at the top of the tank, is swept down a series of trays by a rotating

agitator, and residual unburnt stone is removed from the bottom. The treated liquor is returned from the top of the tank to the tower for recovery of the ammonia.

L. A. Coles.

Furnaces for production of sulphates and hydrochloric acid. H. Frischer (B.P. 344,801, 10.12.29).— In furnaces for this purpose, the bottom, walls, pipes, stirring and scraping devices, and other such parts as come in contact with molten sulphates resulting from the interaction of chlorides and sulphuric acid are made from silicon carbide or materials containing it.

H. ROYAL-DAWSON.

Treatment of [production of alkali aluminates or pure alumina from] bauxite, alunite, and like aluminous material. Sulphates Proprietary, Ltd. (B.P. 344,959, 21.12.29. Austral., 22.12.28).—A mixture of the material with an alkali hydrogen sulphate or with an alkali sulphate and sulphuric acid (Na<sub>2</sub>O: Al<sub>2</sub>O<sub>3</sub> = 1·1—1·5:1 mol.), worked up into briquettes or nodules, is heated in a vertical shaft furnace in the presence of a countercurrent of a reducing gas (watergas), and the product, after removal from the furnace, is treated in thin layers with an oxidising gas and then lixiviated to extract the alkali aluminate.

L. A. Coles.

Manufacture of high-grade zinc oxide. F. E. Pierce (U.S.P. 1,781,702, 18.11.30. Appl., 9.7.21. Renewed 30.3.28).—Zinc vapour produced by smelting zinc ores or the crude oxide in a reverberatory furnace is burned at 1090—1290° by admixture with preheated air in a combustion chamber connected with the furnace; the oxide, which leaves the chamber at 600—700°, is drawn by a fan through a flue or trail in which it is cooled to about 260° by the admission at intervals of cold air, and thence passes successively through a cyclone or centrifugal separator to remove impurities and gritty particles of oxide, a bag house, and a furnace in which it is reheated for about ½ hr. at 550—600°.

L. A. Coles.

Manufacture of finely-divided antimony trioxide. Deuts. Schmelz-u. Raffinierwerke A.-G. (B.P.345,034, 6.2.30).—The upper surface of a stationary charge of antimony, or of a mixture of ores which on roasting will give oxides of antimony, is heated to about 1000° and the metal is burned or the ore roasted in air diluted with inert gases (carbon dioxide, nitrogen, or combustion gases).

H. Royal-Dawson.

Refining of heavy spar. Metallges. A.-G. (B.P. 345,186, 21.7.30. Ger., 28.8.29).—The spar, after dressing if necessary, is calcined at 1000—1200° under oxidising conditions, cooled dry, treated with hot (preferably hydrochloric) acid, and washed with water and finally with dilute ammonia solution. Alternatively, the ore is calcined to burn away any bitumen and then treated directly with the acid. L. A. Coles.

Production of hydrogen. F. T. SNYDER (U.S.P. 1,781,935, 18.11.30. Appl., 16.12.25. Can., 12.3.25).—Natural gases, gas mixtures containing hydrocarbon vapours, etc., and a current of air are passed alternately, but in opposite directions, through a furnace filled with refractory chequerwork so that the carbon deposited

during the cracking stage is burned during the combustion stage to maintain the chequerwork at about 980°.

L. A. Coles.

Apparatus for decomposing water into hydrogen and oxygen gases. S. Bessho and T. Sakakibara (B.P. 345,149, 14.5.30. Jap., 28.9.29).—Water maintained at a constant level by a float regulator in a vertical cylindrical chamber passes up a narrow annular space between two concentric axially-disposed tubes, and is vaporised therein by an electrically-heated nichrome wire in the inner tube; the vapour passes into a compartment above the cylinder in which it is decomposed by an electrically-heated platinum wire stretched around the chamber in the form of a polygon.

L. A. Coles.

Manufacture of hydrogen peroxide. J. B. Pierce, Jun., Assr. to Barium Reduction Corp. (U.S.P. 1,781,859, 18.11.30. Appl., 14.12.27).—Strontium peroxide, prepared, e.g., as described in U.S.P. 1,325,043 (cf. B.P. 130,840; B., 1919, 719 a), is treated with an equivalent weight of cold, dilute sulphuric acid and a small quantity  $(0\cdot 2-0\cdot 1\%)$  of the wt. of  $H_2SO_4$ ) of an acid yielding a soluble strontium salt, and the strontium sulphate is removed by filtration. L. A. Coles.

Recovery of sulphur from pyrites. R. F. BACON (U.S.P. 1,782,225—6, 18.11.30. Appl., [A] 6.4.27, [B] 13.4.27).—(A) The pyrites is heated in a rotating furnace at 600—700° in the presence of steam, the sulphur is deposited in a condenser maintained above 100°, and the residual steam, after preheating, is returned to the furnace. Sufficient sulphur dioxide to interact with any hydrogen sulphide present is also introduced into the condenser. (B) Part of the sulphur is recovered from the pyrites as described in (A), and the remainder is removed as sulphur dioxide by heating the residues in an air blast in a separate furnace. The gaseous products from the two sets of furnaces may pass into a common condenser so that the hydrogen sulphide from the one set reacts with the sulphur dioxide from the other, or the hydrogen sulphide may be stored for treatment with sulphur dioxide as required.

L. A. Coles.

Preparation of chlorine dioxide. W. Becher (B.P. 345,190, 26.8.30. Holl., 26.8.29).—The gas is prepared by the action below 65° of concentrated sulphuric acid on prepared lumps comprising chlorates and non-reacting material, e.g., gypsum; a catalyst (oxide of manganese, vanadium, iron, or osmium) and a carbonate may be added to the lumps, and a non-reacting gas, which may be saturated with an organic reducing agent (formaldehyde), may be passed through the reacting mixture.

L. A. Coles.

Apparatus for compressing carbonic acid snow. Maschinenfabr. Esslingen, and J. Stoffels (B.P. 347,050, 12.6.30. Ger., 20.9.29. Addn. to B.P. 334,682).

Installations for endothermic reactions. Sp. gr. of battery acid. Gas producer. Liquefaction of gases.—See I. Ammonia etc. from gases. Sulphur from gases.—See II. Lithopone. Titanium pigments.—See XIII. Manures etc.—See XVI. Silicyl compounds.—See XX.

# VIII.—GLASS; CERAMICS.

Variations caused in the heating curves of glass by heat treatment. A. Q. Tool and C. G. Eichlin (J. Amer. Ceram. Soc., 1931, 14, 276—308).—Heating curves have been obtained for a series of glasses which represented the progression of the change taking place during the annealing of a glass. A particular glass representative of its type (chilled) gave a heating curve which rose rapidly between 350° and 550° and then declined to a lower level at a somewhat higher temperature. These phenomena are due to an exothermic effect followed by either an endothermic effect or a rapid change in sp. heat and conductivity. Another glass (annealed) showed no exothermic effect, but exhibited a marked decline at the higher temperature, which is consequently definitely attributed to an endothermic effect. The determination and interpretation of the heating curve is discussed at length, especially in connexion with annealing. Among the many points which are noted, the importance of the following facts in practical annealing is emphasised: Properties such as density, refractive index, etc. may be varied 1% by suitable treatment in the lower part of the annealing range, and such manipulation may be utilised to correct differences in property resulting from slight variation between the compositions of different melts. Incidentally great caution is needed, in determining the effect of a change in certain constituents on a particular property, that the same annealing schedule is followed. To produce a homogeneous product temperature gradients should not exist during the annealing process. J. A. SUGDEN.

Expansion measurements of several glasses by means of a self-registering apparatus. W. M. Cohn (J. Amer. Ceram. Soc., 1931, 14, 265—275).—
The apparatus (which is described) records photographically the expansion relative to vitreous silica, and reveals clearly certain inflexion points in the temperature—expansion curve which are liable to be undetected by visual observation. Although one glass (a special apparatus glass) showed no inflexion point and another (a tube glass) two points, most glasses showed only one. The annealing range of a glass is defined by the softening and inflexion points. Complete analyses of the glasses are given.

J. A. Sugden.

Electrochemistry applied to glass. A. Sinverman (Trans. Amer. Electrochem. Soc., 1931, 59, 183—191).— Applications of electrochemistry in the glass industry include chromium-plating of moulds, plating on glass for decorative purposes, and conductivity determinations as a measure of the viscosity of glass. The results of investigations on electrolytic conduction and ionic migration through glass are reviewed.

H. J. T. ELLINGHAM.

Comparison of bodies containing blended felspars and one-mine felspar of similar composition.

C. W. Parmelee and C. R. Amberg (J. Amer. Ceram. Soc., 1931, 14, 309—312).—An electrical porcelain body was made up with single and blended felspars of similar composition and fired at cones 7—13. Measurement of the volume shrinkage and modulus of rupture showed that single and blended felspars were not the same in effect. This is due to variations in the soda—

lime ratio of the plagioclase and the degree of weathering. It is recommended that blending be restricted to felspars from the same mine.

J. A. Sugden.

Tshasov-Jar clays. K. Köhler (Trans. Ceram. Res. Inst., Moscow, 1929, No. 20, 88—150).—The characteristics of the clay from the point of view of its use in the ceramic industry have been investigated. A rational classification of clays is proposed.

CHEMICAL ABSTRACTS.

Use in ceramic industry of the sandy Tshasov-Jar clay called "balyki." P. N. KAVOKIN (Trans. Ceram. Res. Inst., Moscow, 1929, No. 20, 172—181).— The use of this clay, previously discarded, for the manufacture of stoneware and firebrick is described.

CHEMICAL ABSTRACTS.

Influence of high temperature on Prosyanaya kaolin. V. V. Yurganov and M. V. Zusmanovich (Trans. Ceram. Res. Inst., Moscow, 1929, No. 21, 28—55).
—At 880° metakaolin is formed; at 980° kaolin decomposes into free silica and the sparingly soluble form of clay, and shows the first sign of formation of one or more aluminium silicates. At 1050—1100° there is little change except further combination of free silica and alumina. No sillimanite was found. At 1200° the sparingly soluble residue, 5Al<sub>2</sub>O<sub>3</sub>,4SiO<sub>2</sub>, increases; at 1320° its composition is 4Al<sub>2</sub>O<sub>3</sub>,3SiO<sub>2</sub>. Kaolin burned at 1400—1470° (3Al<sub>2</sub>O<sub>3</sub>,2SiO<sub>2</sub>), when examined in thin layers, exhibits crystals.

Weathering of plastic fireclays. S. M. Phelps and C. G. Denney (J. Amer. Ceram. Soc., 1931, 14, 319—324).—Extensive tests were made on clays stored in the laboratory, on the same clays after 2—3 years' weathering on the roof of a building, and on commercially weathered clays. The properties studied were water of plasticity, plasticity, drying and firing shrinkages, refractoriness, loss on ignition, transverse strength, sieve analysis, and rate of settling. Weathering appeared to have no appreciable effect. It is concluded that either there is no satisfactory method of measuring the changes or that the weathering effect is over-estimated and that it can be reproduced to a large degree by finer grinding and treatment with electrolytes.

J. A. Sugden.

Currents and temperatures in glass tanks. I. G. Gehlhoff, W. Schneekloth, and M. Thomas (J.

Soc. Glass Tech., 1931, 15, 12—29 T).

Silicon carbide.—See I.

See also A., May, 546, Glass for ultra-violet rays. 579, Coloration of silicate glasses. 593, Filtering discs. 595, Minerals of the clay group.

PATENTS.

Making sheet glass. Mississippi Glass Co., Assees. of M. L. Begeman (B.P. 346,927, 5.3.30. U.S., 21.5.29).

Apparatus for working fused silica. Brit. Thomson-Houston Co., Ltd., Assees. of G. A. Millar (B.P. 346,940, 13.3.30. U.S., 13.3.29).

Preparation [drying etc.] of ceramic articles. W. and M. LENGERSDORFF (B.P. 347,020, 9.5.30. Addn. to B.P. 328,777).

Treatment of laminated materials.—See I. [Adhesive] compositions.—See XIII.

#### IX.—BUILDING MATERIALS.

Suitability of tar oils for the impregnation of mine timber. W. Engels (Chem.-Ztg., 1931, 55, 285—286, 307—308).—A comparison is made between tar oils and a number of preparations consisting of salt solutions possessing germicidal action. Besides having certain disadvantages such as objectionable odour, action on the skin, and risk of fire, tar oils, which depend for their action mainly on their tar acid content, afford protection to the wood for only a limited period compared with the salt solutions.

F. R. Ennos.

#### PATENTS.

Rotary cement kiln. M. Vogel-Jorgensen (B.P. 345,731, 21.1.30).—The total inside area of a rotary dry cement kiln is at least 120 times as large as the internal cross-sectional area of the kiln at normal diameter, and for a wet kiln this figure excludes the drying section of the kiln. The length of the kiln may be adjusted, maintaining the normal diameter in the preheating and calcining zones, which preferably are fitted with some form of agitator. The sintering zone may be enlarged and the preheating and calcining sections split up into a number of tubes to effect better contact of the material with the hot gases.

C. A. King.

Production of cementitious material. G. O.
Case, E. M., Ellis, and L. H. Montique (B.P. 345,145,
24.12.29. Cf. B.P. 330,278; B., 1930, 770).—A puzzuolanic
cement is produced by mixing 25% of calcium (and/or
magnesium) oxide with 75% of the product obtained by
heating an aluminium silicate, e.g., slate, clays, or
shale, at about 750°. Calcium sulphate (5—15%) may
be incorporated as an accelerator. C. A. King.

Composition for waterproofing cement. F. W. Huber (U.S.P. 1,772,999, 12.8.30. Appl., 7.8.28).— High early strength and proofing of cement is attained by mixing the cement batch with an emulsion containing an oil (petroleum) as the external phase and an accelerating solution of an alkali or alkaline-earth salt, or an acid which will produce such a salt by reaction.

C. A. King.

Surface treatment of concrete or other materials pervious to moisture. E. O. Cowper (B.P. 345,671, 18.12.29 and 24.5.30).—A mixture of a dilute emulsion of rubber latex (with preservative, if desired) and dilute sodium silicate solution is sprayed on to concrete, brick, etc. and penetrates before the latex coagulates. Approximately equal quantities of emulsion and solution are used, the degree of dilution being never less than 2:1 in the case of either ingredient (latex or silicate).

Manufacture of porous building materials. A. V. Carlson (U.S.P. 1,782,460, 25.11.30. Appl., 6.8.26. Swed., 19.8.25).—A mixture comprising acidic material (pumice, argillaceous schist, hydraulic residues, cements, etc.), sufficient unslaked lime to give a foundation mass, and material for evolving gases (powdered metals or carbides, and, if desired, alkalis) is hydrated by the addition of water or steam, and the product is moulded and then hardened by treatment with hot air etc.

HILL some entities of L. A. Coles.

Colouring of slate granules. H. C. FISHER, ASST. to PHILIP CAREY MANUFG. Co. (U.S.P. 1,782,648, 25.11.30. Appl., 29.4.25).—The granules are wetted with a solution containing compounds which form hydroxides and then oxides on roasting, e.g., with a solution containing ferrous sulphate (6 mols.) and sodium dichromate (1 mol.), which gives a brown colour on roasting. The colour may be modified by varying the proportion of the compounds and the oxidising or reducing properties of the roasting gases.

L. A. Coles.

Making of cold tar road coverings. D. J. Pickée (B.P. 346,025, 30.12.29. Ger., 23.8.29).—Tarry substances are prepared for cold application to road surfaces by admixture with hydrated lime. The lime is preferably used in aqueous solution or suspension, and its weight should be 10—12% of that of the asphaltic material. The viscosity of the mixture may be reduced by adding a solvent such as benzene. Dry lime may also be added to the tar, and the necessary water then stirred in or added on the road surface.

R. H. GRIFFITH.

Cold bituminous paving composition. O. H. Berger, Assr. to W. P. McDonald Construction Co. (U.S.P. 1,776,763, 23.9.30. Appl., 14.7.28).—A hard asphalt containing 60% or more of bitumen, e.g., raw Cuban asphalt, is mixed with a "fresh oil" (petroleum containing a fair proportion of volatile oil) and then with a mineral base. Finally 3—5% of water and "fresh oil" together are incorporated until the mixture is light brown in colour. C. A. King.

Plant for manufacture of artificial wood. O. Kayser (B.P. 345,191, 10.6.30).—A conveyor belt is led under the lower part of a filter drum, and a closed conduit supplies wood pulp, preferably tangentially, between the filter and the band. C. A. King.

Preservation of wood. D. Steinherz, Assr. to Grubenholzimprägnierung Ges.m.b.H. (U.S.P.1,777,235, 30.9.30. Appl., 27.6.29. Ger., 3.7.28).—Wood is impregnated with a water-soluble substance and then with a separate solution which will reduce the solubility of the first salt. Thus a 2% solution of sodium fluosilicate or an aqueous solution of sodium 2-napthalenesulphonate may be followed by a 4% solution of sodium chloride. C. A. King.

[Rubber] floor coverings. N. Kenyon, and Spen Rubber Works, Ltd. (B.P. 344,956, 19.12.29).—In a floor covering comprising a layer of rubber united by vulcanisation with a backing consisting of a vulcanised mixture of rubber and wood flour, integral portions of the backing extend through the rubber layer and form part of the tread surface.

D. F. Twiss.

Furnace. Separation of granular etc. materials.
—See I.

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

Influence of oxygen and sulphur on the malleability, hot-shortness, and other properties of pure iron. E. W. Fell (Arch. Eisenhüttenw., 1930—1, 4, 393—400; Stahl u. Eisen, 1931, 51, 386—387).—Armco iron containing up to 0.07% O can be readily hot-forged

after melting in vacuo, but the presence of 0.01— 0.02% S considerably reduces the malleability at a red heat; with 0.03% O and 0.03% S the metal is definitely hot-short. After melting in an oil-fired furnace, however, iron relatively rich in sulphur and oxygen forges well if the treatment is commenced at a high temperature, e.g., 1200°. Alternate bending tests at 1100— 1150° of square rods on the anvil showed the marked superiority of vacuum-melted metal over metal melted in an oil-fired furnace. Hot-shortness above 800° is shown by iron containing more than 0.03% O and 0.02% S; thus metal with 0.054% O and 0.01% S is distinctly hot-short in the bending test although it forges well. Notched-bar impact tests confirmed the deleterious effect of a combined oxygen and sulphur content above about 0.04%. The depth of penetration of the carbide layer in the cementation test is highest with pure iron and decreases with increasing oxygen content. A. R. POWELL.

