

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

DEC. 20, 1929.



I.—GENERAL; PLANT; MACHINERY.

Dissociation of water in steel tubes at high temperatures and pressures. C. H. FELLOWS (J. Amer. Water Works' Assoc., 1929, 21, 1373—1387).—In high-pressure boilers, particularly those in which distilled water is used and which are otherwise unprotected against corrosion, the corrosive action of pure water is of primary importance. Small-scale experiments having indicated that superheated steam contains more free hydrogen than saturated steam, a stream of water vapour was passed under varying conditions of temperature and pressure through tubes of either low-carbon steel (0.10% C) or chrome steel (16.35% Cr) and the amount of hydrogen determined before and after. It was found that a low-carbon steel was more liable to cause dissociation than chrome steel, that the rate of decomposition increases with rise of temperature and decreases as the thickness of oxide film formed increases, whilst within the limits used variation in steam pressure was without effect. The velocity of the steam through the tube was very considerably lower in the experiment than in actual practice, and since later investigations indicate that at still higher velocities no hydrogen whatever can be detected, it would seem that there is a definite relation between steam velocities (which are an important factor in determining the temperature of the metal) and oxidation of the latter due to the dissociation of steam.

C. JEPSON.

Determining thermal conductivity of heat-insulating materials. H. STILES (Chem. Met. Eng., 1929, 36, 625—626).—For the rapid determination of the thermal conductivity of such materials as wall-boards, two similar lagged brass cylindrical vessels are adapted to fit one above the other with the wall-board between the bottom of the upper and the cover of the lower, which serves as a boiler. Inside the upper vessel, which is an ice container, a small bakelite cylinder is soldered through the medium of a brass ring to the base; this cylinder has a tightly-fitting cover through which a glass tube with two bulbs and index marks is fitted. The ice container and central cylinder are filled with ice (weighted) and water, and when steady conditions have been attained, as indicated by the level in the glass tube, the vessel is put on the boiler. As the ice in the bakelite cylinder melts, the time taken by the water-level in falling between the index marks is observed. Repetition experiments may be conducted quickly by refilling the bakelite cylinder to above the upper index mark and continuing observations on the contraction.

C. A. KING.

Sampling of gas over mercury at a constant

rate. S. PEXTON and W. K. HUTCHISON (J.S.C.I., 1929, 48, 242—244 T).—A gas sampling device is described which is portable and self-contained, with mercury as the confining liquid. It will sample continuously for long periods at a rate which is constant to within 1%. The theory is described, and a full mathematical analysis is given.

Gas analysis. E. OTT (Gas- u. Wasserfach, 1929, 72, 862—863).—The author's portable apparatus has been improved, particularly in the means for determination of carbon monoxide, which is converted into carbon dioxide by the action of iodine pentoxide. For measuring the inert constituents of a gas mixture, the combustible material is eliminated in passing over copper oxide heated in a small electric furnace.

R. H. GRIFFITH.

Determinations of inert gas content of gas mixtures by means of calcium as an absorbent. M. LEATHERMAN and E. P. BARTLETT (Ind. Eng. Chem. [Anal.], 1929, 1, 223—225).—An apparatus is described which permits the analysis of various gas mixtures for the noble gases by a method involving absorption of all common gases by hot metallic calcium and the measurement of change of pressure at constant volume. Samples of air, nitrogen, and hydrogen have been analysed. Results are reproducible to within 0.002—0.003%. Sources of error and possible improvements in the apparatus are discussed.

H. S. GARLICK.

Liquorice root. HOUSEMAN and LACEY.—See XX,

PATENTS.

Furnaces. G. and D. M. NAISMITH (U.S.P. [A] 1,725,879, 27.8.29. Appl., 24.12.27, and (B) 1,727,898, 10.9.29. Appl., 12.12.27).—The furnaces are suitable for reheating ingots etc., that described in (A) being recuperative, and (B) regenerative. In (A) a horizontal mixing and combustion chamber comes between the recuperators and goods chamber; in the latter the hot gases pass downwards, but in the recuperators the exhaust gases and air pass to and fro horizontally, gases downwards, air upwards. In (B) the air-heating chambers are above the gas-heating chambers, and the air and gas mix before they enter the goods chamber and pass horizontally among the goods.

B. M. VENABLES.

Furnace for sintering, burning, and roasting of substances. E. C. LOESCHE (B.P. 298,651, 13.10.28. Ger., 14.10.27).—The material is fed upwards through the centre of a hearth, and is heated by a downward flame or electric arc immediately above the same point; the feed opening is therefore always covered with material, which may be allowed to form a cone from the

base of which the new material is pushed upwards to the apex and, after contact with the heat, rolls down the sides.

B. M. VENABLES.

[Reverberatory] furnaces. H. S. KNOWLES and W. BURKINSHAW (B.P. 318,748, 12.9.28).—The combustion chamber is divided into a number of compartments by transverse walls extending to the roof, but provided with ports for the burning gases. In front of each row of ports is a bridge wall so that the gases are deflected upwards on entering each compartment; at these points additional air is blown in vertically from below.

B. M. VENABLES.

Rotary [annular] furnaces. TROCKNUNGS-, VER-SCHWELUNGS-, U. VERGASUNGS-GES.M.B.H. (B.P. 301,904, 26.11.28. Ger., 9.12.27).—A form of furnace is described in which several annular hearths for the material, heated by fluid in the hollow hearths, are supported with a certain degree of flexibility above a common annular carriage. The furnace is suitable for the continuous heat-treatment of pulverulent material in very thin layers (of the order of 1 mm.), one use being the low-temperature distillation of coal.

B. M. VENABLES.

Boilers and like apparatus for evaporation or heating of liquids. A. LAMB (B.P. 320,482, 6.9.28).—The hot gases from a fire or waste-heat flue pass downwards through a bank of horizontal, superposed tubes through which the liquid rises in zig-zag fashion by thermo-siphon action, aided in the case of evaporation by the vapour bubbles. When an integral combustion chamber is provided, the upper pair of tubes only may extend through the inlet port at the top of the main heating chamber into the upper part of the combustion chamber.

B. M. VENABLES.

Steam accumulators. J. D. TROUP (B.P. 319,108, 31.7.28).—A storage vessel is provided with an external circulation pipe in which is inserted an injector device operated by the incoming steam.

B. M. VENABLES.

Apparatus for effecting contact between gases and divided solids. E. J. BRADY, Assr. to UNITED GAS IMPROVEMENT CO. (U.S.P. 1,731,223, 8.10.29. Appl., 21.7.28).—The comminuted solid is allowed to fall between two confronting, perforated walls having relative movement, and the gas is caused to pass across the stream several times in a zig-zag manner generally countercurrent to the solid, *i.e.*, upwards.

B. M. VENABLES.

Apparatus for heating by circulation of hot fluids. J. A. REAVELL (B.P. 318,652, 8.5.28).—A heat-transmitting fluid is passed in thin streams over electric heating elements provided with ribs or projections to ensure that the flow of liquid is turbulent.

B. M. VENABLES.

Thermostats. A. B. and T. F. C. POTTERTON, and T. POTTERTON, LTD. (B.P. 320,270, 22.11.28).—The multiplying lever of a known type of temperature-sensitive device operates a bell-crank lever or other device attached to a gas valve by means of a pin and slot, so that the gas is turned suddenly off or on; the thermostat is therefore suitable to control Bunsen burners.

B. M. VENABLES.

Thermometer, pyrometers, and the like [for hot-air furnace gases etc.]. W. GILBERT (B.P.

320,194, 30.8.28 and 12.1.29).—To prevent errors due to radiation to or from the walls of the conduit conveying the gases, the thermometer bulb or hot-junction is enclosed in a number of spaced concentric tubes or plates, which come up to the temperature of the gases.

B. M. VENABLES.

Grinding and crushing. E. BARTHELMESS (B.P. 291,460, 2.6.28. Ger., 4.6.27).—The material is charged into a helical passage and propelled along it by a gaseous or liquid stream blown in with sufficient velocity to produce disintegration of the material merely by friction and centrifugal impact. Forms of construction are described in which the helical passages are built of a number of superposed similar units. (Cf. B.P. 251,665; B., 1926, 775.)

B. M. VENABLES.

Grinding mills. FULLER LEHIGH Co., Assecs. of R. M. HARDGROVE (B.P. 311,688, 7.7.28. U.S., 14.5.28).—The mill is similar in principle to that of a cup-and-cone ball-bearing, the balls not being positively driven. The angle between the faces of the upper ring (cup) and lower table (cone) is between 20° and 70°, and both surfaces slope outwardly downwards. The ring is usually stationary and the table rotated, relative adjustment is provided, and, to avoid grooving, balls of different sizes may be used simultaneously.