"Through improvement" of constructional steels with reference to the effect of cross-sectional area and alloying constituents. H. KALLEN and H. Schrader (Arch. Eisenhüttenw., 1930—1, 4, 383—392; Stahl u. Eisen, 1931, 51, 387).—The term "through improvement" (Durchvergütung) is applied to that process of treating a steel which produces a structure such that the yield point is constant throughout the cross-section of the specimen. The "state of improvement" (Vergütungszustand) of a steel is characterised by the relation between the yield point and tensile strength, and is determined by the proportion that the area of improved structure bears to the total area of the cross-section of the specimen. Numerous technical steels in specimens 20-200 mm. in diam, have been tested to determine their "state of improvement" and the degree of "through improvement," and from the results obtained the various technical steel alloys are divided into five classes. Alloy steels with a high nickel and chromium content are in the most satisfactory condition whatever their cross-sectional area, and plain carbon steels are usually the least uniform in their A. R. POWELL. mechanical properties.

Indirect determination of silicon in 48—52% ferrosilicons. G. T. Dougherry (Ind. Eng. Chem. [Anal.], 1931, 3, 158—159).—Practical details are given of a rapid method of determining silicon in ferrosilicons by difference instead of directly. E. S. Hedges.

Cause of the "iron disintegration" of blast-furnace slags. A. Guttmann and F. Gille (Arch. Eisenhüttenw., 1930—1, 4, 401—410; Stahl u. Eisen, 1931, 51, 432—433).—The so-called "iron disintegration" of slags is due to the presence of finely-divided iron and manganese sulphides in the slag; these sulphides in contact with moist air become hydrated and increase in volume by about 38%, thus causing the slag to break up into scales and crusts on the surface. This action occurs only when the slag contains at least 1.5% FeO and 0.5% S and when it has been left in contact with the molten iron in the settler or mixer for a long time. To prevent such slags from disintegrating they may be heated in air at above 1000° or, better, treated with sand before they solidify. A. R. Powell.

Rapid volumetric determination of manganese in ores and alloys. I. Majdel (Arh. Hemiju, 1931, 5, 45—49).—The finely-powdered material (1 g.) is fused with 6 g. of sodium peroxide in an iron crucible, the cold mass extracted with warm water, and the solution, without filtering, boiled for 5—10 min. with, if necessary, the addition of a few drops of alcohol to destroy manganate and permanganate; 50 c.c. of 0·1N-oxalic acid and 50 c.c. of 1:5 sulphuric acid are added to the hot liquid to dissolve the ferric hydroxide and manganese dioxide, followed by 10 c.c. of phosphoric acid (d 1·7) to decolorise the iron salt, and the excess of oxalic acid is titrated with permanganate.

A. R. POWELL.

Removal of antimony from white metals. R. Thews (Metallbörse, 1930, 20, 1545—1546; Chem. Zentr., 1930, ii, 2434).—Removal by addition of sodium, aluminium, or zinc, which form alloys or mixtures with the antimony, is discussed. Iron and magnesium are unsuitable.

A. A. Eldridge.

Rapid test of corrosion-resistance. L. W. HAASE (Chem. Fabr., 1931, 169—170, 184—185).—Existing methods are criticised, mainly because they take no account of the oxygen content of the corrosive fluid and do not continuously renew the fluid. In the electrometric method described the fluid, at constant head, is fed in succession through two vessels, each of which contains the material under test and a platinum electrode; the liquid entering the second vessel is previously saturated with oxygen. The electrical apparatus is so arranged that each pair of electrodes is connected either to the measuring instrument or across a high resistance; it is thus possible to compare the corrosion-resistance of a material towards two different liquids or of two different materials towards one liquid. With a suitable choice of the velocity of flow of the liquid comparative results may usually be obtained within 24 hrs. The rate of formation of a protective layer may also be determined. The applicability of the method to materials of the same chemical composition, but different in structure, e.g., alloys before and after heat treatment, and the interpretation of the results obtained, are H. F. GILLBE. described.

Corrosion of the tinplate container by food products. T. N. Morris and J. M. Bryan (Dept. Sci. Ind. Res., Food Invest., Spec. Rept., 1931, No. 40, 85 pp.).—The previous literature on corrosion is reviewed and the apparatus used in this investigation is described. The condition of the metal surface is shown to be important. A large number of tests are described showing the effect of  $p_{\rm H}$  and of oxygen on the corrosion of tin, iron, and tinplate, from which it is concluded that low acidity leads to greatest corrosion of tin, and the presence of air especially at high  $p_{\rm H}$  induces corrosion of iron. Sugars, colloids, and tin salts are good inhibitors of corrosion, but traces of sulphur compounds act as accelerators. The discoloration of foodstuffs by tin and iron salts is discussed and conditions favourable to the production of hydrogen "swells" and perforations are described with suggested remedial measures. The report closes with an appendix on the examination of canned foods and an extensive bibliography. E. B. Hughes.

Studying the stress-strain relation in the notchedbar impact test. T. Kawai (Sci. Rep. Tôhoku, 1930, 19, 727-743).—A Charpy machine is used to apply a small impact repeatedly to a test-piece and the bending at each impact is measured until the test-piece is broken. From these observations the energy-bending relation in the test is calculated and the stress-bending relation obtained therefrom by evaluating the tangent at each point on the curve representing the former relationship. W. E. DOWNEY.

Effect of cold-working on the density and electrical resistance of metals. T. Uêda (Sci. Rep. Tôhoku, 1930, 19, 473—498).—The change in the density and electrical resistance of Swedish steel, copper, and brass, when stretched by a tensile-testing machine have been measured. The density of steels always decreased with an increasing tension, but a discontinuous decrease occurred in low-carbon steels at the yield point. The sp. electrical resistance always increased with the tension, at the yield point a discontinuous increase in the case of low-carbon steels and a decrease of resistance in that of medium- and high-carbon steels being observed. In the case of copper and brass the decrease of density and increase of sp. electrical resistance increased in magnitude with tension; in this latter case, however, discontinuity was not observed at the yield points as in the case of carbon steels. W. E. DOWNEY.

Electrolytic refining of aluminium and production of aluminium coatings in an aluminium chloride-sodium chloride bath. W. A. PLOTNIKOV, N. S. FORTUNATOV, and W. P. MASCHOWETZ (Z. Elektrochem., 1931, 37, 83-88).—Electrolysis of mixed aluminium and ammonium chlorides at 170° yields a crystalline deposit of the metal, which, however, reacts with the bath. Aluminium containing Fe 0.043-0.060%, Si 0.023%, and Na 6.015% may be obtained by electrolysing a bath containing 1.5—2 mols. of sodium chloride per mol. of aluminium chloride at 160-180° with an aluminium cathode. The deposit is crystalline and spongy, and is not improved by variation of the temperature nor by the use of a rotating cathode. Addition of alkaline-earth chlorides merely raises the m.p. and reduces the conductivity of the bath without altering the nature of the deposit; no improvement results from replacement of sodium chloride by potassium chloride, or from addition of a variety of other substances. The presence of lead chloride aids the formation of a coherent deposit, which, however, always contains lead. On copper and iron cathodes coherent deposits are readily obtainable; a 0.04—0.05-mm. coating may be obtained by electrolysis at 160° with a current density of 1-1.5 The chemical inertness of the refined aluminium and the high corrosion resistance of the aluminium coatings are noted. Corrosion in moist air ceases completely when a layer of aluminium 0.003 mm. thick has become oxidised. H. F. GILLBE.

Chromium deposits directly on aluminium. H. K. Work and C. J. Slunder (Trans. Amer. Electrochem. Soc., 1931, 59, 175-181).—Chromium can be satisfactorily deposited directly on aluminium and its alloys from a plating bath of the ordinary type if the surface has been suitably cleaned and etched. The nature of these preliminary processes to be used under various conditions is described. The best current density for chromium deposition depends on the nature of the work, but for thin deposits from a solution at 45° usually exceeds 21.5 amp./dm.2 At lower temperatures the deposit is darker in colour, but is more easily buffed to a high lustre. For thick deposits, however, good adhesion is obtained by working at 60° and 40 amp./dm.2 The thin deposits have a fairly good resistance to outdoor exposure and to the salt spray, and thicker plates afford considerable protection against strong alkaline solutions. Deposits 0.025-0.1 mm. thick on an alloy containing 4% Cu, 0.5% Mn, and 0.5% Mg showed good resistance to abrasion, and pistons so plated have behaved well in tests.

H. J. T. ELLINGHAM.

Independence of the hardness of electrolytic metals of their content of hydrogen. Guichard, CLAUSMANN, BILLON, and LANTHONY (Compt. rend., 1931, 192, 623—625; cf. Hugues, B., 1926, 159).— Samples of electrolytic iron and cobalt, respectively, lose all their hydrogen when heated at 250° and 450°, with no and very slight loss of hardness. Heating to higher temperatures causes a steady decrease in hardness. It is concluded that the hardness of electrolytic metal is in no way due to the presence of hydrogen, but results solely from their very fine structure.

C. A. SILBERRAD.

Progress of the hot nickel[-plating] solution. O. P. Watts (Trans. Amer. Electrochem. Soc., 1931, 59, 193—197).—Information regarding composition and  $p_{\rm H}$ value of baths, temperature, current density, material for tanks and linings, nature of work plated, and quantity of solution maintained has been supplied by a number of firms, mostly in the U.S.A., which have adopted the hot nickel-plating process recommended by the author in 1916. Most of the baths consist of nickel sulphate and chloride with addition of boric acid, but the  $p_{\rm H}$  values reported range from 2.2 to 6.5, temperatures from 32° to 71°, and current densities from 10 to 100 amp./ft.2 Comments by platers on the respective merits of hot and cold baths are also reported.

H. J. T. ELLINGHAM.

Electrodeposition of cobalt-nickel alloys. S. GLASSTONE and J. C. SPEAKMAN (Trans. Faraday Soc., 1931, 27, 29-35; cf. B., 1930, 1074).—The investigation of the compositions of alloys deposited from wellbuffered solutions of definite hydrogen-ion concentration containing various mixtures of cobalt and nickel sulphates at a series of current densities has been extended to 50° and 95°. The relative tendency for cobalt and nickel to deposit is independent of the hydrogen-ion concentration. The alloys deposited at very low current densities contain decreasing proportions of cobalt as the temperature is raised, and this proportion is, in general, less than that in the solution for depositions at 90°. With increasing current density the proportion of cobalt in the alloy increases rapidly up to a maximum, independent of the current density. This maximum decreases with rise in temperature. The influence of cobalt on the deposition of nickel at 50° and at 90° is very similar to that of iron at 15° and 50°, respectively (cf. A., 1928, 851). The theoretical basis of the results

is discussed in terms of the deposition potentials and overvoltages of the metals concerned. O. J. Walker.

Silicon carbide. Applications of rubber.—See I. Low-carbon pig iron.—See II.

See also A., May, 556, Brasses. Systems Cu-Ag, Cd-Ag, Al-Ag, and Si-Al. Ternary silver alloys. 557, Fe-Si-C and Fe-W-C systems. 561, Colloidal silver and gold. 569, Ternary alloys. 570, Dissolution and deposition of metals. Brass. 574—5, Corrosion. 583, Rhenium. 584, Reduction of iron oxide. Reactions of iron etc. with oxides of iron. 586, Analysis of iron and steel. 588, Determination and separation of lead and bismuth. 589 and 590, Determinations of molybdenum, zirconium, and iridium. 590, Detection of gold, palladium, and silver.

#### PATENTS.

Drum furnace for melting metals. C. Brackelsburg (B.P. 344,980, 31.12.29).—The interior of a revolving drum is divided by an annular passage into a relatively long melting chamber of conical formation and into a second chamber, preferably inclined to the axis of the drum, for mixing and superheating. A pulverisedfuel burner is situated at one end and the gases pass through both chambers, leave at the narrow end of the cone, and enter an inclined chute which also serves as a feed for metal. The melted metal overflows the constriction into the superheating chamber.

C. A. KING.

Metal-melting pots. H. T. Thorp and W. Geddes (B.P. 345,697, 7.1.30).—A furnace for melting metals of low m.p., e.g., lead, is divided into two intercommunicating compartments, the first, into which metal ingots are fed, being at rather a higher level so that molten metal flows into the second by gravity. The two pots are heated independently by means of oil or gas and a float on the molten metal in the delivery compartment controls the fuel supply to the ingots in relation to any predetermined level of molten metal. It is desirable to arrange the communicating passage and outlet in alinement to facilitate cleaning. C. A. King.

[Casting of] steel ingots. A. G. EGLER (B.P. 345,983, 1.11.29. U.S., 9.11.28).—The mould is continuously jarred during the casting operation, thereby causing the slag and impurities to rise to the top and the depth of pipe to be considerably reduced.

A. R. POWELL.

Casting of metals or alloys. Brit. & Dominions Feralloy, Ltd., and C. Sykes (B.P. 345,047, 14.2.30).— To prevent oxide inclusion in the metal, the casting of aluminium or of alloys containing iron and aluminium is carried out in an atmosphere composed chiefly of carbon and chlorine or of a chlorinated hydrocarbon (e.g., trichloroethylene or chlorinated pitch).

H. ROYAL-DAWSON.

[Inhibitors for the] pickling of metals and like processes. Imperial Chem. Industries, Ltd. (B.P. 346,162, 4.4.30).—Acraldehyde derivatives of the general formula R'·CH: CR·CHO, in which R and R' are the same or different alkyl groups, are claimed, specifically α-ethyl-β-propylacraldehyde. The addition of sulphite-cellulose waste and sodium hydrogen sulphate to the

pickling bath prevents the disengagement of objectionable spray.

A. R. POWELL.

Treatment of ferromagnetic alloys to obtain enhanced magnetic properties. Verein. Stahlwerke A.-G. (B.P. 346,013, 23.9.29. Ger., 24.10.28).—
Iron alloys containing a constituent the solubility of which is greater at high than at ordinary temperatures are quenched from a temperature above that at which the solid solubility commences to increase and below that at which a duplex structure appears, *i.e.*, in the homogeneous α-range, and subsequently reheated at temperatures below the quenching temperature. [Stat. ref.]

Electrodeposition of platinum metals. (A) C. W. Keitel and (B) H. E. Zschiegner, Assis. to Baker & Co., Inc. (U.S.P. 1,779,436 and 1,779,457, 28.10.30. Appl., [A] 2.7.29, [B] 7.10.27).—(A) For the electrodeposition of platinum, rhodium, or palladium the bath consists of a 10% solution of ammonium sulphate or nitrate containing 5% of free ammonia and 2% of the corresponding diammino-platinum metal nitrite. The bath is operated at 95° with 2·2 volts and its composition is maintained by adding ammonia and the nitrite as required. (B) The bath is prepared by boiling the platinum-metal chloride with sodium nitrite until the solution is colourless or pale yellow and then adding an excess of ammonia. A. R. POWELL.

Improving copper or [silicon-iron-]copper alloys and hardener for use therein. U. DE BERKER, P.M.G. METAL TRUST, LTD., W. MACHIN, and W. B. O'B. GOUDIELOCK (B.P. 347,098, 12.12.29).—See U.S.P. 1,777,192; B., 1931, 353.

[Apparatus for] metallising by the melting and projection of metals. Soc. Nouvelle de Métallisation (B.P. 346,656, 9.12.29. Fr., 7.12.28).

Processing of furnace dust. Heat removal by mercury. Installations for endothermic reactions.
—See I. Refining furnaces. Welding.—See XI.

### XI.—ELECTROTECHNICS.

Decomposition of iron oxide in vacuum tubes. E. R. Wagner (Trans. Amer. Electrochem. Soc., 1931, 59, 205—207).—In studying the possibility of using superficially oxidised iron as anode in a vacuum tube it was found that with a gradient of 300 volts/cm. between the plate and the filament the black surface layer was gradually reduced to metallic iron and gas accumulated in the tube. The amount of gas, however, was far less than that corresponding to the oxygen in the oxide layer. On increasing the voltage gradient a large amount of gas is suddenly released, as though it had accumulated during operation at the lower voltage, but remained occluded in the plate. It is concluded that the observed phenomena are due to electron bombardment rather than to electrolysis.

H. J. T. ELLINGHAM.

Silicon carbide. Applications of rubber.—See I. Oxidation of ferrocyanide.—See VII. Electrochemistry and glass.—See VIII. Test for corrosion resistance. Resistance of metals. Aluminium. Chromium deposits on aluminium. Hardness

of electrolytic metals. Nickel-plating. Cobaltnickel alloys.—See X. Conductivity of molasses.—See XVII. Nitrogenous matter in milk.  $p_{\rm H}$  of cheese whey.—See XIX.

See also A., May, 546, Glasses for ultra-violet rays. 555, System AlCl<sub>3</sub>-NaCl. 569, Ternary alloys. 570, Dissolution and deposition of metals. 577, Sodium and lead arsenates. 578, Reduction of carboxylic acids. 593, Colorimeters etc.

#### PATENTS.

Electric [metal-]refining furnace. T. F. Bally (U.S.P. 1,778,578, 14.10.30. Appl., 28.1.28).—A resistance furnace comprises a vertical refining chamber of uniform cross-section, a top electrode movable throughout the height of the chamber, and a bottom electrode; the cross-sectional dimensions of the electrodes are about one half that of the molten slag resistor column extending between the electrodes and having a height at least three times its transverse dimension.

J. S. G. THOMAS.

Electric induction furnaces [for refining metals]. D. F. Campbell, and Electric Furnace Co., Ltd. (B.P. 345,635, 25.11.29).—Slag is heated from above by radiation from conducting material, e.g., of carbon or metal, forming a secondary circuit for a helical primary coil extending to the level of the conducting body and surrounding the furnace lining. J. S. G. Thomas.

Electric electrode furnaces. E. Bornand and H. A. Schlaepfer (B.P. 345,082, 11.3.30. Fr., 27.3.29).

—A refractory plunger surrounding the electrodes and adapted to be lowered into, and to mix, molten metal contained in the furnace is claimed. J. S. G. Thomas.

Manufacture of electric heating elements. K. Noguchi, Assr. to Mitsubishi Tosen Kabushiki Kaisha (U.S.P. 1,779,822, 28.10.30. Appl., 25.6.28. Jap., 31.10.27).—The space between a heating element and a surrounding metallic sheath is packed with a powdered mixture of aluminium (2 pts.) and lime (1 pt.) to which water is added, whereby alumina is produced.

J. S. G. THOMAS.

Electrode [for welding etc.]. T. S. Fuller, Assr. to Gen. Electric Co. (U.S.P. 1,778,668, 14.10.30. Appl., 30.6.27).—An electrode, containing 96% Cu, 0.8% Si, and 3.2% Ni, having hardness (Brinell) 125—175 and sp. resistance about 3—4 microhms/cm.<sup>3</sup>, is claimed.

J. S. G. Thomas.

Unilateral conductor for rectifying alternating currents. H. G. André (U.S.P. 1,780,057, 28.10.30. Appl., 29.10.26).—A film-forming metallic cathode, e.g., of nickel-silicon alloy (23% Si), wound with a helix, e.g., of silver or other metal forming a conducting oxide, is immersed in sulphuric acid (d 1·8), and alternating current passed between the cathode and helix. On removal from the acid the rectifier is dried or baked to remove excess acid.

J. S. G. Thomas.

Photoelectric cells. Westinghouse Lamp Co., Assees. of H. C. Rentschler and D. E. Henry (B.P. 345,043, 11.2.30. U.S., 28.2.29).—Metallic cæsium is produced in the cell by reduction of cæsium dichromate with silicon. Excess of alkali metal is fixed as a substantially inert compound by combination with a getter

forming stable solid compounds therewith. Thus, e.g., part of the envelope of the cell is formed of lead glass, which, on heating, cleans up excess cæsium.

J. S. G. THOMAS.

[Filling for] photoelectric cells. F. A. LINDEMANN (B.P. 345,242, 18.12.29).—In photoelectric cells comprising a sensitised cathode, e.g., a unimolecular cesium cathode, the necessary operating voltage is reduced and the sensitivity increased by employing a filling consisting of krypton and/or xenon.

J. S. G. THOMAS.

Photoelectric cells. M. A. E. Pressler (B.P. 345,189, 29.7.30. Ger., 29.7.29).—Cells for television purposes are filled with xenon and/or krypton.

H. ROYAL-DAWSON.

Manufacture of photoelectric cathodes. Gen. Electric Co., Ltd., and N. R. Campbell (B.P. 345,375, 6.1.30).—The supply of electropositive metallic vapour, e.g., cæsium, to a photoelectric cell heated considerably above 50°, e.g., at 200°, is controlled by a constriction, and is stopped when the space current in the cell decreases rapidly.

J. S. G. Thomas.

[Electrodeless] electric-discharge devices [containing condensable vapour]. Brit. Thomson-Houston Co., Ltd., Assees. of T. E. Foulke (B.P. 344,906, 3.12.29. U.S., 3.12.28).—An electrodeless discharge is maintained in a vessel containing mercury vapour, part of which is continuously condensed in a condensing chamber, so that the vapour pressure in the discharge vessel is maintained below that at which a high-pressure discharge occurs. J. S. G. Thomas.

Direct-reading [photochemical] photometer. S. Wein, Assr. to Radiovision Corp. (U.S.P. 1,779,574, 28.10.30. Appl., 8.11.29).—A portable photometer comprising a photochemical cell, e.g., a cell consisting of two platinum electrodes immersed in an acidulated solution containing potassium iodide and ferric chloride, and an indicating device, e.g., a micro-ammeter, connected with the electrodes, is claimed. J. S. G. Thomas.

Chemical apparatus [for determining the hydrogen-ion concentration of solutions]. S. L. Handforth, Ass. to E. I. Du Pont de Nemours & Co. (U.S.P. 1,779,232, 21.10.30. Appl., 29.6.29).— Electrode potential apparatus, comprising a standard calomel cell and an electrode cell separated by a bridge cell containing a buffer solution, is claimed. Electrical connexion between solutions contained in the bridge cell and the other two cells, respectively, is effected by means of porous plugs fused into tubes sealed into partitions between the cells. J. S. G. Thomas.

Polarising electrolyte. E. Banning (B.P. 344,776, 9.9.29).—A solution of a higher polysaccharide, e.g., gum arabic, in glycerin or water is claimed.

J. S. G. THOMAS.

Electric arc furnace [for melting ceramic materials, e.g., mullite]. W. E. Evans. From VITREFRAX CORP. (B.P. 345,236, 10.12.29).—See U.S.P. 1,747,756; B., 1930, 672.

Generation of alternating electric currents for use in induction furnaces etc. N. R. Davis, C. R. Burch, and Associated Electrical Industries, Ltd. B.P. 347,248, 20.1.30).

Electric welding [of tubes of different degrees of hardness]. G. Kalschne, Assr. to Gen. Electric Co. (U.S.P. 1,779,278, 21.10.30. Appl., 15.9.27. Ger., 30.9.26).

Sealing of glass to metal [for vacuum-tube apparatus]. Brit. Thomson-Houston Co., Ltd., and W. N. Mischler (B.P. 346,746, 13.12.29. U.S., 24.12.28).

Gas producer. Viscosimeter. Sp. gr. of battery acid.—See I. Treatment of hydrocarbons. Insulating oil. Testing hydrocarbons.—See II. Antioxidant [for oils].—See III. Nitric acid. Decomposition of water.—See VII. Ferromagnetic alloys. Platinum deposits.—See X. Concentration of latex.—See XIV.

# XII.—FATS; OILS; WAXES.

Splitting of oils and fats nearly to completion. G. Petrov and A. Pichugina (Masloboino Zhir. Delo, 1929, No. 9, 53—56).—Hydrolysis (97·87—99·6%) was effected by the use of naphthalenesulphonic acids (0·9—1% of the oil) in two stages.

CHEMICAL ABSTRACTS.

Lecithin retards hydrolysis of fats. R. B. TRUSLER (Oil & Fat Ind., 1931, 8, 141—143, 157).—Examination of the Twitchell saponification indicates that the resistance to hydrolysis evinced by certain seed oils, codliver oil, etc. is attributable to the presence of lecithin or related substances; these impurities are readily inactivated by treatment with hydrochloric or sulphuric acid (20%) prior to saponification. E. Lewkowitsch.

Some titer points of mixed fatty acids. I. Mixtures of commercial oils, fats, and fatty acids. G. W. Jennings (Ind. Eng. Chem., 1931, 23, 413—415).— The titers of a number of binary mixtures of commercial fatty acids have been determined and the results plotted against the composition of the mixture. In the case of the mixed fatty acids of coconut oil and red oil the curve is practically identical with the straight line joining the values for the individual components; otherwise smooth curves a little above or below the theoretical lines are obtained. Red oil mixed with cottonseed oil fatty acids shows a marked irregularity at 25—30% content of the latter.

E. Lewkowitsch.

Preparation and properties of highly purified oleic acid. J. H. SKELLON (J.S.C.I., 1931, 50, 131-134 т).—Existing methods of preparation of pure oleic acid have been investigated and two modified processes formulated, by which a highly purified oleic acid can be obtained from the crude acids of olive oil. One of these methods is based on Twitchell's lead salt-alcohol process and a modification of the method of Armstrong and Hilditch. The crude acids from olive oil are treated with sufficient lead acetate to combine with 23.5% of total fatty acids in a volume of 8 times their weight of boiling alcohol, which results in a quantitative separation of solid from liquid lead salts. The recovered liquid acids, dissolved in 5 times their weight of absolute alcohol, are neutralised with a boiling solution of lithium hydroxide containing 0.15 g. of base per gram of acid. The lithium salts are separated, dried, and recrystallised from a solution in 4 times their weight of alcohol. The recovered acid is further purified by fractional

distillation of the methyl ester in a vacuum. In the second method the liquid acids obtained by the above modified Twitchell process are converted into barium salts, in a solution of 12 times their weight of moist benzene and ethyl alcohol. The solution is quickly cooled, and the crystallised barium oleate is further purified from a mixture of 3 times its weight of moist benzene and alcohol. The properties of highly purified oleic acid have been investigated and criteria of purity established.

Evaluation of stearine pitch. G. Siebert and E. Blennemann (Farben-Ztg., 1931, 36, 1090).—A general account is given of the properties of stearine pitch, the need for evaluation from technical as well as purely chemical aspects being stressed. Details are given of the classification of the pitch into hard, medium, and soft grades, according to flow characteristics. Both stearine pitch and wool-fat pitch give an acraldehyde test, but the former is completely soluble in hot alcoholic potash, whereas the latter gives a residue from which a fatty acid (m.p. 80-82°) may be isolated. Relatively high saponif. value of the pitch and acid value of distillation product therefrom distinguish stearine pitch from petroleum pitches. Tests for solubility in solvent naphtha, drying time, and general film properties are S. S. WOOLF. also described.

Determination of the oil content of seeds in series. A. Lebediantzev and N. Dmitriev (Masloboino Zhir. Delo, 1929, No. 10, 3—10, No. 11, 3—9, No. 12, 9—17).—If the material is very finely powdered, extraction is complete in 6 hrs. at 30 siphonings per hr. with ether free from alcohol and dried over calcium chloride. Drying oils must be dried below 60° in nitrogen or carbon dioxide. The material should be dried or mixed with calcined calcium sulphate.

CHEMICAL ABSTRACTS.

E. Lewkowitsch.

Application of certain research methods of colloid chemistry to the drying of linseed oil. H. Freundlich and H. W. Albu (Z. angew. Chem., 1931, 44, 56-60).—Linseed oil, with and without driers, and bodied oils prepared from them by blowing (up to 300 hrs. at ordinary temperature) or by heating in air or carbon dioxide (at 250°) were examined. Differences in the "depolarisation" of the various oils were very small and non-characteristic. The oils were tested in a Couette torsion viscosimeter, but no deviations from normal behaviour could be observed, even in the case of blown oils of high viscosity (e.g., absolute  $\eta = 82$ ). In confirmation of these results, ultramicroscopical examination (in conjunction with E. Hauser), using a Spierer condenser (cf. B., 1930, 844), revealed no trace of colloidal structure. The changes produced in the oil by the various treatments appear to be entirely chemical in nature, but the possibility is not excluded that colloid processes may play a greater part at a more advanced stage of drying (or in wood-oil drying).

Seeds and oil of *Sinapis dissecta*, Lag. N. Beliaiev (Masloboino Zhir. Delo, 1929, No. 6, 25—26). —The seeds of this variety of *S. alba*, L., contain moisture 8.80, oil 28.95, fibre 15.06, ash 4.48, total nitrogen 3.83, crude protein 23.90, essential oil 0.09%. The oil

has d 0.9150, f.p. 15°,  $n_{\rm D}^{\rm so}$  71.9, saponif. value 172.4, iodine value (Hübl) 100.52, acid value 1.64. The fatty acids have iodine value 99.38, m.p. 21°, f.p. 18°.

CHEMICAL ABSTRACTS.

Soya-bean oil. M. SERGEEV (Masloboino Zhir. Delo, 1929, No. 3, 47—51).—The oil from Kuban beans (yield 19·6—25·3%) gives low values for  $n_D^{20}$ , acid value, and iodine value.

CHEMICAL ABSTRACTS.

Relationship between acidity and titer [solidification point] of oils. I. Stetzenko and I. Panteleyev (Masloboino Zhir. Delo, 1929, No. 10, 20—21).—Although the f.p. of the fatty acids is higher than that of their glycerides, a high f.p. is not an indication of high acidity, since the neutral oil forms a cutectic mixture with the free fatty acids.

Chemical Abstracts.

Saponification values of highly coloured oils. H. S. Jois, B. L. Manjunath, and S. V. Rao (Mysore Univ. J., 1930, 4, 241—242).—The Albert method (cf. Coburn, B., 1930, 623) is of general applicability for the determination of saponification values of coloured or non-coloured oils.

H. Burton.

Cresol soap solutions.-See XX.

See also A., May, 597, Oleastene. 602, Hydrogenation of linseed oil etc. Separation of glycerides. Rhodanometry of tung oil. 644, Colouring matter of milk. 658, Cod-liver oil.

#### PATENTS.

Spray-drying of soap and sprayed soap product. E. P. STEVENSON and B. B. FOGLER, Assrs. to A. D. LITTLE, INC. (U.S.P. 1,779,516—7, 28.10.30. Appl., [A] 28.8.28, [B] 18.12.29).—(A) The soap solution is superheated and discharged horizontally by atomisation in a concurrent stream of heated air: the spray passes into a vertical vessel from which the hot air may be vented at the top (carrying with it any very fine overpuffed particles), whilst the soap granules separate and fall by gravity through a zone of relatively cold air. The moisture content of the powder may be controlled by admission of low-velocity conditioned air at the base of the cooling vessel. The design of the atomiser cup is detailed. Products bulking as high as 15—20 lb./ cub. ft. and containing 15-20% of moisture are obtainable. (B) Sodium silicate incorporated in the soap so processed is retained in an easily soluble form in the non-dusty product. E. LEWKOWITSCH.

Recovery of rosin soap material from spent wood liquors. V. Drewsen, Assr. to West Virginia Pulp & Paper Co. (U.S.P. 1,778,523, 14.10.30. Appl., 11.1.28).—The spent liquors are concentrated to  $d \cdot 1 \cdot 1 - 1 \cdot 15$ , so that the sp. gr. of the liquor differs from that of the soap, and is centrifuged at 25—60° to separate the rosin soap.

E. Lewkowitsch.

Separation of fatty or wax-like materials from their solutions. J. K. Pfaff, K. Böttger, and A. Sieweke, Assrs. to Deuts. Gasolin A.-G. (U.S.P. 1,779,287, 21.10.30. Appl., 2.7.29. Ger., 16.5.27).— An approximately equal weight of a soluble substance which crystallises well, but is chemically different, e.g., naphthalene, is added to the hot solution of the soft or badly-crystallising waxy material (such as paraffin wax, petroleum jelly, montan wax, etc.) in a petroleum

distillate or organic solvent: on cooling the solution to 0° the waxy materials are precipitated together with the added substance and may be readily separated by filtration. The naphthalene is removed from the precipitate and filtrate by steam-distillation etc.

E. Lewkowitsch.

Manufacture of derivatives of sulphonated fatty acids. A. Carpmael. From I. G. Farbenind. A.-G. (B.P. 344,828, 2.9.29. Cf. B.P. 343,989; B., 1931, 501).

—Unsaturated fatty oils are esterified with a monohydric alcohol (e.g., butyl alcohol) in the presence of a catalyst (e.g., hydrochloric acid) and sulphonated; the order of the processes may be reversed. [Stat. ref.]

E. Lewkowitsch.

Extraction of [cashew] nut[shell] oil. T. M. Rector, Assr. to Franklin Baker Co. (U.S.P. 1,777,808, 7.10.30. Appl., 8.11.28).—The whole nuts are submerged for about 1 min. in a bath of molten metal or alloy (m.p. not above 204°). The shell oil (about 12% on the raw nut) is expelled and floats to the surface, protecting the metal from oxidation; further, the shells are thereby conditioned for easy removal from the uninjured kernel. E. Lewkowitsch.

Manufacture of emulsions of waxes. I. G. FARBENIND. A.-G. (B.P. 345,184, 21.7.30. Ger., 19.7.29).

—A cellulose ether, ester, or ether-ester and a wax, e.g., beeswax, are dissolved by heating in a mutual solvent, in which, however, the wax is soluble only in the hot, and the solution is cooled rapidly while stirring. Pigments may be added to the emulsion, which is suitable for use as a protective coating.

E. Lewkowitsch.

Milling of soap etc. Press for extracting liquids. Sp.-gr. balls.—See I. Wetting etc. agents.—See III. Silicyl compounds.—See XX.

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

Viscosity and "brushability" of paints. Wolff (Farben-Ztg., 1931, 36, 1088—1089, 1131— 1132).—Results in concordance with those obtained by Beck, using a Couette torsion viscosimeter (B., 1929, 785) were obtained by means of the "Turbo viscosimeter." It is shown that the plasticity of a paint system may vary with the velocity of flow (the viscosity not being in linear relationship therewith) or be constant (the viscosity being in linear relationship with the velocity), zinc oxide in oil and nitrocellulose in butyl acetate illustrating these two types. Painting tests carried out by various observers indicate that the general speed of brushing paints is such that viscosity approaches its minimum value and its plasticity is negligible. Considerations of the change in viscosity of a paint owing to volatilisation of solvent etc. lead to the conclusion that viscosity is not a measure of brushability. S. S. WOOLF.

Red lead. H. Hebberling (Farben-Ztg., 1931, 36, 1129—1130).—A commentary on the work of Wagner, Grohn, and others on the rust-preventative and general properties of red lead and the comparison of iron oxide pigments with it in this connexion. (Cf. B., 1930, 468, 622.)

S. S. Woolf.

Hydrogen-ion concentration and the colour of lead chromate pigments. R. C. Ernst, E. Pragoff,

Jun., and E. E. Litkenhous (Ind. Eng. Chem. [Anal.], 1931, 3, 174—176).—Precipitates of lead chromate were made by mixing 0.5N-solutions of lead acetate and potassium dichromate and the acidity of the solutions was varied by the addition of acetic, hydrochloric, nitric, and sulphuric acids. The  $p_{\rm H}$  was determined by means of the quinhydrone electrode. With decreasing hydrogen-ion concentration the colour changes from yellow at  $p_{\rm H}$  3.5 to orange of a maximum brightness at  $p_{\rm H}$  9, and thereafter the colours become dirty. With rise of temperature the colour changes from yellow at low temperatures to a maximum orange at 80° and becomes lighter again at higher temperature. Changes in concentration have no appreciable effect on the colour. E. S. Hedges.

Colouring of paper.—See V. Hop resin.—See XVIII. Pigment of cereals.—See XIX.

See also A., May, 564, Ferric oxide colours. 625, Cannabis indica resins. Euphorbium resins.

#### PATENTS.

Manufacture of paints. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 344,458, 6.12.29).—Inorganic or organic dyestuff pigments are suspended in an aqueous emulsion of a synthetic resin derived from a polyhydric alcohol, a polybasic acid, and a monobasic acid; a salt of ammonia or a volatile amine with an organic carboxylic acid of high mol. wt., e.g., a wax acid, is used as emulsifier.

S. S. Woolf.

Reducing the zinc oxide content of lithopone. A. B. Laftman, Assr. to Grasselli Chem. Co. (U.S.P. 1,780,559, 4.11.30. Appl., 22.12.26).—Crude lithopone slurry is treated with a predetermined small amount of an acid, e.g., sulphuric acid, to render any acetic acid-soluble zinc compounds water-soluble, whence they are precipitated by adding the calculated amount of barium sulphide.

S. S. Woolf.

Production of titanium pigments. R. H. Monk and L. Firing (B.P. 345,668, 18.11.29).—Precipitated titanium hydroxide is treated with a peptising agent, e.g., nitric or hydrochloric acid or barium chloride, and the colloidally dispersed mass resulting is mixed with a coagulating agent containing an ion of opposite charge to that of the dispersed hydroxide, e.g., barium carbonate, in amount chemically equivalent to the titanium dioxide present. The coagulated product is washed, dried, and calcined, e.g., at 840°, giving porous particles of high opacity and soft texture. Excess of barium chloride may be used in order to provide a catalyst for the reaction effected by calcination. (Cf. U.S.P. 1,760,513; B., 1930, 1151.)

Production of cellulose solutions [of high or known viscosity]. Deuts. Gold- U. Silber-Scheidean-stalt vorm. Roessler (B.P. 345,613, 18.11.29. Ger., 26.11.28).—In the preparation of such solutions (particularly from cuprammonium and xanthate solutions of cellulose) both solvent and cellulose are completely freed from oxygen by means of, e.g., ammoniacal cuprous oxide or chloride, and the dissolution process is carried out in the absence of air and active light. If oxygen be quantitatively introduced into the mixture, e.g., as

persulphate, solutions of desired viscosity may be obtained. S. S. Woolf.

Cellulose acetate [film-forming] compositions containing mixed volatile solvents. Kodak, Ltd. From Eastman Kodak Co. (B.P. 345,505, 29.3.30).—A mixture of 50 pts. by wt. of methyl acetate, 40—10 pts. of ethyl acetate, and 10—40 pts. of acetone forms a superior solvent for cellulose acetate to be used in the production of film.