B. M. VENABLES.

Construction of grinding mills. A. E. G. MACCALLUM (B.P. 318,873, 11.5.28).—A form of construction of roller and block mill (as used for paints) is described, ensuring accurate alinement of roller and block. The base and bearing supports are in one piece, and the bearing for the rollers can be machined while practically the whole machine is assembled.

B. M. VENABLES.

Separation of mixed materials. T. FRASER (B.P. 299,777, 24.10.28. U.S., 31.10.27).—In a process for the separation of mixed dry materials by floating or sinking in a fluid mass of dry particles, kept fluid by upward currents of air, the fluid mass is subjected also to segregating or transitional movements, which increase the separating effect so that lighter and cheaper material such as sand may be used instead of expensive heavier material such as magnetite for the fluid mass. The segregating movements may be horizontal or vertical and unidirectional or pulsating. Suitable apparatus is described.

B. M. VENABLES.

Apparatus for [air] separation of solids. W. A. DUERR, Assr. to CABOT Co. (U.S.P. 1,728,046, 10.9.29. Appl., 21.7.27).—To one end of a worm-conveyer a blast of air is supplied, at the other end is a feed hopper for the material, and intermediately (near the hopper) is an air-separating device.

B. M. VENABLES.

Centrifugal separator. ATELIERS T. ROBATEL J. BUFFAUD & C^{ie}. (B.P. 306,124, 15.1.29. Fr., 16.2.28).—A centrifuge with horizontal shaft and adapted either to filtration or decantation is described. The casing is easily renewable independently of the supporting framework, and the discharging scraper can be easily withdrawn and when in operation will cease travelling should the resistance become too great.

B. M. VENABLES.

Centrifugal separators. SOC. GÉN. D'ÉVAPORATION PROC. PRACHE & BOUILLON (B.P. 313,097, 6.6.29. Fr.,

6.6.28).—A centrifuge for the separation of solid matters in suspension in a liquid is described, having continuously acting, gear-driven scrapers for cleaning the outlets for solids.

B. M. VENABLES.

Centrifugal cleaning of liquids. AKTIEBOLAGET SEPARATOR (B.P. 307,933, 15.3.29. Swed., 17.3.28).—To a centrifugal separator having continuous discharge of separated solid matter is added a pulp rich in solid matter with the object of preventing too great an outflow of liquid through the discharge ports for solids, while permitting the ports to be of reasonable size. The additional or circulating solid matter may be of the same nature as the impurities to be separated, the quantity may be regulated less or more according as there is more or less solid in the new feed, and it is preferably supplied to points near the discharge openings.

B. M. VENABLES.

Centrifugal machines, especially for purification or separation of liquids. KOEFOED, HAUBERG, MARSTRAND, & HELWEG A./S. TITAN, and S. HAGERUP (B.P. 320,300, 26.1.29).—A form of construction of a centrifuge having a horizontally divided bowl directly above an electric motor is described.

B. M. VENABLES.

[Pressure] filtering apparatus. A. R. JAHN (B.P. 318,687, 18.6.28).—The filter comprises a fixed head carrying a number of filter units which are enclosed in a detachable pressure-tight cover forming the reservoir for prefilter. The filter walls of each leaf or unit are kept apart by a corrugated or ribbed plate which prevents choking of the passages for filtrate. Brushes and/or compressed air in reverse flow may be used to remove deposited solids.

B. M. VENABLES.

Apparatus for separating impurities from liquid stored in tanks and the like. LIQUID MEASUREMENTS, LTD., and F. HAMMOND (B.P. 320,239, 15.10. and 7.12.28).—Some of the liquid is withdrawn from the point where the impurities have accumulated (usually the bottom of the tank) by means of a pump, and is delivered to a separating vessel, where the impurities are retained by settling and/or filtration; the excess clean liquid runs back into the tank. The whole apparatus may be self-contained and made airtight with the tank (and is then suitable for petrol), and the separating vessel may have a glass wall.

B. M. VENABLES.

Breaking a foam into liquid particles and vapours and gases. J. F. HARKOM (B.P. 320,191, 27.8.28).—A centrifugal rotor is situated in the vessel where the foam is produced, and the gases and vapours are exhausted through the rotor and its hollow shaft against centrifugal force, the liquid being flung off the rotor.

B. M. VENABLES.

Gas scrubber. J. P. FLIPPEN (U.S.P. 1,732,086, 15.10.29. Appl., 6.1.26).—A gas scrubber is divided into two compartments and a different liquid used in each. The scrubbing surfaces are provided by co-operating stationary and rotary grids.

B. M. VENABLES.

Removal of dust from gases. INTERNAT. COMBUSTION, LTD., and P. W. MCGUIRE (B.P. 320,068, 3.4.28).—In the process of removing dust from hot products of combustion by scrubbing with water, there is injected

first into the gases water in such quantities that it entirely evaporates and leaves the gases substantially saturated. The gases are then scrubbed by a counter-current rain of water.

B. M. VENABLES.

Separation and recovery of gases and vapours by solid absorbents. A. GODEL, and SOC. DE RECHERCHES ET D'EXPLOIT. PÉTROLIFÈRES (B.P. 317,629, 3.8.28. Addn. to B.P. 267,369; B., 1927, 689).—In the apparatus described in the prior patent, the cooling and drying of the absorbent after regeneration is effected by a gas following a closed circuit, cooled at one point and heated at another, thereby preventing loss of adsorbed solvent. (Cf. also B.P. 295,504; B., 1928, 773.)

B. M. VENABLES.

Physical or chemical treatment of gases with solid materials. J. S. MORGAN, and M. NEWBOULD & PARTNERS, LTD. (B.P. 320,450, 9.8.28).—In heterogeneous physical or chemical reactions between a gas and a finely-divided solid, the temperature is controlled by the addition of solid materials of high heat conductivity or capacity which may be inert to the reaction. *E.g.*, in the removal of a constituent of a gas by absorbent carbon, the reaction being exothermic, small metallic balls are mixed with the absorbent, and, after the reaction, are separated from the carbon; the carbon is re-activated and the balls are cooled for re-use.

B. M. VENABLES.

Utilising the heat of gases. C. G. HAWLEY, ASSR. to CENTRIFIX CORP. (U.S.P. 1,726,828, 3.9.29. Appl., 11.3.26).—In an apparatus suitable for the heating of liquids by contact with hot gases, the gas enters a casing in a whirling manner outwardly from a central, rising inlet conduit, and leaves through the top of the casing where are also situated water-sprays. In addition to a sludge outlet at the bottom, there is a circumferential port at the top through which liquid and dust are thrown out by the whirling motion.

B. M. VENABLES.

Viscosimeter. C. D. MILLER (U.S.P. 1,727,836, 10.9.29. Appl., 12.6.22).—The liquid is admitted between two surfaces which are closely adjacent over a large area and have relative motion. The flow of the liquid is checked at some point remote from the inlet and is allowed to pass out at some intermediate point. The pressure of the liquid at the check is a measure of the viscosity.

B. M. VENABLES.

Pulverising mill. E. C. LOESCHE (U.S.P. 1,733,417, 29.10.29. Appl., 10.2.26. Ger., 12.2.25).—See B.P. 247,562; B., 1926, 695.

Heat exchanger. J. P. RATHBUN, ASSR. to WESTINGHOUSE ELECTRIC & MANUF. Co. (Re-issue 17,480, 5.11.29, of U.S.P. 1,708,031, 9.4.29).—See B., 1929, 457.

Extraction of substances from fluids. A. H. PEHRSON (U.S.P. 1,732,819, 22.10.29. Appl., 17.11.27. Swed., 7.10.26).—See B.P. 278,722; B., 1929, 192.

Apparatus for detection and determination of impurities and dissolved matter in water and other liquids. C. E. PERRY, ASSR. to EVERSLED & VIGNOLES, LTD. (U.S.P. 1,734,342, 5.11.29. Appl., 8.4.27. U.K., 18.6.26).—See B.P. 268,597; B., 1927, 400.

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Mixing of gases and liquids. E. PLAYER, Assr. to PARC ENGINEERING Co., LTD. (U.S.P. 1,733,101, 22.10.29. Appl., 21.6.27. U.K., 23.12.26).—See B.P. 286,812; B., 1928, 352.

Refrigerating machines. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of C. L. HEISLER (B.P. 301,833, 5.12.28. U.S., 6.12.27).