E. Lewkowitsch.

Compositions of matter [containing polymerised vinyl derivatives and cellulose derivatives] and articles or products obtainable therefrom. Brit. Celanese, Ltd. (B.P. 345,521, 10.4.30. U.S., 13.5.29. Cf. B.P. 308,658; B., 1930, 1146).—An increased proportion of a polymerised vinyl compound, e.g., vinyl acetate, can be tolerated in a cellulose ether or ester composition if a natural or synthetic resin, which is compatible with the cellulose derivative, is also incorporated. Solvents, plasticisers, pigments, etc. are added as required. The products are suitable as lacquers, for making photographic films, as adhesives for reinforced glass, etc.

E. Lewkowitsch.

Coating composition. W. E. Lawson, Assr. to E. I. Du Pont de Nemours & Co. (U.S.P. 1,780,652, 4.11.30. Appl., 18.5.28).—Compositions containing polymerised vinyl compounds, e.g., an ester or chloride, and tetrahydrofurfuryl alcohol are claimed.

S. S. Woolf.

Cellulosic [coating] composition. E. E. Reid and G. L. Schwartz, Assis. to E. I. Du Pont de Nemours & Co. (U.S.P. 1,778,567, 14.10.30. Appl., 16.5.25).—Mixed alkyl cyclohexyl esters of phthalic acid, e.g., cyclohexyl butyl phthalate, are added to cellulose esters as softeners.

F. R. Ennos.

Synthetic resin varnishes. IMPERIAL CHEM. INDUSTRIES, LTD., A. A. DRUMMOND, and H. H. MORGAN (B.P. 345,633, 19.9.29).—Small proportions of anhydrous metallic salts of a dehydrating character, e.g., ferric or calcium chloride, potassium hydrogen sulphate, dissolved, if desired, in an organic solvent, e.g., ethyl acetate, cyclohexanol, Cellosolve, are added to synthetic resin varnishes of the phenolic compound-formaldehydefatty acid ester type. Increased drying rate is claimed. S. S. Woolf.

[Urea-formaldehyde] varnishes and lacquers. A. V. Keller and J. Taylor (B.P. 345,845, 17.4.30. Addn. to B.P. 331,428; B., 1930, 919).—A small proportion of an accelerating agent, e.g., sodium phosphate, is added to the urea paste before mixing. Formaldehyde and phenolic substances may replace part of the paraformaldehyde and urea derivative, respectively.

S. S. Woolf.

Resinous compositions and lacquers. IMPERIAL CHEM. INDUSTRIES, LTD., R. HILL, and E. E. WALKER (B.P. 344,401, 30.8.29).—Resins of the polyhydric alcohol-polybasic acid type (including such resins containing drying or non-drying oils or fatty acids) are heated with formaldehyde-urea or -thiourea condensation products, or substances capable of yielding these, in an alcoholic solvent of b.p. above 100°, e.g., ethylene-chlorohydrin, a glycol monoalkyl ether, butyl alcohol,

cyclohexanol. The resulting resins are rapid-hardening and, in the presence of an acid catalyst, e.g., sulphuric, chlorosulphonic, hydrochloric, trichloroacetic, or tartaric acid, are capable of being hardened in the cold.

S. S. Woolf.

Substituted guanidine-aldehyde condensation product. C. M. Stine, Assr. to E. I. Du Pont de Nemours & Co. (U.S.P. 1,780,636, 4.11.30. Appl., 8.10.25).—Infusible synthetic resins obtained by condensation of guanidine, its salts, or substitution products with aldehydes, e.g., formaldehyde or benzaldehyde, are claimed.

S. S. Woolf.

Manufacture of [resinous] aldehyde condensation products. H. Kappeler (B.P. 345,891, 12.6.30. Switz., 12.6.29).—Furfuraldehyde or a derivative thereof, e.g., furfuramide, is condensed in one or more stages with another aldehyde or polymeride thereof, e.g., paraformaldehyde, in the presence or absence of an indifferent solvent or diluent and in the presence of an acid condensing agent, e.g., hydrochloric acid; dyes, fillers, or softening agents may be introduced at any stage.

S. S. Woolf.

Manufacture of white resin. B. N. Lougovoy (U.S.P. 1,779,551, 28.10.30. Appl., 10.12.23. Renewed 15.6.25).—Hexamethylenetetramine is incorporated with the resinous reaction product of phenol, formaldehyde, and urea, giving light-coloured, substantially light-fast resins.

S. S. Woolf.

Manufacture of synthetic resins. Brit. Celanese, Ltd. (B.P. 344,413, 23.11.29. U.S., 26.11.28).—A diphenylolcycloparaffin, e.g., diphenylolcyclohexane, is condensed with formaldehyde in the presence of acid, neutral, or alkaline catalysts (preferably the first, e.g., hydrochloric, phosphoric, or boric acid) and, if desired, other reactants capable of forming synthetic resins, e.g., diphenylolpropane, urea, toluenesulphonamide. The product improves the adhesive properties of cellulose acetate lacquers etc. S. S. Woolf.

Manufacture of artificial resins and lacquers and artificial masses therefrom. J. Y. Johnson. From I. G. Farbenind. A.-G. (B.P. 344,626, 10.2.30. Addn. to B.P. 327,673; B., 1930, 624).—The urea, thiourea, methylolurea or its anhydride of the prior patent is replaced by a mixture of such urea compounds.

Resinous compositions. Brit. Thomson-Houston Co., Ltd., Assees. of L. V. Adams (B.P. 344,405, 28.10.29. U.S., 27.10.28).—A terpene compound, e.g., terpene hydrate, camphoric anhydride, borneol, gutta-percha, is (a) added to a polyhydric alcohol and polybasic acid or its anhydride, with or without a monobasic acid radical, during the reaction period; (b) heated with either acid or alcohol component, the other component being added subsequently and the heating continued; or (c) caused to react with the condensation product of the alcohol and acid while the latter is still in the fusible, soluble condition. Improved resistance to water is claimed.

S. S. Woolf.

Moulding compositions and moulded articles made therefrom. K. Ripper (B.P. 344,872, 12.10.29. Austr., 17.10.28).—Formaldehyde (less than 4 mols.) is

heated with a mixture of urea and thiourea (2 mols. of the mixture) in a solution having  $p_{\rm H} < 5$  (preferably at  $p_{\rm H}$  3) until a hydrophobe mixed condensation product is formed, which is subsequently dried and polymerised. Alternatively, the urea constituents may be introduced individually. Fibrous materials are admixed before or after the condensation, and after the incorporation of fillers, if desired, the compositions are hot-pressed at  $150-300~{\rm kg./cm.^2}$  in non-cooled moulds, and the pressed articles are given an additional air- or vacuum-drying. S. S. Woolf.

Moulded compositions. Brit. Thomson-Houston Co., Ltd., Assees. of L. E. Barringer (B.P. 345,390, 15.1.30. U.S., 28.1.29).—Strings or shavings of wood or metal, steel wool, rope waste, etc., of a sufficiently springy character, are coated with a solution of a binder, e.g., an ammoniacal aqueous solution of a resin, bloodalbumin, phenolic resin, and then drained and hotmoulded as required.

E. Lewkowitsch.

Manufacture of plastic compositions. Brit. Thomson-Houston Co., Ltd., Assees. of J. M. DeBell (B.P. 345,489, 17.3.30. U.S., 15.3.29).—The filler is impregnated or associated with a slow-hardening binder, e.g., drying oils, gilsonite, and hardened by polymerisation or oxidation; the product is comminuted and mixed with a small proportion, e.g., 15%, of a fast-moulding phenolic resin and hot-moulded.

E. LEWKOWITSCH.

Manufacture of artificial masses. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 345,197, 7.11.29).— A powdered filling material, e.g., zinc oxide or asbestos powder, is mixed with liquid or partly polymerised styrene, or a homologue thereof, and after polymerisation to the required degree the whole is moulded.

E. Lewkowitsch.

Manufacture of hollow objects of synthetic resin materials. E. E. NOVOTNY, ASST. to J. S. STOKES (U.S.P. 1,776,366, 23.9.30. Appl., 12.8.27).—A core of a solidified and cooling gaseous product, e.g., carbon dioxide ice, is inserted in a predetermined position in a mass of mouldable, potentially reactive synthetic resin; heat and pressure are then applied to gasify the core, cause the resin to react, and give a hollow object of desired form.

S. S. WOOLF.

Treating porous and non-porous [resinous] materials. T. F. Bradley, Assr. to Amer. Cyanamid Co. (U.S.P. 1,780,375, 4.11.30. Appl., 25.11.29).—Such materials are treated with aqueous solutions of soluble salts, e.g., ammonium salts, of polybasic acid-polyhydric alcohol resins, with or without the addition of casein solution, the resin being subsequently rendered waterinsoluble, e.g., by treatment with multivalent metal salts, acids, etc.

S. S. Woolf.

[Linoleum] floor covering. Armstrong Cork Co., Assees. of W. F. Kaufman and J. C. McCarthy (B.P. 347,527, 12.6.30. U.S., 15.7.29).

Milling of paint etc. Sp.-gr. balls.—See I. Pigment dyes. Azo dyes in varnishes.—See IV. Impregnated sheets.—See V. Impregnated fabrics.—See VI. Antimony trioxide.—See VII. Rosin soap material. Emulsions of waxes.—See XII. Rubber composition.—See XIV.

# XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Graphical tensile-testing machine for rubber threads. S. H. Hahn and E. O. Dieterich (Ind. Eng. Chem. [Anal.], 1931, 3, 218—221).—An especially designed curve-drawing machine is described for making tensile tests on thin rubber thread such as is used in golf balls and elastic fabrics.

D. F. Twiss.

Application of rubber.—See I. Manuring on rubber estates.—See XVI.

#### PATENTS.

Treatment of rubber latex. F. H. UNTIEDT (U.S.P. 1,777,945, 7.10.30. Appl., 20.6.27. Renewed 12.8.30).—Rubber latex containing a foam-stabilising agent, e.g., 1% of ordinary soap or 0.5% of saponin, is agitated, while air or other suitable gas is introduced, until a stiff dense foam is obtained which on drying yields a porous rubber. Compounding ingredients may be introduced prior to conversion into foam, and vulcanisation is best effected in the dried foam.

D. F. Twiss.

Concentration of rubber latexes and other aqueous dispersions of rubber. Soc. Ital. Pirelli, and U. Pestalozza (B.P. 344,875, 8.11.29).—Such dispersions are concentrated by disposing therein a filtering medium, e.g., an unglazed earthenware filter, and moving the medium and dispersion relative to one another at such a rate that the pores of the filter are maintained substantially free for the passage of the serum.

D. F. Twiss.

Concentration and purification of [rubber] latex. Naugatuck Chem. Co., Assess. of J. McGavack (B.P. 344,647, 25.2.30. U.S., 16.3.29).—The creaming of latex, to which a hydrophilic colloid or ammonium alginate has been added, is accelerated by subjecting the latex to electric strain, e.g., by applying an alternating current to two electrodes immersed in it. Using an alternating current of 60 cycles with electrodes of iron, steel, copper, or platinum, no deposition of rubber occurs at the electrodes. The amount of current passing may be restricted by thinly covering one of the electrodes with bakelite or collodion.

D. F. Twiss.

Treatment of rubber latex. J. McGavack, Assr. to Naugatuck Chem. Co. (U.S.P. 1,777,045, 30.9.30. Appl., 2.7.25. Renewed 8.5.29).—Latex is treated with an organic colloid, preferably pectin, in the presence of an acid, such as citric acid, in insufficient quantity to cause coagulation. The acid expedites the separation of a cream of concentrated latex. Separation may be further accelerated by centrifugal means. The separated concentrate may be purified by diluting with water and repeating the creaming operation. D. F. Twiss.

Preservation of natural and synthetic varieties of rubber. A. Carpmael. From I. G. Farbenind. A.-G. (B.P. 344,491, 2.12.29).—The ageing of rubber is retarded by incorporating a substance of the formula RR'CXY, where R and R' are benzene or alkylbenzene nuclei, at least one of which is substituted by at least one hydroxyl or alkoxyl group, and X and Y are hydrogen atoms or XY is an alkylene residue. Examples are p-ethoxydiphenylmethane and di-p-hydroxyphenylcyclohexane.

D. F. Twiss.

Manufacture of coloured rubber. H. D. Elkington. From N.V. de Bataafsche Petroleum Maats. (B.P. 344,533, 31.10.29).—Unvulcanised or cold-vulcanised rubber in which fibrous material, e.g., kapok, has been incorporated, preferably before coagulation of the rubber latex, is treated with a solution of a colouring matter which has no action on rubber, so that the colour is absorbed by the fibres. After washing for the removal of excess dye and drying, a translucent coloured rubber material is obtained.

D. F. Twiss.

Production of coloured synthetic rubber. I. G. FARBENIND. A.-G., and J. Y. Johnson (B.P. 345,239, 16.12.29. Addn. to B.P. 241,214; B., 1927, 948).—By incorporating components of colouring materials into solutions or emulsions of intermediate products in the formation of synthetic rubber from diolefines, converting the components into practically insoluble colouring materials, and then completing the preparation of the solid synthetic rubber, improved coloured products are obtained. As examples of components of colouring materials may be instanced leuco-compounds which are subsequently oxidised or the phenolic and diazo reagents for the formation of an azo dye.

D. F. Twiss.

Manufacture of coloured articles from dispersions of rubber or the like. Dunlop Rubber Co., Ltd., R. G. James, and D. F. Twiss (B.P. 344,537, 12.12.29).—The negative electric charge on the individual particles in an aqueous dispersion of an organic material such as rubber is converted into a positive one by adjustment of the  $p_{\rm H}$  to a suitable value on the acid side of the isoelectric point; the dispersion is then mixed with a soluble organic dye, e.g., methyl-violet, of which the coloured ions are cations. Latex so coloured can be used either for the manufacture of articles by spreading, spraying, etc., or to give a prior coating to formers which are subsequently immersed in latex the electric charge of which has not been inverted. D. F. Twiss.

Compounding of rubber. W. S. CALCOTT, W. A. DOUGLASS, and M. A. DAHLEN, ASSTS. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,778,548, 14.10.30. Appl., 13.6.29).—Compounds of the formula  $\text{HO} \cdot \text{R} \cdot \text{O} \cdot \text{R}' \cdot \text{Y}$ , where R and R' represent aryl or alkyl-substituted aryl residues and Y represents hydrogen or hydroxyl, do not affect the rate of vulcanisation of a rubber mixture, but greatly increase the resistance of the vulcanised product to deterioration. Examples are p-hydroxy- and pp'-dihydroxy-diphenyl ether. D. F. Twiss.

Manufacture of rubber compositions. Dunlop Rubber Co., Ltd., W. H. Chapman, and D. W. Pounder (B.P. 344,602, 20.1.30).—Scrap rubber-sponge trimmings and other fibrous or granular materials, such as comminuted vulcanised rubber or paper pulp, are mixed with an aqueous dispersion of rubber or similar material; the mixture is converted into a frothy condition, e.g., by whipping, and is then caused or allowed to set to a permanent spongy or cellular structure. D. F. Twiss.

Rubber[-decarboxylated rosin] composition. I. W. Humphrey, Assr. to Hercules Powder Co. (U.S.P. 1,776,857, 30.9.30. Appl., 7.10.27).—The residual product obtained by heating rosin with fuller's earth, e.g., with 10% at 300—325°, is used as a softening

ingredient in rubber. The "decarboxylated rosin" is a viscous oil containing neutral rosin oil and a pitch which is different from ordinary rosin pitch; abietic acid may also be present.

D. F. Twiss.

Rubber composition and preservation of rubber. C. W. Bedford, Assr. to B. F. Goodrich Co. (U.S.P. 1,777,634, 7.10.30. Appl., 13.1.30).—The products obtained by interaction of an unsaturated aldehyde, e.g., crotonaldehyde, and an amine, such as aniline or α-naphthylamine, in the presence of an acid, are substantially without accelerating influence on vulcanisation, but are effective in retarding the deterioration of rubber. D. F. Twiss.

Preparation of an elastic-plastic body. J. Baer (B.P. 345,175, 20.6.30. Ger., 24.6.29).—The product obtained from halogenated hydrocarbons of the  $C_nH_{2n+2}$  group and polysulphides in aqueous or alcoholic solution is dissolved in carbon disulphide and the solution mixed with rubber latex.

H. ROYAL-DAWSON.

Vulcanisation of rubber. A. C. Burrage, Jun. (B.P. 344,406, 29.10.29).—In the vulcanisation of rubber with the aid of an ultra-accelerator, e.g., mercaptobenzthiazole or tetramethylthiuram disulphide, the action of the accelerator is controlled and scorching is prevented by the additional presence of a disubstituted nitrosoamine such as diphenyl-,  $\beta\beta'$ -dinaphthyl-, or phenyl-p-tolyl-nitrosoamine. D. F. Twiss.

Vulcanisation processes. J. E. Pollak. From W. B. Wiegand (B.P. 345,335, 19.12.29).—In the vulcanisation of rubber articles by "live" steam, in particular of automobile inner tubes, improved results such as absence of porosity are obtained by initially subjecting the articles to additional gas pressure, this pressure being progressively reduced during the course of the vulcanisation process.

D. F. Twiss.

Rubber vulcanisation process. W. Scott and W. P. TER HORST, ASSTS. to RUBBER SERVICE LABS. Co. (U.S.P. 1,777,737, 7.10.30. Appl., 20.12.26).—Phenyl-substituted guanidines containing an ethoxy-group para to a phenyl nucleus, e.g., di-p-phenetidylguanidine, are used as accelerators. Di-p-phenetidylguanidine, m.p. 125·2—126·2°, is prepared by the action of cyanogen chloride on p-phenetidine or by desulphurisation and subsequent amidation of the reaction product of this base with carbon disulphide. Phenyl-p-phenetidylguanidine, m.p. 107·8—108·6°, s-tri-p-phenetidylguanidine, m.p. 186—188°, o-tolyl-p-phenetidylguanidine, m.p. 130·6—132·4°, and p-phenetidyldiguanide, m.p. 160·4—161·2°, are other examples.

D. F. Twiss.

Vulcanisation of rubber. W. Scott, Assr. to
Rubber Service Labs. Co. (U.S.P. 1,777,738, 7.10.30.

Appl., 24.2.28).—Accelerators of the thiol type, particularly mercaptobenzthiazole, are applied to the vulcanisation process in the form of their reaction products with a diguanide, especially monophenyldiguanide.

D. F. Twiss.

Antioxidant [for rubber].—See III. Coated fabrics. Aircraft covering. Balloon fabric. Mineventilating tubing.—See VI. Treatment of concrete. Floor coverings.—See IX.

# XV.—LEATHER; GLUE.

Method for testing agar and gelatin jellies. H. C. Lockwood and R. S. Hayes (J.S.C.I., 1931, 50, 145—151 T).—The principle of the method depends on the rigidity of the jelly when the supporting vessel is removed. The jelly is made of standard volume and shape, and the height measured, first when supported and then when inverted and the container removed. The decrease in height is dependent on quality, and an apparatus, essentially a depth micrometer, called the Ridgelimeter, is used to obtain the necessary readings. The strength of the standard agar jelly is 0.5%, which gives a Ridgelimeter decrease of 10.8% at 18°, whilst 0.3% and 1.0% jellies give decreases of 24.8% and 0.8% respectively. A 5% standard gelatin jelly shows a similar decrease of 10.8% at 9°, whilst the results for 3% and 10% are 29.6% and 2.4% respectively. The low temperature was more satisfactory for gelatin owing to the great susceptibility of gelatin jellies to temperature changes, especially above 12°. From tables compiled from experimental data, the Ridgelimeter decrease in height can be converted into "relative strength," arbitrary standards having been adopted for agar and gelatin jellies. Duplicate determinations of "relative strength" agreeing to well within 2% can readily be obtained after short experience and, as gelatins having "relative strengths" ranging from 0.66 to 1.79 are considered, this accuracy is sufficient. Tables and graphs show the effect of temperature on the rigidity of jellies, and such factors as the effect of tartaric acid and the time of heating are also discussed. For example, when a 0.5% agar solution is boiled for 1 min. with 0.05% of tartaric acid, the jelly strength is reduced by 50%, whilst gelatin jellies boiled for 10 min. with 0.1% of tartaric acid suffer a reduction of 6.0-8.0% in jelly strength.

Fur dyes.—See VI.

See also A., May, 627, Tea tannin.

# PATENTS.

Preparation of hides and skins for vegetable tanning. G. Peace (B.P. 344,417, 27.11.29).—Limed hides or skins are treated with a solution adjusted to  $p_{\rm H}$  3.5—5 by means of mineral acid and a salt of a weak acid, or a solution of sodium acetate and acetic acid, or an exhausted tan liquor with added acid, and are then tanned with vegetable tanning liquors maintained at a similar  $p_{\rm H}$  till the hides are struck through. D. Woodroffe.

Waterproofing of leather. C. J. M. M. LE PETIT (B.P. 344,346, 22.11.29. Fr., 28.11.28).—The latex of certain species of *Euphorbia* and cactaceous plants, *e.g.*, *E. candelabra* or *resinifera*, before or after polymerisation or hydrogenation by means of catalysts or radiations, is extracted with a solvent, *e.g.*, tetrachloroethane, which is afterwards evaporated off *in vacuo*, and the product, to which may be added sodium sulphoricinate and/or aluminium stearate, is used for treatment of leather.

D. Woodroffe.

Manufacture of inextensible leather suitable for transmission belts or belting etc. R. Chavand (B.P. 345,382, 10.1.30. Fr., 12.2.29).—A narrow band



of skin is stretched in a suitable frame (which is described) and tanned thereon. D. WOODROFFE.

Protecting leathers, skins, fabrics, etc. during their manipulation or making up into manufactured goods. Etabl. R. Schneider, and A. Poelman (B.P. 344,453, 6.12.29. Fr., 8.12.28).—Materials used in the manufacture of fancy leather goods are protected from soiling during making up, by applying a film prepared with a cellulose derivative such as cellulose acetate, nitrocellulose, benzylcellulose, or rubber; the film is stripped off after making up. A. J. Hall.

Spangled etc. leather.—See VI.

#### XVI.—AGRICULTURE.

Laws of soil colloidal behaviour. V. Ion adsorption and exchange. S. Mattson (Soil Sci., 1931, 31, 311-331; cf. B., 1931, 556).—The base-exchanging properties of isoelectrically-precipitated "silicates," phosphates," and "humates" described previously are examined. At  $p_{\rm H}$  7.0 the exchange capacities increased with the proportion of acidoid (silicate, phosphate, etc.) to ampholytoid (sesquioxide) present in all materials examined, ferric complexes showing higher capacities than the corresponding aluminium complexes. Acidoid and ampholytoid constituents are assumed to exist in partial combination with each other, the products exhibiting both acidic and basic properties. Free acidoid valences are the active agents in cation exchange. The exchange capacity can be altered by a rearrangement of the linkings. Treatment of electronegative complexes with neutral salts produces exchange acidity, and electropositive complexes similarly cause exchange alkalinity. Exchange "neutrality" does not coincide with the isoelectric point, but varies with the nature of the ions of the salt. cataphoretic and the Donnan isoelectric points do not appear at the same  $p_{\rm H}$  value. A. G. POLLARD.

Absorptive capacity of soils. J. F. RADU and T. A. RUSSEV (Bul. Acad. Inalte Studii Agron. Cluj, 1930, No. 1; Proc. Internat. Soc. Soil Sci., 1931, 6, 11—12).—In Knops' method for determining the ammonia-absorptive power of soils the period of absorption may be reduced to 2 hrs. The absorptive power of soil is related to its maximum water capacity. Absorption of calcium is mainly influenced by the type of soil and the period of contact allowed. Absorption of phosphate is affected chiefly by the period of contact. Maximum absorption of potassium is reached after 2 hrs. A. G. POLLARD.

Soil building and improvement. V. FALCKE (Oesterr. Chem.-Ztg., 1931, 34, 64-65).—Soil-like composts are described in which sand is mixed with organic matter (manure, vegetable refuse, etc.), mineral nutrients, and water-glass. Use of the last-named leads to an improved physical condition in the fermented compost. It may be used as an ameliorant for difficult soils.

A. G. POLLARD. Soil-forming processes in the Hawaiian Islands from the chemical and mineralogical points of view. H. S. Palmer (Soil Sci., 1931, 31, 253-265).-Analyses of original and weathered minerals are utilised in a discussion of the changes involved in soil formation.

A. G. POLLARD.

Conception of flow-plasticity as applied to soils. G. W. S. Blair (Soil Sci., 1931, 31, 291-298).—Experimental data indicate that variations in the flow-plasticity of soil pastes are produced by the addition of chalk or lime, by the addition of sand to heavy soils, and by the normal leaching process. Flow-plasticity measurements should be of value in characterising the physical properties of soils. A. G. POLLARD.

Soil groups and their relationship to the failure of plants, a new soil relationship. S. Goy (Pflanzenbau, 1930, 6, 351; Bied. Zentr., 1931, 60, 97—99).—Soils are classified into six groups according to their chalk and exchangeable calcium contents and their total and exchange acidities. The acid status, adsorptive capacity, and buffer power of soils are interrelated and changes in any one factor quantitatively influence the others. The bearing of these on liming practice is discussed. A. G. POLLARD.

Sampling market-gardening soils for nitrates. J. E. BLANEY and J. B. SMITH (Soil Sci., 1931, 31, 281—290).—Nitrate determinations, by the phenoldisulphonic acid method, in soils variously cropped and fertilised are recorded. The distribution of nitrates in soil was much less uniform than is usually supposed. For areas of  $\frac{1}{30}$  acre at least 50 samples were necessary to reduce the probable error for nitrate content to 5% A. G. POLLARD. of the mean value.

Soil organic matter-temperature relationship in the eastern United States. H. Jenny (Soil Sci., 1931, 31, 247-252).—In soils from the numerous areas examined the organic contents decreased exponentially with increasing mean temperature (cf. B., 1929, 407). A. G. POLLARD.

Azotobacter reaction in Moravian regional soil types. F. Kostiuk (Mitt. Czechoslov. Akad. Landw., 1930, 480; Proc. Internat. Soc. Soil Sci., 1931, 6, 21—22).—Podsols, brown earth, black earth, and rendzina (humus-carbonate) soils are examined for the presence of Azotobacter. The ability of the organism to survive in these soils in the natural condition and after chalking is tested by inoculation experiments.

A. G. POLLARD.

Relationship between the development of Azotobacter and the buffer power of soils. H. JOHL (Diss., Göttingen, 1930; Bied. Zentr., 1931, 60A, 27).—

Azotobacter can tolerate temporary acid conditions

(p<sub>H</sub> 4.5) without injury. Growth of Azotobacter is a satisfactory indicator of the buffer power of soils. For determining buffer capacity the soil is shaken with a buffer solution and, after filtration, the solution is titrated. The method is not suited to humus soils.

A. G. POLLARD.

Soil acidity, with special reference to the electrolyte content of soil suspensions. A. Teräsvuor (Valtion Maatalouskoetoiminnan Julkaisuja, 1930, No. 29; Bied. Zentr., 1931, 60A, 19—20).—Air-drying of soils prior to analysis does not, in most cases, affect the  $p_{\rm H}$  or electrolyte contents to an appreciable extent. Mineral soils dried at 100-110° have an increased electrolyte content and a lower  $p_{\rm H}$ . Peat soils are less affected. Storage of water-saturated soils causes a decrease in electrolyte content and an increased  $p_{\rm H}$ . Reverse effects are observed in soils stored at half saturation and less. Changes in  $p_{\rm H}$  resulting from additions of acid or salt solutions are most marked in soils the electrolyte content of which is small.

A. G. POLLARD.

Acidity and degree of saturation of moorland soils. E. Kasakov (Arb. Melior. Vers.-Stat. Leningrad, 1930, [5]; Bied. Zentr., 1931, 60A, 21).—Types of acidity exhibited by moorland soils are examined. The degree of saturation with bases varied considerably with the nature of the soil, averaging 12—18% in high-moor soils ( $p_{\rm H}$  3·6 approx.) and up to 91% in low-moor soil ( $p_{\rm H}$  6·8—7·0).

A. G. POLLARD.

Transient changes in acidity and the humus content of forest soil. D. Fehér (Pflanzenbau, 1930, 4, 74—87; Chem. Zentr., 1931, i, 993).—The reaction of forest soils shows regular periodic changes. The highest values (summer) correspond with the highest humus content.

A. A. Eldridge.

Acidity and plant growth. E. MORGENROTH (Pflanzenbau, 1929, 1, 434—470; Chem. Zentr., 1931, i, 993).—The  $p_{\rm H}$ , hydrolytic acidity, exchange acidity, and buffering power of loam, sandy loam, and humous sand were studied in relation to their plant-physiological utilisability.

A. A. Eldridge.

Sulphur oxidation and reaction effects in Alberta soils. O. R. Younge (Sci. Agric., 1931, 11, 535—541). —The sulphur-oxidising power of a number of soils is examined. Use of fertilisers did not affect the rate of sulphur oxidation, except in one soil treated with superphosphate. Organic matter (dried clover meal) stabilised the oxidation process, the amounts of sulphate produced appearing in steady increments. In soils of different buffer capacity no relationship existed between the change in  $p_{\rm H}$  and the amount of sulphur oxidised.

A. G. Pollard.

Nitrification [in soils]. A. Harder (Bot. Arch., 1931, 31, 312—348).—Nitrification in soil is largely dependent on accessibility of oxygen and may be depressed where other oxygen-consuming processes predominate. Intermediate products have an inhibitory effect. In some instances nitrification proceeds in soils of ph 3.0. During the activity of nitrifying organisms a peroxide is produced. A. G. Pollard.

Base exchange in soils rich in organic matter. L. Smolík (Bull. Czechoslov. Acad. Agric., 1930, 6, 912; Proc. Internat. Soc. Soil Sci., 1931, 6, 6).—The base-exchange capacity (Ca¨+Mg¨+Na˙+K˙+H˙) of highly organic soils was much greater than that of mineral soils. No relationships exist between the nitrogen content and the total exchange capacity. Removal of organic matter from soils by hydrogen peroxide reduced their exchange capacity.

A. G. POLLARD.

Electrodialysable bases in soils. L. Smolíκ (Bull. Czechoslov. Acad. Agric., 1930, 645; Proc. Internat. Soc. Soil Sci., 1931, 6, 9).—The amounts of exchangeable bases removed from soils by electrodialysis corresponded with those obtained by leaching with ammonium chloride solution. Repeated electrodialysis yielded the same values. Bases removed during electrodialysis may be determined by titration.

A. G. Pollard.

Relationships between the degree of saturation with bases and the  $p_{\rm H}$  of certain soil types. S. ODÉN and LÖDDESÖL (Medd. 368, Centralanst. försöksv. jordbruks. Avdel. lantbrukskemi, No. 40; Bied. Zentr., 1930, 60A, 20).—Changes in the  $p_{\rm H}$  of soils accompanying the removal of bases by electrodialysis are examined. The rate of removal of bases by dialysis decreases with falling  $p_{\rm H}$  to a minimum at  $p_{\rm H}$  5.2 and subsequently increases as the process continues. The first stage of dialysis (to  $p_{\rm H}$  5.2) corresponds with the removal of K', Na', and Ca' ions. In moorland soils the Ca" ions are more firmly held than in mineral soils. In the second stage much Fe" and Al" are removed. The relationship between the degree of saturation with bases ( $\beta$ ) and the  $p_{\rm H}$  may be thus expressed:  $\beta = (p_H - C_1)C_2$ , where  $C_1$  and  $C_2$  are characteristic constants of the soil examined. A. G. Pollard.

Production of carbon dioxide in arable soil and its diffusion into the atmosphere. H. Magers (Pflanzenbau, 1929, 2, 472—544; Chem. Zentr., 1931, i, 994).—An apparatus for sampling soil-air is described. The course of respiration and the effect of crops, rain, and tilling were studied.

A. A. Eldridge.

Relative solubility of the phosphoric acid of surface and sub-soils of different types. O. Engels (Z. Pflanz. Düng., 1931, 10B, 181—187).—In soils poor in phosphate, subsoils contain less phosphorus soluble in 10% hydrochloric acid, and less root-assimilable phosphorus (Neubauer) than do the corresponding surface soils. Also the subsoil phosphate has a low relative solubility. The differences in the nature and proportion of phosphate in soil and subsoil cannot be correlated with the reaction of the soil or its calcium and magnesium contents (cf. Hasenbäumer, B., 1930, 579).

A. G. Pollard.

Determination of the matrix and manner of weathering of soil from the composition of the kaolinic silicate. R. Ganssen, K. Utescher, H. Pfeiffer, A. Laage, H. Haller, and M. Trénel (Mitt. Lab. Preuss. Geol. Landesanst., 1930, No. 10, 1—24; Chem. Zentr., 1931, i, 672).—The ratios of base to alumina for the zeolitic and kaolinic portions of soil are compared and related to the weathering process.

A. A. Eldridge.

Determination of organic material in soil with chromic and sulphuric acids. G. Leoncini (Ann. Sperim. Agrar., 1930, 1, 241—246; Chem. Zentr., 1931, i, 673).—The dry soil (2—5 g., 1 mm.), coarse sand (20 g.), and dry, finely powdered potassium dichromate (25 g.) are treated (at first in the cold) with sulphuric acid (40 c.c.), the carbon dioxide being passed through barium chloride solution into 0·5N-potassium hydroxide. After air free from carbon dioxide has been drawn through the apparatus the carbonate is determined titrimetrically. Soils containing much carbonate must first be treated with phosphoric acid.

A. A. Eldridge.

Determination of plant-nutrient contents in soils. P. Lohmann (Bot. Arch., 1931, 31, 489—518).— Existing methods are compared. Analyses of soil extracts indicate much lower nutrient contents than does the *Aspergillus* method, differences being much more marked in the case of phosphates than of

potassium. The amount of soil phosphate utilised by the fungus far exceeds that indicated as being available by the customary chemical extraction methods. Values obtained by this method are 4—5 times the citric-soluble phosphate content, and approximate to the amounts extracted by boiling the soil with 50% acid. Neubauer values for assimilable phosphate were relatively small. The Aspergillus method yields values for assimilable potassium which are of the same order as those shown by the Neubauer and Wiesmann tests.

A. G. Pollard. Determination of the phosphorus requirement of soil. A. L. Maslova and O. M. Dobrotvorskaya (Udob. Urozhai, 1930, 2, 276—283).—Soils (10 g.) were treated with  $0\cdot1N$ -hydrochloric acid (2—12 c.c.) and the mixture was diluted with water to 100 c.c.; after 24 hrs. the phosphate content and  $p_{\mathbf{H}}$  value were determined. The  $p_{\mathbf{H}}$  values at which the same quantity of phosphate is dissolved differ (chernozem  $3\cdot36$ — $4\cdot45$ , dark grey loams  $2\cdot97$ ). The  $p_{\mathbf{H}}$  of soil is correlated with the response to phosphorus fertilisation.

CHEMICAL ABSTRACTS.

Chemical method for determinating fertiliser requirements [of soils] and the action of phosphatic fertilisers. L. Dworak (Z. Pflanz. Düng., 1931, 10B, 201—203).—Němec's remarks (B., 1930, 1000) are criticised. The method is purely a relative one, the limiting values assigned varying with type of soil and crop.

A. G. Pollard.

Microbiological analysis of soil fertility. IV. Nitrification and soil fertility. J. Ziemiecka (Wydane Zapomogi Akad. Nauk Technicz., 1930; Proc. Internat. Soc. Soil Sci., 1931, 6, 36).—The rate of nitrification of ammonium salts by soil organisms was examined both in silica-gel cultures and in soils. The oxidation of nitrite was consistently slower than the first stage of oxidation. The most rapid nitrification occurred in soils treated with ammonium sulphate. In unfertilised soils nitrification was slow. Nitrification is not markedly affected by phosphate deficiency in soils. In garden soils nitrification was rapid, less in field soils of  $p_{\rm H}$  7.0, and least in soils of  $p_{\rm H} < 6.4$ . The growth of Azotobacter is a more delicate test of soil reaction than is that of nitrifying organisms. (Cf. B., 1930, 160.) A. G. POLLARD.

Theory of the hydrochloric acid extract in comparison with other methods for determining soil fertility. R. G. Penseler (Kühnarchiv [Sonderb. 5], 1930, 26, 413—443; Bied. Zentr., 1931, 60, 99—102).— Methods for determining soil fertility, other than the Ganssen hydrochloric acid method, are limited by petrographic, genetic, and climatic factors. The molecular ratios as calculated by Ganssen cannot be correlated closely with acidity values in chalk soils for which too great an alkalinity is indicated. Modification of Ganssen's method, by allowing a more intensive action of acid on the soil, leads to calculated molecular ratios more closely in agreement with acidity figures. Moreover, lime-requirement values so obtained accord well with those indicated by Gehring's method. By prolonged acid extraction a decreased silica : base ratio as compared with the molecular ratio of the clay is obtained; such action of the acid is, however, too severe

for the examination of zeolites or exchangeable soil permutits. For the determination of silica in the residues from acid extraction of soils, the use of 5% sodium carbonate solution is unsatisfactory. The methods of Utescher (2.5% sodium hydroxide solution) and of van Bemmelen (4% potassium hydroxide solution) give consistent results. High proportions of chalk in soils reduce the concentration of the extracting acid sufficiently to vitiate results. The exchangeable or plantassimilable potassium, calcium, and phosphate in soils constitute only a fraction of that removed by hydrochloric acid. The exchangeable potassium determined by sodium chloride leaching agrees closely with the assimilable potassium content indicated by Neubauer tests. These values show a general parallelism with the K: Ca saturation values of Gehring. A. G. POLLARD.