Absorption refrigerating apparatus containing an inert gas. ELECTROLUX, LTD., Assees. of PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAG (B.P. 298,575, 11.10.28. Swed., 11.10.27).

[Device for producing gyratory movement of flue gases in] furnaces. G. HIRSCH (B.P. 320,768, 19.10.28).

Pressure gauge for high vacua. MASCHINENFABR. OERLIKON (B.P. 301,454, 26.10.28. Switz., 30.11.27).

Energy from water etc. (B.P. 319,604).—See VII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Transformation of the fatty acids during geological periods. III. G. STADNIKOV and A. WEIZMANN (Brennstoff-Chem., 1929, 10, 401—403; cf. B., 1929, 158).—Further examination of the Suchokujaski boghead coal shows that the residue after extraction with organic solvents and with alcoholic potash consists of the polymerisation products of unsaturated fatty acids. These products possess carboxyl groups and can form salts and esters. A comparison of coorongite with the Siberian boghead coals shows these to have been produced from similar parent substances, viz., the fats of the algæ, but in the coorongite, of which a high proportion is soluble in organic solvents, the polymerisation processes are not so far advanced as in the boghead coals. A. B. MANNING.

Examination of coal in small and large gas-works. W. BERTELSMANN (Gas- u. Wasserfach, 1929, 72, 965—968).—The equipment of a gas-works laboratory is discussed, and a description is given of the laboratory under construction at the Berlin Gas Works. Provision is there made for the proximate analysis of 80—100 samples of coal per day. Any sample showing a low volatile matter content can be further examined in a high-temperature assay apparatus consisting of a wrought-iron retort holding a charge of 700 g. of coal; this can be heated in an electrical furnace, the usual carbonising temperature being 1100°. If desired, a full-scale carbonisation test can be carried out in an experimental retort setting attached to the laboratory. A. B. MANNING.

Determination of the oxidisability of bituminous coals by dilute potassium permanganate solution. D. J. W. KREULEN (Brennstoff-Chem., 1929, 10, 397—400).—Samples weighing 0.0025, 0.005, and 0.0075 g. of the finely-ground coal were heated with 20 c.c. of 0.01*N*-potassium permanganate at the b.p. for 10 min., 20 c.c. of 0.01*N*-oxalic acid were then added, and the excess was titrated back with permanganate. The values so obtained for the oxidisability of coal, expressed as c.c. of oxygen taken up per g. of ash-free, dry coal, were extrapolated back to 0.0 g. in order to eliminate variations due to differences in the final concentration of

the permanganate solution. The final values vary from 66 for an anthracite to 886 for a coal of high volatile matter content (38.7%). For a number of coals a rough proportionality exists between the oxygen taken up and the volatile matter content, but there are coals of the same volatile matter content which yet differ widely in oxidisability. The results give a measure of the tendency of the coals to oxidise on storage, although for this determination a method based on oxidation by air is preferable. Durain and vitrain possess approximately the same oxidisability, 990 and 969 respectively; fusain possesses a very much lower value, about 80, which is of the same order as that of wood charcoal. Pre-oxidation of coal in the air at 100—170° has little effect on its oxidisability by potassium permanganate. A. B. MANNING.

Comparison of methods for testing the caking properties of coal. L. SLATER (Dept. Sci. Ind. Res., Fuel Res. Brd. Rept., 1928—9, 110—119).—The caking indices of 20 British coking coals determined by the methods due to Gray and Campredon, Meurice, Barash, and Burdekin, respectively, exhibited no close agreement in the order of the values given by each method, nor was it possible to correlate these results with the shatter indices of the cokes prepared from the same coals in full-scale by-product coke ovens. None of the laboratory methods examined therefore appears capable of assessing the value of a wide range of coals in their application to the production of by-product cokes. Within the limits of the range of carbon content examined, the shatter index increases fairly regularly with increasing carbon content, whilst the caking value rises sharply to a maximum with coals of approx. 86% C and then falls rapidly. A knowledge of the caking index is of value for coals which are to be modified by blending prior to carbonisation. A. B. MANNING.

Separation of the constituents of coke-oven gas by washing under pressure. F. FISCHER, K. PETERS, and W. TER-NEDDEN (Brennstoff-Chem., 1929, 10, 348—349).—Coke-oven gas has been treated with various solvents under a pressure of 20 atm., and the composition of the dissolved and residual gases has been determined. Using petroleum as solvent, the methane content of the gas could be raised from about 23% to 48% (in the dissolved gas), the hydrogen content at the same time falling from about 32% to 17%. Water, a saturated solution of ferrous sulphate, or a soap solution acts as preferential solvent for carbon dioxide. It is sometimes of advantage to use two non-miscible solvents simultaneously; thus, by using water together with petroleum, the gas dissolved in the latter has its methane content raised, and at the same time its carbon dioxide content lowered. A. B. MANNING.

Determination of the sulphur content of gases from boiler furnaces. E. TAYLOR and H. F. JOHNSTONE (Ind. Eng. Chem. [Anal.], 1929, 1, 197—199).—Apparatus and methods are described for sampling the gases after they have passed the boiler tubes and from the furnace. These are designed to reduce the possibility of catalytic action between the points of sampling and analysis. At the lower furnace temperatures an iron pipe with glass lining is used, and at furnace tempera-

tures a copper, glass-lined, water-cooled sampler. A suction pump draws the gases at a constant rate of 0.1 cub. ft. per min. through the apparatus which absorbs the sulphur gases, after which they pass through a flowmeter. The absorption apparatus for the determination of sulphur dioxide and trioxide consists of one bottle with a fine-grained dry alundum thimble which retains the droplets of sulphuric acid, and a second with a coarser alundum thimble immersed in a sodium hydroxide solution containing hydrogen peroxide, which absorbs the sulphur dioxide. Water is run through the glass tubing into the first thimble and bottle, and the sulphur trioxide is determined by titrating with sodium hydroxide. The second bottle is titrated with standard acid to determine the sulphur dioxide. Results checked against known mixtures show a high degree of accuracy. When only the total sulphur is required, the first bottle is omitted. If the gases contain soot and tar, which would clog the alundum thimble, the impinger principle is used, there being two impinger bottles in series. The composition of the gases is also determined by an Orsat apparatus, and from these data and the coal analysis, the ratio of sulphur in the gases to that in the coal can be computed. H. S. GARLICK.

Deposition of carbon in reaction between carbon dioxide and hydrogen. M. RANDALL and W. H. SHIFFLER (Ind. Eng. Chem., 1929, 21, 941).—A contribution to the discussion of Randall and Gerard (B., 1929, 82) in which the equation $\text{CO}_2 + 2\text{H}_2 = \text{C} + 2\text{H}_2\text{O}$ is offered as a possible explanation of the experimental results obtained by these investigators. H. S. GARLICK.

Carbon deposits from lubricating oils. Experiments with heavy-duty engines. C. J. LIVINGSTONE and W. A. GRUSE (Ind. Eng. Chem., 1929, 21, 904—908; cf. B., 1926, 571).—Three very heavy lubricating oils of different characteristics have been used in experiments made with 18 motor coaches having sleeve-valve engines. These coaches travelled 180,000 miles during the tests. The same oils were tested in a single-cylinder laboratory engine with poppet valves. It was found in the road tests that whereas on the average the use of a naphthenic oil made cleaning of the ports of the engines necessary once in about 9300 miles, the corresponding mileages for low- and high-carbon paraffinic oils, respectively, were 2400 and 1900. Although the carbon-residue test gives a fairly reliable index of carbon deposition in a poppet-valve engine, some other test seems desirable for predicting the behaviour of a lubricating oil in a sleeve-valve engine. A distillation test which appears to place the oils employed in the order of their suitability for this type of engine is described. H. INGLESON.

Synthetic benzene from water-gas. H. TROPSCH and H. KOCH (Brennstoff-Chem., 1929, 10, 337—346; cf. B., 1928, 324).—The benzene fraction of the synthetic petroleum obtained by the catalytic reduction of carbon monoxide under ordinary pressures consisted of a mixture of olefines (65%) and paraffins, containing only small amounts of aromatic hydrocarbons (benzene 0.1%, toluene 0.4—0.5%), and no naphthenes. The benzene was fractionated and the olefines and paraffins in

suitably-chosen fractions were separated by means of a modified mercuric acetate method. The following hydrocarbons were isolated and identified: Δ^{α} - and Δ^{β} -pentenes, Δ^{α} - and Δ^{β} -hexenes, $\gamma\gamma$ -dimethyl- Δ^{α} -pentene, heptene, (?) Δ^{β} -octene, nonene, *n*-pentane, *n*-hexane, *n*-heptane, *n*-octane, γ -methyloctane, *n*-nonane. A small quantity of a solid crystalline substance (m.p. 106°) of unknown constitution, but possessing some of the properties of a γ -pyrone derivative, was isolated from the fractions of b.p. 100—120°. A. B. MANNING.