Determination of the lime requirement of soils. G. Rohde (Pflanzenbau, 1929, 1, 226—272; Chem. Zentr., 1931, i, 995).—Comber's qualitative method is accurate only for sandy soils. The result of the Azotobacter test depends not only on the lime content, but also on the microbiological condition of the soil, and is qualitative only. The  $p_{\rm H}$  determination is qualitative; for sandy soil the limit is  $p_{\rm H}$  6, and for loam and clay soils  $p_{\rm H}$  7. The hydrolytic acidity of the soil depends on the salt used, the volume of the liquid, and the temperature. The exchange-acidity method gives good results with sandy soils, but low results on clayey and loamy soils. Hissink's method gives high limits for lime requirement; Gehring's method is inapplicable to soils high in lime. An improvement is suggested.

A. A. Eldridge.

Method of determining total exchangeable bases in soils. A. N. Puri (Soil Sci., 1931, 31, 275—279).— The total exchangeable base content of soils is calculated as the difference between the carbonate content determined by the author's method (cf. B., 1930, 1000), and the total base content ascertained by treatment with dilute hydrochloric acid.

A. G. Pollard.

Simplified Kappen method for determining absorbed bases [in soil]. D. V. Druzhinin and Z. I. Stroganova (Udob. Urozhai, 1930, 2, 194—199).—The Kappen and Bobko-Askinasi methods give equally good results. For approximate determination, 1 pt. of soil is treated with 2·5 pts. of 0·1N-hydrochloric acid, the mixture is shaken for 1 min., kept for 5 min., filtered, and an aliquot part is titrated, using methyl-orange. All the methods are satisfactory when the soil is free from lime and calcium phosphate and is low in absorbed bases. Chemical Abstracts.

Effect of admixture of magnesium with lime applications. O. K. ZIKHMAN-KEDROV (Udob. Urozhai, 1930, 2, 186—189).—Application to soil of magnesium carbonate had no injurious effect; it stimulated the effectiveness of calcium carbonate.

CHEMICAL ABSTRACTS.

Absorption of ammonium- and nitrate-nitrogen by various plants at different stages of growth. J. A. Naftel (J. Amer. Soc. Agron., 1931, 23, 142—158). —Absorption by cotton, wheat, and maize takes place at all stages of growth; at first the ammonium-nitrogen is the more rapidly absorbed, but later the reverse holds. The absorption of the former is increased, and of the

latter only slightly affected, when the acidity of the culture solution is decreased. The highest total nitrogen absorption took place with both forms present at  $p_{\rm H}$  6·0. Chemical Abstracts.

Comparison of [crop] yields on weakly acid and weakly alkaline soils. JORET (Compt. rend. Acad. Agric., 1930, 16, 374; Proc. Internat. Soc. Soil Sci., 1931, 6, 32-33).—In the absence of nitrogenous fertilisers wheat yields from slightly acid soils (pH 6.5— 7.0) were definitely lower than those from similar but slightly alkaline soils, especially in a dry season. Differences were small after treatment of the soils with ammonium sulphate and disappeared entirely when sodium nitrate was used. In general, on slightly alkaline soils, ammonium sulphate proved superior to nitrates. The reverse was the case on slightly acid soils. On the soils examined, the optimum  $p_{\rm H}$  for oats in a dry season was 7.0 and at pH 6.0-6.5 (the usually accepted optimum) yields were depressed. A. G. POLLARD.

Comparison of various forms of nitrogen fertilisers. E. I. Ratner (Udob. Urozhai, 1930, 2, 291—300).—Using oats, the effects of sodium nitrate, ammonium sulphate, urea, calcium cyanamide, and ammonium hydrogen carbonate on podsolised sandy loam, loam, degraded chernozem, dark chestnut-brown, Turkestan loess, and red soils were compared. For the unsaturated soils calcium cyanamide was preferable; on heavy loams ammonium sulphate was slightly superior. On limed soils ammonium sulphate was as good as were the other forms. If acid phosphate was used as the source of phosphorus cyanamide was superior, even in presence of lime. With precipitated phosphate calcium cyanamide was inferior.

Urea fertilisers. J. Bordas and G. Mathieu (Compt. rend. Acad. Agric., 1930, 16, 904; Proc. Internat. Soc. Soil Sci., 1931, 6, 33—34).—The transformation of urea into ammonium carbonate in soils is more rapid where much organic matter is present. Application to soil of cultures of ammonifying bacteria or of urease accelerated the process, notably in soils normally less active. The formation of ammonium carbonate from urea is accompanied by the dissolution of soil organic matter and a partial mobilisation of reserve nitrogen. In soils in which the urea transformation is slow a portion of the urea-nitrogen is converted into organic compounds of other types. The co-existence in such soils of urea-, ammonia-, and nitrate-nitrogen leads to considerable nitrogen losses. Such losses do not occur when urease or ammonifying organisms (in small amounts of fresh manure) is added to the soil. The latter effect is confirmed by field trials.

A. G. Pollard.

Composition and action of basic slag. A. Wilhemy (Tagung Ges. Naturf. Ärtze Königsberg, 1930; Bied. Zentr., 1931, 60A, 45).—The solubility of the phosphate of slag in water varies with the soluble silica content. By shaking slag with water and a soil poor in phosphate the soluble-phosphate content of the mixture decreases due to fixation of phosphate by the soil. With soils rich in phosphate an increased phosphate solubility is observed. The plant-assimilable phosphate in slag (Neubauer test) is paralleled by its

soluble-silica content. The soluble phosphate of slag is in the form of pure calcium tetraphosphate and not as a double compound with calcium silicate.

A. G. POLLARD.

Influence of antiseptics on the supply of nutrients in the soil. G. A. Chigarev and N. B. Myakiva (Udob. Urozhai, 1930, 2, 312—317).—The supply of nitrate is largely inhibited by carbon disulphide, but is stimulated by chlorobenzenes. The phosphorus régime is scarcely affected; the ammonia is stimulated.

CHEMICAL ABSTRACTS.

Toxic action of aluminium in connexion with plant growth. B. E. Gilbert and F. R. Pember (Soil Sci., 1931, 31, 267—273).—In water-culture experiments the yield of lettuce was not appreciably affected by changes in  $p_{\rm H}$  in the nutrient from 3·2 to 7·5, but was greatly influenced by the addition of small amounts of aluminium salts. Yields of barley in a variety of soils under different climatic conditions were more closely correlated with the amounts of "active" aluminium (Burgess, B., 1923, 617 A) in the soils than with their  $p_{\rm H}$  values.

A. G. Pollard.

[Toxicity to seeds of] nickel and cyanogen compounds. A. Niethammer (Pflanzenbau, 1930, 4, 607—634; Chem. Zentr., 1931, i, 994).—The toxicity falls in the order: potassium nickel cyanide, nickel nitrate, carbonate, chloride, sulphate; the penetrability of the seed is of importance.

A. A. Eldridge.

Utilisation of agricultural wastes. H. GILMAN, R. E. Brown, J. B. Dickey, A. P. Hewlett, and G. F. Wright (Proc. Iowa Acad. Sci., 1929, 36, 265—266).—Experiments relating to the preparation and application of various furan derivatives are summarised.

CHEMICAL ABSTRACTS.

Effect on sugar-cane culture of fertilisation with molasses. G. Dorfmüller (Deut. Zuckerind., 1930, 55, 1230—1231; Chem. Zentr., 1931, i, 1026—1027).—
The effects on the biological processes in the soil are discussed; the nitrate-nitrogen may completely disappear by conversion into organic nitrogen compounds. Use of molasses with ammonium sulphate is advantageous.

A. A. Eldridge.

Utilisation of nutrients by sugar beet. BOTTRICH (Ernähr. Pflanze, 1931, 27, 103—105).—Field trials with sugar beet are recorded in which heavier dressings of potash fertilisers gave proportionally greater relative increases in yield than lighter dressings. It is suggested that beet are able better to utilise potash supplies in soil when the latter is saturated with potash.

A. G. POLLARD.

Time for applying sodium nitrate to sugar beet. E. T. Sykes (J. Min. Agric., 1931, 38, 162—170).— Applications of sodium nitrate, up to 3 cwt. per acre, may be made with the seed of sugar beet without loss of sugar yield.

A. G. Pollard.

Ensilage of beet leaves of poor feeding value. G. Ruschmann and G. Gräf (Bied. Zentr., 1931, 60A, 17—18).—The ill-effects of feeding beet leaves to milch cows are examined. In certain districts beet leaves had a higher oxalate content than normal and were alkaline. The oxalate content was considerably reduced by ensiling the leaves, both hot- and cold-fermentation

processes giving similar results. The cold-fermented material still produced injurious effects, which are ascribed to the high nitrate content of the leaves. More suitable manuring of the growing beet with reduced dressing of sodium nitrate is recommended for the production of leaves of lower oxalate and nitrate contents.

A. G. POLLARD.

Fodder experiments with soured and with dried beet slices with added lactic acid. K. Pospišil and V. Sázavský (Z. Zuckerind. Czechoslov., 1931, 55, 390—394).—Cows fed with the dried beet slices to which lactic acid had been added failed to produce the same favourable effect on milk production which results from the customary use of soured slices. On changing over from soured slices to dried slices, besides a decrease of milk the live weight also diminished. On drying the slices the fodder value of the product is diminished by about 20%.

J. P. OGILVIE.

Fertiliser trials with potatoes. K. Vieweg (Ernähr. Pflanze, 1931, 27, 135—137).—The use of 40% potash salts (potassium and magnesium sulphates) in potato trials produced heavier crops than potassium chloride of equivalent potassium content, and, moreover, increased the resistance of the plants to drought conditions.

A. G. Pollard.

Manuring of early potatoes. T. Remy (Ernähr. Pflanze, 1931, 27, 145—148).—A discussion of the fertiliser requirements of early and late potatoes with reference to the time of application and relative availability of fertilisers used.

A. G. Pollard.

"Leaf diagnosis" of Lagatu applied to potatoes, with special reference to the root-soluble nutrient content of soils [Neubauer]. W. Sandrock (Z. Pflanz. Düng., 1931, 10B, 188—201).—The phosphorus, potassium, and nitrogen contents of potato leaves varied with the nutrient condition of the soil and with the nature and quantity of fertilisers applied. Relationships exist between differences in the composition of the leaves and the effect of fertilisers on crop yield. Variations in leaf composition cannot be correlated with Neubauer values for soil nutrients, and it is suggested that during the growth period plants utilise nutrients other than those registered by Neubauer tests at one particular growth period. In preparing leaves for analysis, drying whole leaves at 105° for 3 hrs. is satisfactory. A. G. POLLARD.

Manuring of acid soils. H. Kappen (Ergeb. Agrik. Chem., 1930, 2, 77—87; Bied. Zentr., 1931, 60A, 42).—
Increased soil acidity following applications of ammonium sulphate is the direct result of the nitrification process rather than the outcome of a specific physiological acidity of the ammonium sulphate itself. Use of rock phosphate on acid soils gives unsatisfactory results. Applications of potash salts to strongly acid mineral soils sometimes leads to a form of plant injury which may be partly remedied by treatment with magnesium salts.

A. G. Pollard.

Peat manuring. A. A. Kuprianov and N. S. Rosanov (Arb. Centr. Torfstat., Moscow, 1930, 2, 9—40; Bied. Zentr., 1931, 60A, 31).—The water-soluble phosphate in composts of sphagnum peat and phosphorite

was 20—30 times that of similar composts made with wood sedge, and represented 32% of the total phosphate content of the phosphorite. Manure made with peat litter produced heavier crops and was more rapidly nitrified in soil than that made with straw.

A. G. POLLARD.

Manuring experiments on the Government rubber estate "Serpong." W. C. VAN HEUSDEN and J. S. VOLLEMA (Arch. Rubbercultuur, 1931, 15, 125—146).—Experiments on a backward estate with poor soil which had been under cultivation for centuries show that green manures or nitrogen fertilisers have little beneficial effect. Definite benefit, however, accrues from green manuring in conjunction with phosphate-potash fertiliser.

D. F. Twiss.

Manurial action of the anions and secondary constituents of potash salts. O. Eckstein (Ergeb. Agrik. Chem., 1930, 2, 125—142; Bied. Zentr., 1931, 60A, 43).—In soils of low adsorptive capacity the growth of oats was depressed by the Na ion, but on strongly adsorptive soils of low sodium content treatment with sodium salts had a favourable effect. Treatment with magnesium salts produced stronger plants of increased magnesium content. Growth was improved by applications of sulphate only when used simultaneously with magnesium salts. No beneficial effects of the chloride ion was observed.

A. G. Pollard.

Behaviour of and profit from different varieties [of crops] with increased nitrogen manuring. H. Nilsson-Ehle (Z. Pflanz. Düng., 1931, 10B, 169—181).
—Different varieties of an individual crop responded very differently in yield and to some extent in quality to nitrogenous fertilisers. Varietal differences in this respect were more apparent with the heavier dressings of fertiliser.

A. G. Pollard.

"Finger-and-toe" and manuring. J. Kreuz-Pointner (Ernähr. Pflanze, 1931, 27, 172—173).—The adoption of appropriate fertiliser schemes in the prevention of finger-and-toe disease is only reliable if applications of farmyard manure are omitted.

A. G. POLLARD.

Fruit tree carbolineums. E. Profft and G. Goetze (Zentr. Bakt. Par., 1931, II, 83, 127—164).— Chemical examination of numerous carbolineum preparations is recorded, and the physiological effects of the constituents on insects and plants are examined. Chemical characteristics of suitable preparations of this class are enumerated.

A. G. Pollard.

A rust of Clingstone peaches in California.
M. C. Goldsworthy and R. E. Smith (Phytopath., 1931, 21, 134—167).—Urediniospores of the fungus were more susceptible to sulphur and sulphur compounds than to copper salts, whilst mineral oil stimulated their germination.

A. G. Pollard.

Fingerling's process for the ensilage of green plants. M. Gerlach (Tierernährung, 1930, 1, 179—193; Bied. Zentr., 1931, 60, 109—112).—The process (addition of hydrochloric acid to the fodder in the silo to produce  $p_{\rm H}$  2) is discussed. Better results are obtained by increasing the amount of acid-liquor acid and the pressure applied at the surface. The proportion of acid necessary varies with the nature of the fodder and its

moisture content. The covering layer of oil, originally used, is unnecessary if the acid is added in portions as the silo is filled. The organic acid content of silage so produced is small.

A. G. Pollard.

Stimulation of Lemna major by organic matter under sterile and non-sterile conditions. N. A. CLARK and E. M. ROLLER (Soil Sci., 1931, 31, 299-309; cf. B., 1924, 484).—Lemna major grew for several years in purely inorganic but non-sterile media. Addition of small amounts of extracts of soil, manure, vegetable matter, etc. under these conditions stimulated reproduction, there being a simultaneous increase in the micro-organisms present in the media. Under sterile conditions the organic materials were without effect on or reduced reproduction, but the original stimulative effects were produced by reinoculation. Sterile plants grew more rapidly than non-sterile. Pure cultures of certain bacteria in the presence of organic matter increased reproduction in sterile media, but others were ineffective. None of the pure organic substances examined caused stimulation under sterile or non-sterile conditions. A method of sterilising the plants by the use of potassium mercuric iodide and bleaching powder A. G. POLLARD.

Ability of dry fungicides to adhere to seeds. A. Simski (Udob. Urozhai, 1930, 2, 206—212).—Experiments with wheat, oats, and barley, employing calcium arsenate, Paris green, copper carbonate, and copper sulphate, are described. Copper carbonate and sulphate adhere best to wheat, and calcium arsenate adheres best to oats.

Chemical Abstracts.

Superphosphate from apatite. Solubility of Bordeaux mixture.—See VII. Butyric fermentation in silage.—See XVIII. Turnip tops.—See XIX.

See also A., May, 596, Soil erosion. 660, Effect of nitrogen on plant yield. Nodule bacteria and leguminous plants.

PATENTS.

Manufacture of phospho-nitrogenous manures, ammonium phosphate, and sodium bicarbonate. Soc. D'Etudes Scientif. et d'Entreprises Industr. (B.P. 344,962, 21.12.29. Holl., 27.12.28).—A mixture of carbon dioxide with phosphorus oxides is used instead of carbon dioxide alone in the ammonia-soda process. After removal of the precipitated sodium bicarbonate, the liquor is concentrated to decompose ammonium carbonate and to crystallise part of the excess sodium chloride; the mother-liquor on cooling deposits a mixture of ammonium chloride and phosphate suitable for use as a fertiliser. Alternatively, the liquor remaining after removal of the sodium bicarbonate is enriched in ammonium phosphate by further treatment with ammonia and the mixed gases. L. A. Coles.

[Fertilisers from] sewage.—See XXIII.

#### XVII.—SUGARS; STARCHES; GUMS.

Importance of a preliminary purification of (beet) diffusion juice. I. B. Mintz and B. E. Krasilshchikov (Nauk. Zapiski Tzuk. Prom., 1930, 9, 515—537).—Diffusion juice should be filtered through asbestos or other fibrous material. The total nitrogen content of

carbonatation juices, obtained from diffusion juices previously boiled or treated with 0.5—1% of kieselguhr, is 9—12% lower than that of ordinary treated juice. Good clarification is obtained by sulphitation to  $p_{\rm H}$  4.0 at 20—40°, followed by neutralisation to alkalinity of 0.1 with lime and rise of temperature to 80°, and then by further filtration and carbonatation to alkalinity of 0.01—0.02. Chemical Abstracts.

Bleaching effect of washing the massecuite of the second skip. V. V. TSCHESHCHEVIK (Zhur. Sakh. Prom., 1930, 4, 305—308).—When massecuite was washed with greens (d 1·26) the yield of yellow sugar decreased by 9·5%, with an increase in purity of 0·9%, the colour decreased by 47·9% and the centrifuging time by 19·2%. Values for washing with hot final molasses were 1·5, 0·9, 23·1, 10%, and for washing with 3·4—5·0% of its weight of molasses at 90—95% 9·2, 3·7, 83·7, and 25%. CHEMICAL ABSTRACTS.

Conditions for increasing the velocity of crystal-lisation in "sugar loaves." M. I. Nakhmanovich and I. F. Zelikman (Nauk. Zapiski Tzuk. Prom., 1930, 9, 450—492).—The best cooling-room temperature is 5—10°. The quality of the sugar is unaffected by cooling to the lower temperature.

CHEMICAL ABSTRACTS.

Cold aqueous digestion [method of determining sugar in the beet]. J. Vondrak and B. Zimmermann (Z. Zuckerind. Czechoslov., 1931, 55, 395—399).— Using pulp prepared by the Herles press, this method gives distinctly lower results than does hot aqueous digestion, the difference depending on the mesh of the sieve used in the press. It is recommended that the hot aqueous method only be employed for fresh slices for factory control purposes, though the cold method be retained for exhausted slices. J. P. OGILVIE.

Electrical conductivity of molasses for afterproduct control. J. Peller (Z. Zuckerind. Czechoslov., 1931, 55, 399—403).—A study of the relationship between the saccharisation (Balling degree) and the electrical resistance of beet molasses showed that besides the water content its composition exerts a great influence on the conductivity of the undiluted product. In the presence of grain the resistance of the product is increased. J. P. OGILVIE.

Iodometric determination of sugar. M. S. Filosofov (Nauk. Zapiski Tzuk. Prom., 1930, 9, 586—589).—Diluted molasses is inverted with hydrochloric acid, cleared with lead acetate, sodium carbonate, and alumina, and treated with Fehling's solution; the excess of copper is determined by addition of potassium thiocyanate and iodide, followed by titration with thiosulphate.

CHEMICAL ABSTRACTS.

Measurement of turbidity with a spectrophotometer, with especial reference to sugar-house products. R. T. Balch (Ind. Eng. Chem. [Anal.], 1931, 3, 124—127).—Existing methods of determining the turbidity of sugar solutions are subject to large errors through lack of colour compensation. A method is described, in which light of a definite wave-length is used and the colour is compensated by using as a standard a portion of the same sugar solution from which suspended solids have been removed. Filtration through

paper, with the aid of a slow-filtering grade of commercial kieselguhr, is recommended in place of asbestos or sand for the preparation of the standard. The turbidities are expressed in  $-\log t$  values, using the equation  $-\log t = (-\log T)/cb$ , where t is the transmittancy, reduced to unit conditions of concentration and depth of solution, c is the concentration in g. per c.c., b the thickness of the solution in cm., and T the transmitted fraction of incident light. This is recommended as being the most convenient method, in spite of the fact that values of —log T are not strictly proportional to the concentration or depth of the suspension. Cube sugar showed distinctly lower turbidity than standard granulated sugars. The turbidity of raw cane sugars can be correlated roughly with the ease of filtration.

E. S. Hedges. Sugar-cane culture. Sugar beet. Beet slices as fodder.—See XVI. Evaluation of honey.—See XIX.

#### XVIII.—FERMENTATION INDUSTRIES.

Bakers' yeast. II. Quick-acting yeast. E. ROSENBAUM (Z. Unters. Lebensm., 1931, 61, 80—84; cf. B., 1931, 217).—Ten samples of quick-acting bakers' yeast (variously known as record-, double-, noble-, and strong-yeast) were examined, and the high rates obtained for fermentation (especially initially), carbon dioxide evolution from 10% sucrose, and increase in dough volume (at 30°) are considered of especial value to bakers. Analyses gave: water 72·1—74·4%, acidity 0.7-2.2 c.c. of 0.1N-sodium hydroxide per 10 g., N 50·92—59·03% (dry solids),  $P_2O_5$  2·36—4·38% (dry solids), total fermentation period 147—186 min. (191—266 after 14 days at 20°). J. GRANT.

Dissolution and precipitation of the a-resin of hops (humulone) during wort boiling. W. Windsch, P. Kolbach, and C. Vogl (Woch. Brau., 1931, 48, 139—144, 149—152).—To 150 c.c. of deoxygenated wort 5 c.c. of an alcoholic solution of humulone were added. The wort was boiled under reflux for various periods and cooled in an atmosphere of nitrogen. The coagulum was collected and washed on a filter-paper and the flask finally rinsed with alcohol into the filtrate. From this the resin was extracted by acidifying and shaking with 30 c.c. of chloroform, from which, after washing and centrifuging, 15 c.c. were pipetted. This was evaporated in vacuo and the residue cooled in a current of carbon dioxide and weighed. The coagulum was treated with 30 c.c. of alcohol and 175 c.c. of water before extraction with chloroform. Corrections were determined and applied for the solubility of the chloroform in the aqueous liquid, also for material extracted from the wort. Duplicate determinations agreed to about 1% on the portion recovered from the wort, or to about 3% on the total recovery. With increasing hydrogen-ion concentration the amount of resin in the wort was found to decrease, as was expected, but the amount recoverable from the coagulum was insufficient to balance this, hence the total recovery decreased with increasing acidity. This is the reverse of the result obtained by boiling humulone in pure buffer solutions (B., 1928, 31), and is attributed to the occlusion of a portion of the resin in the colloid coagulum, in a state

resistant to extraction by chloroform. This view is supported by the facts that when wort of low protein content was used, the apparent loss was less than with normal worts; that longer boiling, hence greater protein precipitation, increased the loss, though the amount of resin recoverable from the coagulum varied irregularly; and that the loss was less when part of the protein was precipitated by boiling before addition of the humulone. The actual apparent losses at  $p_{\rm H}$  6.5, 6.0, 5.5, and 5.0 were with normal worts  $5 \cdot 2$ ,  $7 \cdot 2$ ,  $8 \cdot 9$ , and  $10 \cdot 6\%$ , and with low-protein worts  $3 \cdot 2$ ,  $4 \cdot 6$ ,  $5 \cdot 5$ , and  $6 \cdot 1\%$ , respectively. At the lowest acidity practically all the humulone added (165-187 g./litre) would be in solution as a salt, whilst with increasing acidity larger amounts would exist as the almost insoluble free acid, and would be much more liable to occlusion. The smaller losses observed under the more alkaline conditions do not greatly exceed those actually due to decomposition (1-3%) and previously noted on boiling humulone F. E. DAY. with buffer solutions.

Decarbonatation of brewing waters. J. H. Collett (J. Inst. Brew., 1931, 37, 274—277).—Carbonates are precipitated from the water by the addition of calcium hydroxide. The small amounts of calcium hydroxide and calcium carbonate which still remain in solution are decreased by the addition of calcium chloride, and, after the clear liquid is run into a second tank, the residual amount in solution is exactly neutralised by the addition of acid, or, preferably, by potassium or sodium bisulphate. The sedimentation of the calcium carbonate is completed in 3 hrs. if the addition of the constituents to the water takes place at 65°. C. RANKEN.

Decarbonatation of mashing liquor [in brewing]. R. L. SIAU (J. Inst. Brew., 1931, 37, 272—274).—The raw water is pumped through an exchanger in which it is heated to 43° by the outgoing boiling water. It then enters the condenser in which it is heated to 99° by the steam from the boiling tanks, and thereafter passes through a steam-jacketed pipe before entering the tanks which are provided with steam coils and steam vents. By using this method of regenerative heating, the carbonate content of the water can be reduced to 2.5 pts. per 100,000 without using much more steam than is required to raise the temperature of the water to 65°. C. RANKEN.

Plastered wine. A. Borntraeger (Z. Unters. Lebensm., 1931, 61, 1—38).—Little potassium hydrogen sulphate results from the reaction of tartar with calcium sulphate in grape must treated with gypsum. The variation in acidity of wines from highly plastered musts may be due to reaction, in the presence of alcohol and potassium hydrogen sulphate, of free tartaric acid with the potassium salts of organic acids, and the lowering of the acidity by precipitation of tartar as an acid salt. Plastering therefore raises the "acid energy" (i.e., power to produce acidity) without necessarily raisin the actual acid value. The solubility of calcium sulphate is lowered by potassium sulphate and by alcohol, and raised to slight extents by tartaric, malic, lactic, succinic, and acetic acids (in decreasing order), but not by tannic acid. In alcohol at 96°, equivalent mixtures of potassium sulphate and tartaric acid are converted into

tartar and potassium hydrogen sulphate, the latter then being decomposed into potassium sulphate and sulphuric acid, only the sulphuric acid and residual tartaric acid being soluble. In winter new wine deposits more potassium bitartrate than does old wine, especially if it has been highly plastered. Other points investigated are the formation of potassium hydrogen sulphate in highly plastered wines, the effects of the constituents of wine on the reaction between tartar and gypsum, the separation of calcium sulphate during fermentation, the solubility of tartar in aqueous solutions of the organic acids of wines and of their potassium salts, and the relation of the alkalinity of the ash to the gypsum content.

J. Grant.

Detection of fruit wine in grape wine by the dibenzylidenesorbitol method. M. KLOSTERMANN and W. FACHMANN (Z. Unters. Lebensm., 1931, 61, 100—103).—The wine is decolorised with animal charcoal, the filtrate evaporated under reduced pressure, and the residue extracted with absolute methyl alcohol to eliminate pectins and dextrin. Amino- and organic acids etc. in the extract are precipitated by lead acetate, the excess of lead in the filtrate is removed with hydrogen sulphide, and the extract is evaporated under reduced pressure and acetylated with 3 c.c. of pyridine and acetic anhydride on the water-bath for 15 min. After neutralisation, the resulting oil is extracted in ether and isolated by evaporation and recrystallisation from a solution in hot water (m.p. 98-99°). The method is sensitive to additions of 2.5% of fruit wine (or less if Jahr's microscopical method is used; cf. B., 1930, 787), and is more rapid and reliable than the production of dibenzylidenesorbitol (Werder, B., 1929, 619; also von der Heide and Hennig, B., 1929, 574, 735).

Use of the quartz lamp in the examination of raisin and currant wine. P. Berg and E. Kröger (Wein u. Rebe, 1930, 12, 327—331; Chem. Zentr., 1931, i, 699).—Substances which inhibit luminescence are removed by treatment with carbon; a blue coloration then indicates the presence of raisin wine. The luminescence is given also by fermentation products of other dried fruits.

A. A. Eldridge.

Methyl alcohol in alcoholic drinks. I. Methods of testing and its determination. N. A. Espinosa (Anal. Asoc. Quím. Argentina, 1930, 18, 57—73).— Two methods of determination have been used, after slight modification, for large numbers of analyses. In the Denigès-Villavecchia method the colour is matched with that given by standard solutions after keeping in the cold for 15 hrs. It is sensitive to 0.0002 c.c. of methyl alcohol. The Chapin method, using the Schiff, and not the Schiff-Elvove, reagent, is sensitive to 0.00008 c.c. Glycerol, which is separated by distillation, and formaldehyde, which is allowed for by a blank determination before oxidation, are the only substances liable to interfere with the determination. Rothera's reaction can be used for the determination of acetone.

R. K. CALLOW.

J. GRANT.

Butyric fermentation in silage. G. Ruschmann and L. Harder (Bied. Zentr., 1931, 60A, 18—19).— New methods for determining the number of Amylobacter in silage are described. Results are closely paral-

leled by chemical determinations of the butyric acid present. In silage to which sugar was added the number of *Amylobacter* was small.

A. G. POLLARD.

Sterilisation of enzymes.—See XIX.

See also A., May, 653, Diastase from wheat. 655, Protein crystals possessing tryptic activity. Cellulose-fermenting organism. 656, Acetic fermentation. Propionic group of bacteria.

## PATENTS.

Manufacture of a product containing vitamin [-B]. A. CARPMAEL. FROM I. G. FARBENIND. A.-G. (B.P. 345,669, 22.11.29).—A yeast extract obtained by liquefying pressed yeast is added to an aqueous solution of a fermentable type of sugar and the whole fermented by a distillery yeast or, preferably, a wine yeast.

C. RANKEN.

#### XIX.—FOODS.

Sterilisation of flours and pulverised enzymes.

A. J. J. Vande Velde (Bull. Acad. roy. Belg., 1930, [v], 16, 1396—1403).—The specific activities of malt amylase, powdered pepsinase, takadiastase, trypsinase, pancreatin, and rennin are undiminished, and in some cases slightly increased, by one or two treatments with carbon disulphide. They are, however, completely sterilised.

C. W. Gibby.

Predetermining flour soundness. D. A. COLEMAN (Amer. Miller, 1930, 58, 434).—Maximum normal fatacidity values of wheat are: hard red winter 8.5, hard red spring 5.5, soft red winter 8.0, durum 4.0, white 5.5. Damaged wheat gave values between 8.6 and 44.

CHEMICAL ABSTRACTS.

Pigment of cereals. O. Schamberg (Z. Ges. Mühlenwesen, 1930, 7, 105—106; Chem. Zentr., 1931, i, 866).—The location and artificial variation of the colour are discussed, and means for the detection of sulphur, organic dyes, dichromate, and polishes are described.

A. A. ELDRIDGE. Determination of mixtures of rye and wheat products in flour and bread. Recognition of patent flours. K. Seidel (Z. Ges. Getreidew., 1930, 17, 206— 212; Chem. Zentr., 1931, i, 1034).—Sedimentation tests on dried, sieved crumb in solutions of formaldehyde, chloral hydrate, sodium hydroxide, potassium thiocyanate, sodium monohydrogen phosphate, and calcium nitrate are described. Rye bread gives the greater volume of deposit and exhibits the longer separation time. Conditions affecting the test and the viscosity of alkaline extracts are mentioned. Aqueous extracts cannot be differentiatiated by Millon's test: the biuret reaction gives a blue colour which with rye becomes yellow at 80°. Colours obtained with alkaline and alcoholic extracts in the biuret, iodine, and other colour reactions are described. Tillmans' trifructosan determination affords a qualitative distinction. With Molisch's reaction rye bread gives a dark wine-red, and wheat bread a faint pink solution, mixtures giving intermediate tints. Extracts of patent wheat flour give deep, and of rye faint, colorations with violetblack. A. A. ELDRIDGE.

Coagulation of milk. M. Chrò and S. Repetto (Boll. Soc. Ital. Biol. Sper., 1929, 4, 3 pp.; Chem. Zentr., 1931, i, 1033—1034).—Sodium oleate and stearate coagulate milk with different velocities. Calcium oleate retards, and calcium stearate accelerates, coagulation, whilst the effect of the calcium salts of milk-fatty acids is intermediate. A. A. ELDRIDGE.

Determination of nitrogenous matter in milk by means of the decrease in conductivity. STROHECKER (Z. Unters. Lebensm., 1931, 61, 69—80).— The constitutent sugars, fats, and salts exercise only a small constant influence on the conductivity of milk, marked differences being produced by changes in protein content. The conductivities (1) of 25 c.c. of milk diluted with (1) 25 c.c. of conductivity water, and (2) 25 c.c. of 0·1N-hydrochloric acid, and (3) a mixture of 12.5 c.c. of 0.2N-acid and 37.5 c.c. of water, were determined under similar conditions, and the value  $l_{\rm F}{=}(l_1{+}l_3{-}l_2)$  plotted for a number of milks. The resulting straight line ( $l_{\rm F}$  115 for 22% N and 145 for 3.4% N) enables the nitrogen content of any milk to be determined from  $l_{\rm F}$ . The method is rapid and gives close agreement with the Kjeldahl method for normal, watered, and condensed (unsweetened) milks, and with milks from diseased animals. It is unaffected by acidity or by small amounts of preservatives, but sodium carbonate or sucrose (e.g., in sweetened-condensed or ordinary milk) increases l, a correction for the latter being made by deducting from the nitrogen content the same percentage of its value (usually 10%) as the percentage of sucrose present. J. GRANT.

Refractive index of milk. A. Gronover and F. Türk (Z. Unters. Lebensm., 1931, 61, 85—94).—The sp. gr., acidity, n, and dry non-fatty solids are tabulated for milks as supplied (145), collected (104), and drawn from a single cow (39). In each case the last two values are approximately linearly related, but the graphs are too irregular to allow the method to be used even for sorting purposes (cf. Mundinger, B., 1929, 795).

J. Grant.

Determination of  $p_{\rm H}$  of cheese whey. G. Wode (Lait, 1930, 10, 1083—1087; Chem. Zentr., 1931, i, 706).—An electrometric apparatus is described.

A. A. ELDRIDGE.

Viscosity of egg-albumin, and changes in fresh and preserved eggs. D. Cortese (Ann. Sperim. Agrar., 1930, 3, 125—132; Chem. Zentr., 1931, i, 865).—The viscosity of the albumin of eggs stored at the ordinary temperature decreases in spite of evaporation; in cold storage the decrease is slower, but in both cases rectilinear. The decrease is very rapid in bad eggs owing to the action of liquefying bacteria. A. A. Eldridge.

Determination of caffeine in coffee, tea, and maté. J. Grossfeld and G. Steinhoff (Z. Unters. Lebensm., 1931, 61, 38—56).—The following procedure is based on the Lendrich-Nottbohm (B., 1909, 379) and Fendler-Stüber (cf. Pritzker and Jungkunz, B., 1926, 605) methods, but is quicker, and avoids errors due to adsorption of caffeine, to incomplete removal of impurities, to loss of caffeine by oxidation, and to oxidation of fats to water-soluble fatty acids. A mixture of the ground, ammoniacal sample and sand is extracted under reflux with carbon tetrachloride, and the residue after evaporation re-extracted with hot water,

and (except with tea or raw coffee) the solution oxidised in the cold with 5% potassium permanganate solution (15 min.), the excess of which is removed with 10% sodium thiosulphate after addition of copper sulphate. The mixture is precipitated with N-sodium hydroxide, filtered, the filtrate extracted with chloroform, and the extract weighed after evaporation. A Kjeldahl nitrogen determination serves as a check. The results are satisfactory, though Hag coffee yields a small nitrogen-free impurity (0.02%), and coffee substitutes give high nitrogen values (0.007—0.015%). The results for tea are higher than those obtained by the Lendrich-Nottbohm method.

Evaluation of honey. I. L. LABAND, W. BARTELS, and A. FAUTH (Z. Unters. Lebensm., 1931, 61, 56—69). Gothe's diastase test (cf. Fiehe and Kordatzki, B., 1928, 543, 911) has been tested on 200 German and foreign honeys, and satisfactory results were obtained. A limiting value of 8.3, corresponding with a fermenting power of 17% (i.e., diastase value  $50 \equiv 100\%$  fermentative power), is suggested for honey marketed under the German (March, 1930) regulations, which state that honey should not be heated so as to destroy or appreciably to weaken the diastatic power. The appearance of a stable cherry-red colour in Fiehe's test (loc. cit.) is not always evidence of the presence of artificial invert sugar, since it is also produced by strongly heated unadulterated honeys. The colour of the Fiehe test increases with duration and temperature of heating, and there is evidence of a relationship between the intensity and the J. GRANT. diastatic power.

Juice of sour raspberries. R. Cohn (Deut. Destillateur-Ztg., 1930, 51, 522; Chem. Zentr., 1930, ii, 2453).—The volatile acidity is high, and the non-volatile low (0·36—0·67% instead of 1·0—1·4% as citric acid).

A. A. Eldridge.

Effect of washing of ensiled turnip tops on the loss of crude and digestible nutrients. F. Honcamp and W. Schramm (Bied. Zentr., 1931, 60A, 17).—Washing of turnip tops either before or after ensilage does not involve appreciable losses in nutrient value.

A. G. POLLARD.

Examination for benzoic acid in food products. U. Hordh (Anal. Asoc. Quím. Argentina, 1930, 18, 31—36).—In order to isolate the benzoic acid free from disturbing impurities the material is distilled in steam, the distillate extracted with ether, the extract washed with a little water, and the residue obtained by evaporation is dissolved in the minimal amount of water and tested by Jonescu's reaction (oxidation by hydrogen peroxide in presence of ferric chloride). This is best carried out in the cold, when a stable and intense violet coloration develops after 24 hrs. R. K. Callow.