Determination of total moisture in carbon blacks. C. M. CARSON (Ind. Eng. Chem. [Anal.], 1929, 1, 225).—A 5-g. sample of carbon black is placed in a 500-c.c. round-bottom flask with 25—35 c.c. of dry xylene and 200 c.c. of dry mineral oil. A short air-condenser leads to the bottom of a 25-c.c. distilling flask which, in turn, is connected to two or more calcium chloride tubes. The flask containing the sample is heated to 150—170° in an oil-bath, and a stream of dry nitrogen passed through the apparatus. The water and xylene distil into the small distilling flask and thence by warming in a water-bath into the calcium chloride tubes. The amount of water thus determined is considerably higher than the loss by heating in an oven at 105° for 5 hrs. H. S. GARLICK.

Low-temperature tar from bituminous coal and its utilisation in the artificial resin, the varnish, and disinfectant industries. K. EHRMANN (Brennstoff-Chem., 1929, 10, 405—406).—The commercial utilisation of some of the constituents of the low-temperature tar produced in the K.S.G. plant at Karnap is briefly described. The refined light fractions, preferably mixed with an equal volume of benzol, are used as motor spirit. The tar acids, after removal of the sulphur compounds, are fractionated; the fraction of b.p. 180—230° is used in the artificial resin and the varnish industries; the fraction of b.p. 230—280° possesses marked bactericidal properties and is used, in the form of an emulsion, as a disinfectant; the fraction of b.p. 280—360° finds use as a fungicide. A. B. MANNING.

Effect on concrete of acid water from stored bituminous coal. E. F. WOLF (Ind. Eng. Chem., 1929, 21, 908—910).—The effects of the acid solution formed by the percolation of rain through stored bituminous coal on concrete containing walls are not serious, since the initial rapid reaction which occurs produces an insoluble protective layer which causes further attack to be very slow. H. INGLESON.

Ceramic coatings—an outcome of corrosion difficulties in oil cracking. J. C. MORRELL and W. F. FARAGHER (Chem. Met. Eng., 1929, 36, 596—599).—Those metals and alloys which show the greatest resistance to the destructive corrosion of derived hydrogen sulphide in oil-cracking systems are either inherently unsuitable or too expensive for building the larger elements of a cracking plant. A mixture which has proved suitable for linings consists of furnace cement 60 lb., white silica foundry-sand (90% to pass 50-mesh) 30 lb., short-fibre asbestos 1½ lb., sodium silicate solution (d 1.38—1.42) 1 gal., water 1½ pints. This mixture is sprayed on to the cleaned vessel to a depth of $\frac{1}{8}$ — $\frac{3}{8}$ in., and is then brushed over with a mixture of furnace

cement 6 lb., and sodium silicate solution 1 gal., made to suitable consistency with water. When dried and carefully heated such a lining has proved resistant to corrosion and is of sufficient mechanical strength and tenacity.

C. A. KING.

Compression of refinery and casinghead gases. W. J. MURRAY (Ind. Eng. Chem., 1929, 21, 917—919).—A chart and series of equations are given for calculating the composition and quantity of both the liquid and gaseous phases produced in the compression of refinery and casinghead gases.

H. S. GARLICK.

Stabilising Grozni casinghead gasoline. A. N. SACHANOV and A. I. DOLADUGIN (Neft. Choz., 1928, 15, 464—471).—Suitable apparatus is described.

CHEMICAL ABSTRACTS.

Adsorption of gasoline and of benzene vapour by acidic clay. H. ISOBE, Y. ENDO, and I. KUNISE (Bull. Inst. Phys. Chem. Res. Tokyo, 1929, 8, 805—816).—The adsorption coefficients of acid clays from various Japanese localities have been determined for gasoline and for benzene vapour.

H. F. GILLBE.

Specific heats of petroleum vapours. W. H. BAHLKE and W. B. KAY (Ind. Eng. Chem., 1929, 21, 942—945).—The sp. heats of five petroleum distillates from mid-continent crude having d 0.9—0.68 have been determined at substantially atmospheric pressure over temperatures ranging from just above that of complete vaporisation to 350°. The constant-flow method was used in which the superheated vapours of a given distillate are passed at a constant rate through a calorimeter containing a heater to which a known amount of electrical energy was imparted. An equation was found to fit the experimental data with an average deviation of 1.3%.

H. S. GARLICK.

Action of sulphuric acid on mercaptans. S. F. BIRCH and W. S. NORRIS (Ind. Eng. Chem., 1929, 21, 1087—1090).—During the refining of certain light petroleum distillates by sulphuric acid a small amount of a substance soluble in petroleum, insoluble in concentrated sulphuric acid, and corrosive to copper at the b.p. of naphtha is formed. This is shown to occur only when mercaptans are present in the initial petroleum and to be due to the formation of a polysulphide (probably a trisulphide). Diethyl trisulphide, b.p. 78°/5 mm. (cf. A., 1908, i, 308), possesses all the properties of the corrosive substance. The formation of traces of corrosive polysulphides by the action of sulphur on lead mercaptides in naphtha solution is also described. *Di-n-heptyl disulphide*, b.p. 164°/6 mm., d_{15}^{25} 0.9082, has no corrosive action on copper. *Di-n-butyl disulphide*, b.p. 88°/4 mm., from *n*-butyl mercaptan could not be converted into the disulphoxide by concentrated sulphuric acid in light petroleum solution. D. W. HILL.

Determination of mercaptans in naphtha. P. BORGSTROM and E. E. REID (Ind. Eng. Chem. [Anal.], 1929, 1, 186—187).—By thoroughly shaking the sample of naphtha with excess of 0.5*N*-silver nitrate, adding excess of 0.03—0.05*N*-thiocyanate, and back-titrating with the silver nitrate solution in the presence of iron alum as indicator, results can be obtained with an average absolute error of 0.002%. Alternatively, the mer-

captans can be removed by shaking with silver nitrate, collecting the precipitated silver mercaptides on a filter, and determining the residual sulphur by the lamp method. Shaking the mercaptan solutions with mercury, as in the method for the removal of elementary sulphur, does not remove any of the mercaptans studied, neither does acidified cadmium chloride, which is recommended for the removal of hydrogen sulphide. Glacial acetic acid and zinc used for the reduction of disulphides cause a partial decomposition of the mercaptans; hence the titration method cannot be used. The percentage of sulphur should be determined by the lamp method, applied before the reduction and after the removal of the resulting mercaptans and other substances formed from the disulphides.

H. S. GARLICK.

Sulphuric acid test of motor benzol. E. KAUDELA (Brennstoff-Chem., 1929, 10, 404—405).—The treatment of motor benzol with concentrated sulphuric acid, which is necessary if the benzol is to satisfy the acid test, destroys some of the valuable constituents. To obtain a satisfactory refined benzol only those fractions which would otherwise form gummy deposits in the cylinder or on the valves etc. of the engine need be acid-washed. It is suggested that the acid test be replaced by an evaporation test, a suitable form of which is being developed.

A. B. MANNING.

Comparison of Russian and American petrolatums. A. S. VELIKOVSKI and S. S. NIFONTOVA (Neft. Choz., 1928, 15, 477—489).—The following values for American, Grozni, and Baku petrolatums, respectively, are recorded: d^{60} 0.820—0.875, 0.880, 0.869; viscosity (E^{60}) 10.34—14.68, 3.94, 10.3; colour (Duboscq) 18—157 mm., 21 mm., 9 mm.; acidity (% SO_3) 0.0008—0.006, 0.020, 0.051; ash 0.0002—0.0162, 0.371, 0.130; asphaltene none, none, 0.34%; iodine value 9—15, 8.12, 3.1; oxygen value 0.73—4.30, 2.67, 4.45; paraffin wax 32.88—61.5, 19.01, 19.52%. The paraffin content of Grozni petrolatum can be increased by dissolving in naphtha (initial b.p. 150°), cooling to —10°, decanting the upper layer, and distilling the solvent from the lower layer. CHEMICAL ABSTRACTS.