Use of buffers in the determination of colour [in foods] by means of titanium trichloride. II. O. L. Evenson and R. H. Nagel (Ind. Eng. Chem. [Anal.], 1931, 3, 167—169; cf. Evenson and McCutchen, B., 1928, 704).—Dyes in foods may be determined by reduction with standard titanium trichloride solution in the presence of buffer solutions which also act as catalysts. It is essential to use the correct buffer solution with each individual dye and suitable buffers

are listed for all the water-soluble coal-tar dyes permitted in foods. In all cases it is found possible to use either sodium citrate or sodium hydrogen tartrate.

E. S. Hedges.

Bacteria and moulds of milk curd. Rôle of symbiosis in the ripening of Camembert cheese. F. Sansonetti (Lait, 1930, 10, 627—640, 782—793, 858—874, 989—1001, 1009—1023; Chem. Zentr., 1931, i, 703—704).

Tinplate containers and food products.—See X. Beet leaves. Beet slices as fodder.—See XVI. Bakers' yeast. Silage.—See XVIII.

See also A., May, 627, Tea tannin. 644, Colouring matter of milk. 658, Vitamin-A in maize. 659, Vitamin-B in peanut.

PATENTS.

Conditioning of grain. J. A. Hall and W. F. C. George (B.P. 344,538, 12.12.29).—Grain passing through a tower is submitted to the action of gaseous or liquid chemicals in a totally enclosed recirculating plant. The process may be used for sterilising the grain, or for adjusting the  $p_{\rm H}$  or the enzymic activity.

E. B. HUGHES.

Conservation of heat in milk dryers. C. C. Kershaw and H. L. Solie (U.S.P. 1,776,030, 16.9.30. Appl., 10.5.29).—The wet exhaust from the drum dryers passes through traps and into an expansion apparatus, and its temperature is thus lowered from about 150° to 100° without serious loss of heat. This heat is then used to preheat the milk before drying. E. B. Hughes.

Preservation of animal and vegetable substances. R. Willstätter (B.P. 344,394, 4.12.29).—Meat, fruit, etc. is preserved for several weeks by storage in a closed vessel in an atmosphere containing 0.004-0.01 vol.-% of hydrogen cyanide. Free water, iron, and alkalis must be excluded. The residual acid has no toxic effect, but may be removed by aeration if desired.

E. B. Hughes.

Production of jellifying substances. H. G.
Loesch, Assr. to Gen. Foods Corp. (U.S.P. 1,776,362,
23.9.30. Appl., 29.12.25).—To the cool aqueous extract
of citrus pomace about 0.09% of starch (in solution) is
added to remove the turbidity due to hesperidin. The
almost clear supernatant pectin solution is then drawn
off, filtered, concentrated in vacuo, precipitated with
alcohol, pressed, and dried.

E. B. Hughes.

Smoke-flavour and aroma-imparting binder for food products etc. J. Alsberg, Assr. to B. Heller & Co. (U.S.P. 1,777,026, 30.9.30. Appl., 31.10.25).— Maple, hickory, beech, or birch wood, etc., preferably the first, in small pieces is destructively distilled in the presence of a limited supply of air. The distillate, which contains the flavouring constituents, is evaporated to half its weight by exposure in shallow pans or on a rotating drum to an air blast, to remove deleterious constituents. The desired flavour is imparted to the binder, such as flour, e.g., for sausages, by mixing in about 10% by wt. of this concentrate. E. B. Hughes.

Milling of chocolate etc. Dough mixer. Press for extracting liquids. Centrifugal separator for milk.—See I. Product containing vitamin-B.—See XVIII.

# XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Cresol soap solutions. J. Lévolt (Magyar Gyóg. Társ. Ert., 1930, 6, 474—480; Chem. Zentr., 1931, i, 657).—For the determination of cresol in liquor cresoli sap. the soap is preferably precipitated with barium chloride, the cresol being extracted from the acidified liquid with light petroleum and determined gravimetrically. In presence of resin and naphtha acids the cresol soap solution is treated with concentrated calcium chloride solution; the cresol is distilled with steam and extracted with alkali from the distillate before acidification and extraction.

A. A. Eldridge.

Tincture of iodine: variations and adulteration. Tincture prepared from denatured alcohol. G. Panoroulos and J. Megalokonomos (Ext. Prakt. Acad. Athènes, 1927, 2, 15 pp; Chem. Zentr., 1930, ii, 2405).—The formation of ethyl iodide and hydrogen iodide occurs independently of the action of light, even in 85% alcohol, but it is prevented by the addition of potassium iodide. The tincture prepared from alcohol denatured with 5% of wood spirit, 25% of acetone, 0.5% of petroleum, and traces of dyes quickly forms hydrogen iodide and organic iodine compounds, chiefly iodoacetone.

L. S. Theobald.

Refractometric examination of tinctures. F. Wratschko and J. Kowarz (Pharm. Presse, 1930, 97—99, 115—122, 131—135, 146—152; Chem. Zentr., 1931, i, 655).—The alcohol and extract contents are obtained graphically from determinations of d and  $n_D$ . The application of the method is discussed.

A. A. Eldridge.

Coffeinum natrium benzoicum. A. Heray (Magyar Gyóg. Társ. Ert., 1930, 6, 468—473; Chem. Zentr., 1931, i, 656).—A double salt is formed by 2 mols. of caffeine and 3 mols. of sodium benzoate. The method of analysis is described. A. A. Eldridge.

Titration of Solutio arseniatis natrici. E. V. Christensen (Arch. Pharm. og Chem., 1930, 37, 350—358; Chem. Zentr., 1931, i, 822).—When the sodium arsenate content is up to 25% greater or less than is prescribed, the method of the Danish Pharmacopeia (1907) gives good results if a 0·2—0·5 c.c. excess of silver nitrate is used.

A. A. Eldridge.

Seasonal variations in alkaloid content of Indian Ephedra species. T. P. Ghosh and S. Krishna (Arch. Pharm., 1930, 268, 636-643).—The alkaloids from Ephedra nebrodiensis and E. Gerardiana contain 60-70% of ephedrine; those from E. intermedia usually about 10% (50% in samples from Baluchistan). E. intermedia from the Chini Range, Bashahr Division, Punjab, is a convenient source of ψ-ephedrine (1.8% of the drug). The altitude of the source does not affect the alkaloid content. A mixture of chloroform (1 vol.) and ether (3 vols.) may be used to extract ephedrine without risk of converting it into the hydrochloride. The seasonal variations are dependent on the times of rain- and snow-fall, both of which cause a decrease in the content of ephedrine and of total alkaloid. In general, both these values decrease from May until August and then rise to their maximum in October or November. The drug should be gathered immediately

before the onset of frost. When kept dry it retains its alkaloid content unaltered indefinitely.

H. E. F. NOTTON.

Influence of source and time of collection on Rhizoma filicis. L. Kofler and E. Müller (Arch. Pharm., 1930, 268, 644-652).—Contrary to general opinion, no regular differences in the content of extractive matter and of crude filicin, or in toxicity towards fish, can be detected between samples of this drug gathered at widely-differing altitudes, or between spring- and autumn-gathered samples. Among 22 autumn- and 6 spring-gathered samples the greatest variations in biological activity, using filmarone as standard, are less than 50% of the mean. An indication that the active principle of the drug gradually undergoes a change which cannot be detected by the usual chemical assays is given by the examination of 6 samples, 4-20 years old, which were indistinguishable in extractive and crude filicin content from the fresh drug, but were from 2 to 10 times less active biologically. Of the 22 autumn-gathered samples only 3 complied analytically with the requirements of the D.A.B. VI, which are regarded as too stringent. H. E. F. NOTTON.

Histo-chemical detection of cetraric acid in

Histo-chemical detection of cetraric acid in Lichen islandicus. M. Proner (Pharm. Zentr., 1931, 72, 227).—Cetraric acid is detected by the appearance of an intense brownish-red coloration in the central (gonidial) portion of the fibre when it is treated for 2—3 min. with 25% ammonia solution and then with 5% ferric chloride in 50% alcohol. H. E. F. Notton.

Micro-m.p. determinations with drugs. L. Kofler and W. Dernbach (Arch. Pharm., 1931, 269, 104—114).—The m.p. under the microscope, using the apparatus of Kofler and Hilbek (cf. A., 1931, 189), is recorded for each substance for which the m.p. is given in the D.A.B. VI. The values are, except for substances containing loosely combined water, in good agreement with those determined by the usual method. Most of the substances examined sublime in characteristic forms, the sublimation temperatures being all higher than those recorded by Kempf (A., 1923, ii, 216).

H. E.!F. NOTTON.

Colorimetric determination of strychnine in pharmacopæial preparations. A. François (Bull. Soc. Pharm. Bordeaux, 1930, 68, 158—163; Chem. Zentr., 1931, i, 823).—The procedure is based on comparison with a standard of the colour produced by boiling with zine amalgam and hydrochloric acid, cooling, and adding sodium nitrite solution. Suitable dilutions for pharmacopæial preparations of nux vomica are mentioned.

A. A. Eldridge.

[Quantitative] reaction for capsaicin. K. von Fodor (Z. Unters. Lebensm., 1931, 61, 94—100).— A clear extract (5 c.c. = 1 g. of sample) of the powdered sample in dry acetone is gently shaken with 9 drops of hydrochloric acid and 0·1 g. of ammonia vanadate, when a colour results varying from blue (for 0·08%) through green to green-brown (for 0·01% of capsaicin, the limit of concentration). The brown tint is derived from oxidation of carotene etc. from the sample. Addition of 20 drops of a 1% solution of vanadium oxychloride in carbon tetrachloride to 10 c.c. of an extract

in dry ether gives a more sensitive reaction; the reagent is stable. By means of a range of colours produced from known amounts of capsaicin the reaction is made quantitative. Capsaicin was absent from Spanish pepper, 0.01% or less being found in refined Hungarian paprikas. The blue compound was shown to be vanadylcapsaicin,  $C_{18}H_{26}NO_3,VOCl_2$ . J. Grant.

Determination of santonin in "Trochisci santonini." P. J. Claus (Pharm. Weekblad, 1931, 68, 414—424).—The sample is dried at 105° and extracted with ether for 4-5 hrs.; the residue after distillation of the ether is mixed with 0.5 g. of solid paraffin and refluxed with 50 g., and then with 15 g., of ethyl alcohol for 30 min., and the cooled solutions are filtered. The filtrate is shaken with about 15 g. of light petroleum (b.p. 40—70°), and the alcoholic layer, after separation, is heated to boiling to remove dissolved petroleum and is neutralised to phenolphthalein with 0.1N-alkali. The solution is then refluxed with 10 c.c. of 0.1N-alkali for 20 min. and, on cooling, the excess of alkali is titrated with 0.1N-hydrochloric acid. After compensation, from a blank determination, for alkali derived from the apparatus, the error does not exceed 2%. The method of the D.A.B. VI is criticised. H. F. Gillbe.

Distillation of essential oils. V. I. VARENTZEV (J. Chem. Ind., Moscow, 1929, 6, 1602—1604).—The extraction of essential oils from seeds by steam-distillation is expressed by S = a/(1-r) and  $l = ar^{n-1}$ , where a is the amount of oil extracted during the first hour and r is the ratio for two consecutive hours.

CHEMICAL ABSTRACTS.

Oleonaphtha as absorber of ethereal oils from distillation water. V. I. Varentzev (J. Chem. Ind., Russia, 1929, 6, 966—967).—The condensed water containing droplets of essential oils is passed through a layer of oleonaphtha, whereby a saving of 1.77% of the product is effected. With terpineol the increase in yield was 3.33%. Chemical Abstracts.

Essential oil of fennel. N. Sobyanin and S. Saakov (Masloboino Zhir. Delo, 1929, No. 6, 34—36).—The following variations were observed with oil cultivated in the Krasnodar district from various kinds of fennel: yield  $1 \cdot 21 - 5 \cdot 3\%$ ,  $\alpha + 5^{\circ}$  to  $+20 \cdot 3^{\circ}$ ,  $n \cdot 1 \cdot 528 - 1 \cdot 5405$ , f.p. 3—12°. Chemical Abstracts.

Rosenthaler's reaction for peppermint oil. I. M. Korenman (Pharm. Zentr., 1931, 72, 273—276).—The intensity of the colour formed by the addition of Rosenthaler's reagent (1% vanillin solution in concentrated hydrochloric acid) to peppermint oil is in inverse ratio to the content of menthol, which alone gives no colour. The colour is due to the presence of hydrocarbons. Both menthol and peppermint oil give colour reactions with a 1% vanillin solution in sulphuric acid (d1.8), which is probably due to menthene and dimenthene formed by the dehydrating action of the acid on menthol. Various colour reactions and their sensitivities are described.

E. H. Sharples.

Medicinal carbon.—See II. Calcium sulfuratum solutum.—See VII. Oil of *Sinapis dissecta*.—See XII. Caffeine in coffee etc.—See XIX. Alkyl *p*-hydroxybenzoates for sterilisation etc.—See XXIII.

See also A., May, 561, Colloidal silver and gold. 588, Detection of silver. Lead in tartar emetic. 612, Polycyclic aromatic hydrocarbons. 627, Fluoran derivatives. 629, Local anæsthetics. 636, Aconitine. Derivatives of p-arsanilic acid. 637, Water-soluble organomercury compounds. 639, Determinations of nicotine and pyramidone. Thalleioquinine reaction. Detection of alkaloids in plants. 651, Digitalis leaves. 656, Antipneumococcic and antimeningococcic horse sera. Diphtheria toxin. 658, Pure male hormone. 661, Analysis of tobacco smoke.

### PATENTS.

Manufacture of easily soluble salts of benzylmorphine [peronine]. Chem. Fabr. vorm. Sandoz (B.P. 343,397, 7.5.30. Ger., 24.5.29).—The lower alkylsulphonates (e.g., methane- and ethane-sulphonates) of benzylmorphine are crystalline salts, giving stable aqueous solutions suitable for injection. C. Hollins.

Preparation of [therapeutically valuable] silicyl compounds. H. P. Kaufmann (B.P. 343,165, 15.11.29. Ger., 16.11.28).—Hydroxycarboxylic acids, esters, or salts are treated with a silicon halide (tetrachloride), especially in presence of pyridine or other base; or the salts are used in a solvent (pyridine or ether); or the esters are heated with alkyl silicates. The silicyl derivatives of hydroxylated higher fatty acids (ricinoleic or dihydroxystearic acid) are soluble in fats and in ammonia. Silicyl derivatives of ethyl ricinoleate, ricinoleic acid (calcium salt and dibromide), dihydroxystearic acid, lactic acid, and methyl and amyl salicylates are described.

C. Hollins.

Manufacture of benziminazolonestibinic acids. I. G. FARBENIND. A.-G. (B.P. 343,072, 7.11.29. 7.11.28).—o-Phenylenediaminestibinic acids are phosgenated, or a diazotised aminobenziminazolone is treated with antimonious acid. Examples of starting materials are: 3-amino-4-methylaminobenzenestibinic acid (from 1-chloro-2: 4-dinitrobenzene condensed with methylamine, partly reduced with sodium sulphide, acetylated, reduced, diazotised, and treated with antimonious acid in glycerin and aqueous sodium hydroxide, and finally hydrolysed); o-phenylenediamine-4-stibinic acid (from diacetylated 4-nitro-o-phenylenediamine); 6-chloro-o-phenylenediamine-4-stibinic acid (from 6-chloro-1:2:4-trinitrobenzene); 5-aminobenziminazolone and its 1-methyl, 1-ethyl, and 7-chloroderivatives. C. Hollins.

# XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

See A., May, 541, Sensitivity of photographic plates. 579, Developed films. Ripening. Silver bromide. 592, Filters to reproduce sunlight and daylight. 593, Spectrophotometers.

#### PATENTS.

Manufacture of photographic surfaces for the bleaching-out process. W. W. Groves. From I. G. Farbenind. A.-G. (B.P. 343,212, 2.12.29).—Basic nitrogen-free oxonium dyes other than anthocyanins are used in place of flavindulines for yellows in the bleaching-out process. Examples are 3:7-dihydroxy-

10-methylxanthoxonium chloride, 3:7-dimethoxy-10-phenylxanthoxonium bromide. The latter is used, e.g., with 5-phenylamino-9-diethylamino-2:1-naphthaphenoxazine chloride and 3-methylamino-7-dimethylamino-5-phenyl-2:8-dimethylphenazine chloride, the coloured print being fixed with a 3:1 mixture of benzine and anisole.

C. Hollins.

Photomechanical printing surfaces. H. Wade. From A. R. Golrick (B.P. 346,672, 14.1.30).

[Half-tones on] paper etc.—See V. Cellulose acetate films. Compositions containing cellulose derivatives etc.—See XIII.

## XXII.—EXPLOSIVES; MATCHES.

See A., May, 574, Colloidal powders. 619, Detonation of benzoyl peroxide.

#### PATENT.

Fireworks or pyrotechnic devices. J. Pain & Sons, Ltd., and A. W. Milholland (B.P. 347,003, 25.4.30).

# XXIII.—SANITATION; WATER PURIFICATION.

Alkyl p-hydroxybenzoates for preservation, sterilisation, and disinfection. T. Sabalitschka (Arch. Pharm., 1930, 268, 653-673).—The phenol coefficients of the esters towards Staphylococcus aureus increase with the size of the alkyl group from 2.6 for the methyl ester to 83 for the benzyl ester. All have a very low toxicity to animals. The organisms which are invariably present in distilled water and pharmaceutical solutions of inorganic substances and alkaloids when these are preserved under ordinary conditions are killed within a few days by the addition of 0.15-0.2% of the methyl ester, or of 0.1% of a 1:1 mixture of the methyl and propyl esters. The propyl ester and its sodium salt kill S. aureus, B. coli, and B. paratyphosus in 1 day at concentrations of 0.06% and 0.07%, respectively, and are applicable to the sterilisation of injection and ophthalmic solutions. Powders are also sterilised when the ester is thoroughly mixed with them by gentle heating ("Tyndallisation") and the products act as powerful antiseptics when brought into contact with bacterial cultures or wound surfaces.

# H. E. F. NOTTON.

# Brewing water.—See XVIII.

See also A., May, 589, Determination of mercury in air. 622, Antiseptic derivatives of indan-1:3-dione. 643, Determination of alkali metals in seawater. 656, *B. coli* in water. 657, Sodium hydroxide, carbonate, and phosphate as germicides.

PATENTS.

Drying of sewage sludge. W. W. Harrit, Assr. to L. L. Keene (U.S.P. 1,779,810, 28.10.30. Appl., 14.11.27).—Partly-dried sludge, carried by an endless belt conveyor or on trays stacked on trucks, is passed through a heat-insulated tunnel which is traversed in the opposite direction by a current of hot air. The waste products are discharged through a stack and the dry sludge is crushed and delivered into a storage bin by compressed air for use as fertiliser. C. Jepson.

Filter bed.—See I: Gas from garbage.—See II.