Cracking mazout containing paraffin wax under high hydrogen pressures. V. IPATIEV, N. ORLOV, and M. BIELOPOLSKI (Brennstoff-Chem., 1929, 10, 346—347).—Heating a Grozni mazout with hydrogen under 55—265 atm. and at 440—460° for periods of 1.1—2.5 hrs. yielded 25—34% of benzine (boiling to 150°) and 29—35% of kerosene (b.p. range 150—300°). The benzine was clear and colourless, and unaffected by fuming sulphuric acid. The kerosene darkened on keeping, but could be refined by treatment with concentrated sulphuric acid or powdered silica gel. The yields were unaffected by the addition of such catalysts as iron or copper oxide. In only one experiment was any coke formation observed.

A. B. MANNING.

Composition of paraffin wax. S. W. FERRIS, H. C. COWLES, JUN., and L. M. HENDERSON (Ind. Eng. Chem., 1929, 21, 1090—1092).—Previous work on paraffin wax indicates the presence of straight-chain hydrocarbons only (cf. B., 1922, 809 A; 1923, 173 A; A., 1925, ii, 367; 1926, 43). Evidence is here adduced

for the presence of other types of hydrocarbons, probably branched-chain rather than cyclic or unsaturated. Paraffin wax from American mid-continent petroleum crudes, fractionally distilled at less than 1 mm. and selected fractions crystallised from ethylene dichloride, gave six final fractions, each crystallised 8–14 times. Relative purity of the fractions was indicated by small separation on further crystallisation and by examination of their cooling curves. The m.p. varied from 59.9° to 29.4°, and the highest-melting fractions exhibited the lowest mol. wts. The maximum difference in mol. wt. corresponded to slightly less than two carbon atoms. The last fraction was almost 700 times as soluble in ethylene dichloride at 14° as the first. The close agreement between the observed and calculated molecular refractivities was taken to indicate that the first five and possibly the last fractions are completely saturated.

D. W. HILL.

Acids of montan wax. H. TROPSCH (Brennstoff-Chem., 1929, 10, 403–404; cf. Holde, Bleyberg, and Vohrer, B., 1929, 667).—Holde's criticism of Tropsch's results is briefly discussed. Fractional distillation of the methyl esters *in vacuo* and fractional precipitation of the acids by magnesium acetate leads to a more efficient separation of the acids of montan wax than do the corresponding processes using the ethyl esters and lithium acetate, respectively.

A. B. MANNING.

Sampling of gas over mercury. PEXTON and HUTCHISON. **Gas analysis.** OTT. **Determination of inert gas content of gas mixtures.** LEATHERMAN and BARTLETT.—See I. **Removal of mercaptans.** GREER. **Amyl alcohol from pentanes.** AYRES. **Determination of phenol in presence of salicylates.** HAMILTON and SMITH.—See III. **Utilisation of seaweed.** DILLON and LAVELLE.—See VII. **Carbon black.** CARSON and SEBRELL.—See XIV.

PATENTS.

Plants for washing coal and other minerals. A. FRANCE (B.P. 296,775, 31.5.28. Belg., 9.9.27).—In plants for washing coal etc., in which the material is conveyed in a stream along a suitably inclined launder, the walls of the ports in the bottom of the launder, through which the particles of higher density are discharged, are formed of curved surfaces of adjustable shape and position. The discharge pocket may comprise two superposed chambers separated by a partition to which a flap is pivoted, or by an oblique partition below which a rotary drum is arranged; a shutter rotatable about a horizontal axis serves to adjust the dimensions of the discharge port. The inclination of the shutter may be automatically adjustable according to the height of the liquid stream in the launder.

A. B. MANNING.

Combustion of fuel in furnaces. J. S. CROSSLEY and G. G. TURRI (B.P. 293,461, 3.7.28. Austral., 7.7.27).—The fuel is fed well into the interior of a hollow rotary grate, preferably by means of an invertible trough sliding within a tube lying along the axis of the grate. The fuel may be dried and preheated by being retained for a time within the grate before dropping it. The grate is shaped like the frustum of a stepped cone with its axis horizontal, being made up of circular grate

bars which increase in diameter as they are further from the feed end. Passages between the grate bars serve to admit air and to allow the ash to escape. The inner surfaces of the grate bars are provided with ribs which partition the air passages, and are so designed as to promote longitudinal movement of the fuel.

A. B. MANNING.

Carbonisation and distillation of wood and coal. CARBONISATION SOC. GÉN. D'EXPLOIT. DES CARBONES, Assees. of G. JAKOVA-MERTURI (B.P. 293,321, 2.7.28. Fr., 30.6.27).—The hot combustion gases from a furnace are treated with charcoal to remove any residual oxygen, and are then passed through horizontal cylindrical retorts containing the wood or coal to be carbonised. Steam may be introduced periodically into the retort in order to regulate the progress of the carbonisation.

A. B. MANNING.

Wood distillation. F. S. CLARK (U.S.P. 1,731,242, 15.10.29. Appl., 19.4.27).—The retort containing the wood is heated by the furnace fire, to warm up the walls to prevent condensation of vapours, and also during the final carbonisation stage, but distillation of volatile products, without decomposition, is effected by closed steam-pipes. Steam is also injected into the retort during the distillation and carbonisation stages.

F. G. CLARKE.

[Heat] treatment [and carbonisation] of solid, and especially bituminous, substances. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,147, 13.7.28).—A retort for the low-temperature carbonisation of bituminous fuels consists of a number of inclined cylindrical pipes connected in series and arranged in zig-zag formation. The pipes are mounted at such an angle that the charge travels automatically down the retort under gravity. Baffles are provided to prevent the charge from completely filling the pipes. The carbonisation gases are drawn off at one or more of the angles of the retort without having to pass through the charge. The retort is heated externally by hot combustion gases which are recirculated through the heating flues and the surplus of which is discharged as required.

A. B. MANNING.

Manufacture of activated charcoal. SOC. ANON. DES CHARBONS ACTIFS E. URBAIN (B.P. 294,214, 19.7.28. Fr., 20.7.27).—Vegetable material, preferably such as yields a dense charcoal, is carbonised at below 600° and then treated with a mixture of phosphorus vapour and hydrochloric acid, carried over the material in a current of superheated steam at 500–700°. The product is washed with water until substantially free from phosphoric acid.

A. B. MANNING.

Gas-purification process and apparatus. L. MELLERSH-JACKSON. From KOPPERS Co. (B.P. 290,660, 27.4.28).—Fuel gases are washed with an ammoniacal liquid to free them from hydrogen sulphide and other acidic impurities. The absorbed impurities are oxidised by means of air in the presence of a suitable catalyst, e.g., a compound of nickel, cobalt, or iron, which is added to the liquid in amount sufficient to promote the oxidation of the sulphides to thiosulphates and to prevent the liberation of hydrogen sulphide, but insufficient to cause the liberation of free sulphur. A part of the

liquid is removed from time to time, and is heated and aerated in order to convert the ammonium thiosulphate into sulphate. The air used for oxidation is subsequently washed for the recovery of the ammonia therein.

A. B. MANNING.

Elimination of sulphur compounds from gases.

J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,190, 25.8.28. Addn. to B.P. 310,063; B., 1929, 506).—In a modification of the process of the prior patent, catalysts consisting of highly porous adsorbents of an inorganic nature, *e.g.*, active silica or alumina, are used at temperatures above 250°, and preferably above 400°.

A. B. MANNING.

Distillation of tar. BARRETT Co., Asses. of S. P. MILLER (B.P. 288,270, 26.3.28. U.S., 6.4.27).—Modifications are introduced into the processes described in prior patents, particularly B.P. 284,703 (B., 1929, 803). The tar to be distilled is brought into contact in the common collector main with the hot gases from a battery of coke ovens, and the enriched gases are cleaned by electrical precipitation while still at a high temperature. Means, *e.g.*, a rotary shaft with paddles, may be provided for spraying tar or pitch from the bottom of the main into the gases passing through. The temperatures in the collector main and in the electrical precipitator may be so regulated that different pitch products are obtained therein; these may be collected separately or, if desired, may be combined. The oils in the enriched clean gases are fractionally condensed.

A. B. MANNING.

Separation of the constituents of low-temperature tar and its distillates. G. T. MORGAN and D. D. PRATT (B.P. 318,116, 28.4.28).—The crude phenols obtained from low-temperature tar as described in B.P. 307,382 (B., 1929, 385) are treated with petroleum of low b.p. whereby the crystallisable phenols are extracted. The viscous black residue is separated into two fractions, resinols-C and -D, by treatment with ether, benzene, or low-temperature spirit; the resinols-C are left as an insoluble residue, whilst the resinols-D are precipitated on the addition of petroleum of low b.p. to the solution. These resinols may be used as a basis for lacquers or varnishes, or they may be condensed with formaldehyde to give synthetic resins.

A. B. MANNING.

Distillation of petroleum or the like. P. J. WARD, J. MITCHELL, and STEEL BROS. & Co., LTD. (B.P. 320,135, 14.6.28).—An apparatus is described wherein the latent heat of steam is used for the separation of the more volatile constituents (up to 260°) of the oil, and the latent heat of mercury for that of the less volatile constituents (up to 400°). Distillation is effected in a battery of evaporators connected in series. The incoming oil is preheated in a series arrangement of heat exchangers by condensates or vapours.

W. S. E. CLARKE.

Apparatus for condensing hydrocarbon vapours.

J. E. BELL, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,730,350, 8.10.29. Appl., 22.1.25).—The vapours pass from a still to a condenser; the condensate and uncondensed gases and vapours are further cooled in separate coolers, the condensates from the latter being passed to a common collector. A liquid-seal device maintains full of liquid the cooler for the condensate from the condenser.

F. G. CLARKE.

Conversion of hydrocarbons of high b.p. range into others of low b.p. range. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,421, 7.7.28. Cf. B.P. 315,991; B., 1929, 843).—Mineral oils, tars, and hydrogenation products of these substances and of coal are distilled at 410–600°, under pressure, in the presence of amides, amines, and/or aldehydes or ketones which contain more than 3 atoms of carbon in the molecule; cracking is facilitated by the admixture of metals and their compounds capable of combining with aldehydes and ketones. In an example, a tar fraction, b.p. 200°, which was cracked at 600° with 2% of formamide, in a quartz column filled with pumice, yielded 31% of products boiling below 200°.

C. B. MARSON.

Manufacture of hydrocarbons, particularly those of low b.p. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,473, 31.8. and 22.11.28).—Hydrogenation of coal, tar, mineral oils, etc. is carried out by fractional treatment so that constituents not affected in the first stage are removed and treated separately under more intensive conditions, *e.g.*, under higher temperatures and pressures; in the final stage it is preferable to employ the materials in the form of a vapour, the reaction being carried out at a relatively high temperature. *E.g.*, a heavy American oil, b.p. 350°, which was treated with hydrogen at 200 atm. and 450° in the presence of a molybdenum-zinc catalyst, yielded 60% of benzene and middle oil, which was removed continuously; the residue of high b.p., rich in asphalt, which was then treated with hydrogen at 1000 atm. and 460° in the presence of a molybdenum-chromium-manganese catalyst in a second reaction vessel, yielded 90% of hydrocarbons boiling below 350°.

C. B. MARSON.

Production of hydrocarbons of low b.p. from oils or from solid fuels. R. E. GOLDSBROUGH and H. TEVIS (B.P. 320,619, 13.7. and 25.8.28).—Highly-heated oil or pulverised solid fuel together with superheated steam is injected into a chamber lined with a catalyst containing, *e.g.*, silica 14 pts., sodium silicate 9 pts., alumina 4 pts., zinc oxide 2 pts., and traces of magnesite and lime. The mixture is thereby gasified and is subsequently passed through a conduit also lined with the catalyst where the temperature is reduced gradually to below the gasifying point. Vapours are cooled and condensed out of contact with the catalyst.

W. S. E. CLARKE.

Purification of the gaseous cloud formed by heat transformation of heavy oils. C. CHILOWSKY (B.P. 302,292, 13.12.28. Fr., 13.12.27).—Condensing surfaces on which tar-containing vapours are condensed become inoperative owing to the formation of a covering of heavy tar. In the apparatus described the vapours pass upwards over surfaces that are cooled by water at 40–60°. The tendency for tar to adhere to the cooling surface is thereby minimised and the lower portions are also washed by the condensed light liquids from the higher portions of the condenser. A fan may be used to circulate the hot gases in contact with the cooling surface.

T. A. SMITH.

Decolorisation of hydrocarbon oils. F. GARDNER (U.S.P. 1,732,465, 22.10.29. Appl., 10.1.27).—An apparatus is described in which two separate solids are simultaneously fed into a liquid contained in a mixing

tank, the liquid and solids being fed on to a rotary mixer in the upper portion of the tank. The lower portion of the tank is fitted with an agitator. W. S. E. CLARKE.

Removal of amorphous wax from petroleum oils. P. M. TRAVIS, Assr. to TRAVIS PROCESS CORP. (U.S.P. 1,732,143—4, 15.10.29. Appl., 16.7.27).—(A) The oil is chilled in the presence of a finely-divided solid material, and centrifuged. The resulting layer of wax-free oil is continuously discharged towards the axis of rotation from one point of the centrifuge, and the solids are removed from another point in the same direction. (B) The wax and a solution of the wax-free oil are similarly obtained by conducting the process in the presence of a low-boiling diluent. F. G. CLARKE.

Manufacture of purified montan wax. A. RIEBECK'SCHE MONTANWERKE A.-G. (B.P. 297,102, 24.7.28. Ger., 16.9.27).—Solutions of montan wax in oxygenated solvents such as mono- or poly-hydric alcohols, esters, or ketones are decolorised by boiling with activated carbon ("carboraffin") or fuller's earth. The resinous impurities, though not very soluble in the solvents, pass into solution in the presence of the wax. The bulk of the resin is removed by extracting crude wax with the cold solvent. The purified wax is nearly white and has properties comparable with those of carnauba wax.

T. A. SMITH.

Lubricating oil. D. STRYKER (U.S.P. 1,732,780, 22.10.29. Appl., 23.3.25).—A hydrocarbon oil is mixed with an anti-chattering natural wax, e.g., carnauba, bayberry, japan, spermaceti, or bees-wax.

W. S. E. CLARKE.

Manufacture of lubricants. H. VIMMIG, Assr. to TEXAS CO. (U.S.P. 1,729,823, 1.10.29. Appl., 4.10.21).—A mineral lubricating oil is compounded with lead oleate which is completely and permanently soluble in the oil; at low temperatures the product has a viscosity lower than that of the mineral oil.

H. ROYAL-DAWSON.

Gas-purification process and apparatus. F. W. SPERR, JUN., Assr. to KOPPERS CO. (U.S.P. 1,733,321, 29.10.29. Appl., 18.11.25).—See B.P. 261,755; B., 1928, 357.

Gas purification and regenerating sulphided alkaline solutions. R. A. MORGEN, W. S. YARD, and L. ROSENSTEIN, Assrs. to KOPPERS CO. (U.S.P. 1,732,905, 22.10.29. Appl., 11.10.24).—See B.P. 241,221; B., 1926, 5.

Apparatus for cracking oils. S. SEELIG (U.S.P. 1,732,664, 22.10.29. Appl., 29.12.26. Ger., 25.3.26).—See B.P. 268,323; B., 1927, 805.

Splitting of hydrocarbons. O. SCHMIDT, O. GROSSKINSKY, and G. NIEMANN, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,732,381, 22.10.29. Appl., 31.5.27. Ger., 19.6.26).—See B.P. 297,398; B., 1928, 884.

Production of non-combustible chlorinated hydrocarbon. F. S. VIVAS, Assr. to INTERNAT. FIRE-PROOF PRODUCTS CORP. (U.S.P. 1,733,843, 29.10.29. Appl., 11.3.27).—See B.P. 286,726; B., 1929, 349.

[Screening] apparatus for dry-milling of coal and its by-products. H. M. SUTTON, W. L. and E. G. STEELE (B.P. 320,621, 16.7.28).

Automatic regulators for coke-oven plants. W. M. SHALLCROSS (B.P. 321,084, 1.12.28).

Apparatus for quenching coke. SOUTH METROPOLITAN GAS CO., and A. H. ANDREWS (B.P. 320,651, 18.7.28).

Gas burners. L. H. V. REYNOLDS and W. B. ENSIGN (B.P. 320,827, 16.1.29).

Rotary furnaces (B.P. 301,904). **Thermostats** (B.P. 320,270). **Condenser** (U.S.P. 1,727,403). **Purifying liquid stored in tanks** (B.P. 320,239). **Treatment of gases** (B.P. 320,450).—See I. **Hydrogen from methane** (B.P. 319,957).—See VII. **Asphalt-like materials** (B.P. 291,749).—See IX.

III.—ORGANIC INTERMEDIATES.

Preparation of acetic anhydride. E. FRITZMAN (J. Appl. Chem., Russia, 1928, 1, 27—36).—Chlorine (320 g.) is passed during 1.25 hrs. into a cooled, agitated mixture of sodium acetate (985 g.), sulphur (48 g.), and acetic anhydride (1200 g.), and the mixture is then kept for 1 hr. at 90°. The acetic anhydride (90% yield) is distilled off at 100°/50—100 mm. Cast iron or steel apparatus can be used; lead and tin, but not aluminium, are quickly corroded. Alternatively, equimolecular quantities of sodium acetate and toluene-*p*-sulphonyl chloride afford acetic anhydride in 80—90% yield.

CHEMICAL ABSTRACTS.

Evaluation of acetic anhydride. E. TERLINCK (Chem.-Ztg., 1929, 53, 814—815, 850—851).—The various methods proposed for the evaluation of acetic anhydride are discussed, and Richmond's statement that the direct acidimetric titration of the sample with 0.5*N*-alkali leads to low results is confirmed. A new procedure is recommended in which a sample (1 g.) is treated with 50 c.c. of 0.5*N*-sodium hydroxide, and, after keeping overnight, the excess alkali is titrated with 0.5*N*-sulphuric acid. A second portion (1 g.) is treated with 15 c.c. of a 20% solution of 2:4-dichloroaniline in chloroform; after keeping overnight, 50 c.c. of 0.5*N*-sodium hydroxide are added, the whole is vigorously shaken, and the excess of alkali titrated as before. The anhydride content of the sample may then be calculated from the decreased acidity in the second case. An improved method is described for the preparation of the dichloroaniline, and for its recovery from the analysed samples.

H. F. HARWOOD.

Manufacture of anhydrous ethyl alcohol. D. B. KEYES (Ind. Eng. Chem., 1929, 21, 998—1001).—A résumé of patents to establish American priority in the development of the commercial production of absolute from 95% alcohol by distillation of the latter with a third liquid (benzene, ethyl acetate, carbon tetrachloride, etc.).

D. W. HILL.

Detection of *n*-propyl alcohol in commercial allyl alcohol. O. HÜTHIG (Ber. Schimmel, 1929, 163—165).—The presence of *n*-propyl alcohol in commercial allyl alcohol is proved by treatment of the fraction, b.p. 96—97°, with bromine in ether solution and distillation under reduced pressure. The fraction, b.p. 39°/60 mm., gives a *phenylurethane*, m.p. 50—51°, and an

α -naphthylurethane, m.p. 79–80°, identical with those obtained from *n*-propyl alcohol.

J. W. BAKER.

Amyl alcohols from the pentanes. E. E. AYRES (Ind. Eng. Chem., 1929, 21, 899–904).—The method employed in preparing the various amyl alcohols from *n*- and *iso*-pentanes consists in chlorination of the hydrocarbons followed by hydrolysis. The *n*- and *iso*-pentanes employed are derived, by fractionation, from natural-gas gasoline. Chlorination at temperatures below 100° either in the vapour or liquid phase requires the influence of light or of a catalyst, and produces low yields of primary chloropentanes. At temperatures above 200° chlorination of the vapour without the influence of catalysts or of light gives the highest yields of primary chlorides. The presence of anhydrous chlorides of multivalent metals reduces the yield of primary chlorides. The use of iodine, sulphur chloride, or red phosphorus with liquid pentanes causes excessive formation of polychlorides. Insoluble, non-volatile chlorides in contact with the liquids or vapours give high yields of monochlorides, but cause the transposition of the chlorine from primary to secondary or tertiary positions. In the large-scale process a stream of chlorine obtained by the electrolysis of brine is passed into a very rapid stream of the heated vapour of the *n*- or *iso*-pentane, and the hydrogen chloride evolved is absorbed in water; the hydrolysis of the amyl chloride is carried out by hot, aqueous caustic soda produced with the chlorine. The salt solution resulting is returned to the electrolytic cell. The products of hydrolysis together with unchanged pentane are fractionated and the desired portions are recirculated. Equal amounts of primary and secondary monochlorides are produced in the chlorination of *n*-pentane. The secondary fraction consists largely of γ -chloropentane together with some β -chloropentane. *iso*-Pentane on chlorination yields 85% of primary *iso*amyl chlorides and 15% of *tert.*-amyl chlorides, but no secondary *iso*amyl chloride. Of the primary chlorides, one third is δ -chloro- and two thirds α -chloro- β -methylbutane. It has been found impossible to hydrolyse or esterify the amyl chlorides completely, but since constant-boiling mixtures are formed with water small amounts present in the alcohols can be removed by fractionation in presence of water. In the process outlined 100,000 gals. of pentanes are chlorinated per day. H. INGLESON.

Removal of mercaptans from solution by adsorption on metallic sulphides. E. J. GREER (Ind. Eng. Chem., 1929, 21, 1033).—Ethyl mercaptan is easily removed from solution in hydrocarbons by shaking with amorphous cupric, lead, stannic, cadmium, and arsenious sulphides. Cupric sulphide also removes *sec.*-amyl mercaptan, which is difficult to remove by other means.

C. W. GIBBY.

Determination of phenol in presence of salicylates. E. H. HAMILTON and C. M. SMITH (Ind. Eng. Chem. [Anal.], 1929, 1, 232).—In mixtures (*e.g.*, coal-tar insecticides) of phenol, oil of birch, *p*-cresol, and kerosene in varying proportions, the phenol can be accurately determined by the following modification of Chapin's method (A., 1920, ii, 645). Kerosene (50 c.c.) is added to 10 c.c. of the aqueous solution of the mixture, and the

phenol is extracted by shaking three times with 100-c.c. portions of water and then determined in the combined extracts. Chapin's method for unsaponified cresols gives very high results due to hydrolysis of the methyl salicylate, and the method for saponified cresols gives only slightly high results.

E. H. SHARPLES.

Viscosity of glycerin solutions. COCKS.—See XII. **Acetic and lactic acids from sawdust.** ALLGEIER and others.—See XVIII.

PATENTS.

Refining raw carbon disulphide. P. SIEDLER and E. SCHULTE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,733,171, 29.10.29. Appl., 11.10.26. Ger., 24.10.25).—See B.P. 260,236; B., 1927, 907.

Manufacture of esters. R. WIETZEL, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,732,392, 22.10.29. Appl., 28.9.26. Ger., 5.10.25).—See B.P. 259,204; B., 1927, 571.

Isolation of alcohols or phenols from mixtures. F. O. ZEITSCHEL (U.S.P. 1,733,440, 29.10.29. Appl., 13.7.25. Ger., 1.8.24).—See B.P. 252,570; B., 1926, 691.

Condensing organic [perylene] compounds by means of aluminium chloride. A. ZINKE, Assr. to F. BENSÄ (U.S.P. 1,733,768, 29.10.29. Appl., 7.4.25. Austr., 10.4.24).—See B.P. 232,265; B., 1925, 911.

Production of compounds containing carbocyclic or heterocyclic rings. E. HOFFA and W. LUCE, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,733,458, 29.10.29. Appl., 24.4.26. Ger., 5.5.25).—See B.P. 251,997; B., 1927, 550.

Electrochemical processes (B.P. 303,027).—See XI. **Citric acid (B.P. 302,338).**—See XVIII.

IV.—DYESTUFFS.

Influence of chlorine, alone and in conjunction with the sulphonic acid group, on the colours of substituted benzeneazophenols. H. H. HODGSON and W. ROSENBERG (J.S.C.I., 1929, 48, 287–289 T).—The influence of chlorine substituted in all available positions in benzeneazophenol is found to have varying effects on the photo-activation of the system $C_6H_5 \cdot N : N \cdot C_6H_4 \cdot OH \rightleftharpoons C_6H_5 \cdot NH : N : C_6H_4 : O$ the state of strain of which will be largely determined by its degree of ionisation (incipient or otherwise) and assumed in the paper to be shown by the resultant dyeing. The electronics of such a system are tentatively discussed on current theories. In all cases the chlorine-substituted benzeneazophenols are deeper in colour than is the unsubstituted product, showing the influence of chlorine to be bathochromic, *i.e.*, to cause a resultant increase in electronic mobility, and the descending order from light yellow to pronounced orange is: aniline \rightarrow phenol, 4-chloroaniline \rightarrow phenol, 3-chloroaniline \rightarrow phenol, aniline \rightarrow 2-chlorophenol, 3:5-dichloroaniline \rightarrow phenol, sulphanilic acid \rightarrow phenol, 2-chloroaniline \rightarrow phenol, sulphanilic acid \rightarrow 2-chlorophenol, aniline \rightarrow 3-chlorophenol, aniline \rightarrow 2:6-dichlorophenol, 2:5-dichloroaniline \rightarrow phenol, sulphanilic acid \rightarrow 3-chlorophenol, sulphanilic acid \rightarrow 2:6-dichlorophenol, aniline \rightarrow 2:5-dichlorophenol, 2:6-dichloroaniline \rightarrow phenol, sulphanilic acid \rightarrow 2:5-

dichlorophenol, aniline \rightarrow 3:5-dichlorophenol, and sulphanilic acid \rightarrow 3:5-dichlorophenol, the dyestuffs above being designated by their component intermediates. Evidence in favour of the "general effect" of chlorine is given by 3-chloro- being deeper than 4-chloro-benzene-azophenol, and by the 2-chloro-isomeride having a deeper shade than that resulting from the combined general effects of both chlorine atoms in 3:5-dichlorobenzene-azophenol. The somewhat anomalous positions in the order of benzeneazo-2-chloro- and -2:6-dichloro-phenols are probably due to partial co-ordination at the *o*-chlorophenol end. In all cases examined the presence of the *p*-sulphonic acid group in the benzene residue is accompanied by a deepening of colour. Attempts to prepare unique benzenoid and quinonoid forms of *p*-nitrobenzeneazo-2-chloro- and -2:6-dichloro-phenols and also of *p*-nitrobenzeneazo-2-chloro-1-naphthol were unsuccessful.

PATENTS.

Manufacture of dyestuff preparations. F. FELIX and O. ALLEMANN, ASSRS. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,733,057, 22.10.29. Appl., 28.5.27. Switz., 29.5.26).—See B.P. 271,898; B., 1928, 781.

Manufacture of [vat and acid] dyes. J. BADDILEY, P. DOOTSON, A. SHEPHERDSON, and S. THORNLEY, ASSRS. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,734,789, 5.11.29. Appl., 2.2.28. U.K., 8.2.27).—See B.P. 289,188; B., 1928, 517.

Manufacture of sulphur dyes. A. BERGDOLT, W. NEELMEIER, and T. NOCKEN, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,733,443, 29.10.29. Appl., 13.8.27. Ger., 20.8.26).—See B.P. 299,909; B., 1929, 352.

Production of ice colours. W. CHRIST, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,733,447, 29.10.29. Appl., 19.11.25. Ger., 1.12.24).—See G.P. 433,149; B., 1927, 276.

Manufacture of water-soluble arylazodiazarylamine dyes. H. EICHWEDE, E. FISCHER, and A. SIEGLITZ, ASSRS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,734,246, 5.11.29. Appl., 13.8.27. Ger., 21.8.26).—See B.P. 276,372; B., 1929, 239.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Action of caustic soda on wool. J. B. SPEAKMAN (J.S.C.I., 1929, 48, 321—324 T).—No explanation has previously been given of the increased strength of wool yarns after immersion in 38% caustic soda solution for 5 min. A study of the elastic properties of single fibres shows them to be completely unaffected by similar treatment, and their immunity is referred to the low partial pressure of water vapour in equilibrium with the caustic soda solution and to the formation of the complex hydrate $2\text{NaOH}\cdot 7\text{H}_2\text{O}$. The increased strength of wool yarns, following treatment with concentrated caustic soda solution, is due to surface gelatinisation of the fibres produced by the dilute alkali formed during subsequent neutralisation and washing processes; this binds the fibres firmly together in the dried yarn.

Adsorption of vapours by animal and vegetable fibres. E. V. ALEKSEEVSKI (J. Appl. Chem., Russia, 1928, 1, 184—189).—Experiments with trichloronitro-

methane, arsenic trichloride, methyl sulphate, benzyl chloride, and bromoacetone on various fabric fibres are described.

CHEMICAL ABSTRACTS.

Chemical injury of vegetable fibre products. H. SOMMER (Mitt. Materialprüf. Berlin, 1929, No. 6, 51—55; Chem. Zentr., 1929, i, 3053).—Weakening or rotting is due to the formation of oxy- and hydro-cellulose. Methods for the detection of these substances and of photocellulose resulting from the action of light or weather are considered. A. A. ELDRIDGE.

Wood cooking with magnesium bisulphite solution. VON POSSANNER (Papier-Fabr., 1929, 27, 537—543).—Methods of preparing magnesium bisulphite liquors from dolomite and from the magnesium chloride waste-liquors of the potash industry are described. Dolomite is calcined and treated with water and sulphur dioxide in Mitscherlich towers, thus giving a mixture of calcium and magnesium bisulphite, whilst magnesium salt solutions are treated with lime, and the hydroxide so obtained is converted into bisulphite by means of sulphur dioxide. If magnesium sulphate is used instead of the chloride, insoluble calcium sulphate is formed and interferes with the working of the absorption towers. In cooking, magnesium bisulphite is more resistant to heating under pressure than is the calcium salt, and no separation of monosulphite occurs, whereas with the calcium salt both monosulphite and sulphate are formed and interfere with the penetration of heat through the pulp. Admixture of magnesium bisulphite with calcium bisulphite in the liquor shortens the time of cooking. This time is shortest if 75% of the former and 25% of the latter are present, but the product is more readily bleached and a higher yield is obtained if the magnesium salt alone is used. The yield is higher, and the percentage of ash and lignin and the Sieber numbers of the product are lower, when pure magnesium liquor is used than when either a mixed liquor or calcium bisulphite solution is employed. Although cost is the deciding factor, if large quantities of magnesium chloride liquor are available as raw material, considerable advantages result from the use of magnesium bisulphite.

B. P. RIDGE.

Determination of α -cellulose. G. J. RITTER (Papier-Fabr., 1929, 27, 678—682).—Modifications of the ordinary method of determining α -cellulose have been examined for the purpose of establishing a simple standard procedure which will give accurate and reproducible results. Two samples of cotton linters and three of sulphite-pulp were analysed in several different laboratories by four modifications of the same general method which differed according to the concentration of the sodium hydroxide solution used for mercerisation, time of steeping before maceration, total time of mercerisation, volume of water used to dilute the mercerising alkali, etc. As a result, the fourth modification (known as Method IV of the Cellulose Section of the American Chemical Society) is recommended. In this procedure 3-g. samples of material are treated with 7.5% sodium hydroxide solution, the mixture is kept for 5 min., and is then macerated while further quantities of sodium hydroxide are added. After mercerisation for 45 min., the mixture is diluted with a quantity of water equal to

that of the sodium hydroxide added, and the whole is filtered through a Gooch crucible. The filtrate is poured back through the cellulose mass until all suspended particles are retained, and the residue is washed with water and acetic acid, and dried to constant weight at 105°. All solutions and washing liquids are maintained at 20° throughout the operations. With those materials which presented no difficulties in maceration etc. the same results were obtained in all the laboratories concerned using this procedure, but with one sample of cotton and one of sulphite-cellulose, known to give trouble, the results were less uniform. B. P. RIDGE.

Fractionation of cellulose esters. J. DUCLAUX and R. NODZU (Rev. gén. Colloid., 1929, 7, 241—250).—Fractional precipitation of commercial nitrocellulose from its acetone solution by gradual addition of water or aqueous acetone leads to a separation into several fractions, each giving solutions of very different viscosities; those of high viscosity, in general, precipitate spontaneously, whilst those of low viscosity may be isolated by ultrafiltration or by coagulation. A nitrocellulose may be characterised by its specific viscosity deduced from the viscosity of its 2% solution in methyl ethyl ketone. Fractionation appears to be determined by the different size of the micelles and not by composition, the different fractions having nearly the same nitrogen content. Other methods of fractionation consist in diffusing a nitrocellulose gel in alcohol or in ultrafiltration of a dilute solution, the diffused portion or the ultrafiltrate containing the fine micelles of low viscosity; in this way very pure products may be obtained free from substances in suspension.

F. R. ENNOS.

Effect of thinners on the viscosity of nitrocellulose solutions. B. M. PAM (J.S.C.I., 1929, 48, 223—226 T).—With a mixture of solvents and non-solvents for nitrocellulose which represented a possible schedule for dope manufacture, it was found that the order in which the solvents and non-solvents were added had no influence on the viscosity of the product, provided sufficient time and agitation were allowed. The influence on the viscosity of the addition of excess of liquids already present both individually and in mixture has been examined. The "15% rule" is found to be far from true even with the most efficient thinners, and the reduction in viscosity % on the addition of thinners is proportional not to the percentage of thinner added, but to the logarithm of that addition.

Storage properties of transparent celluloid. O. C. ELLINGTON (J.S.C.I., 1929, 48, 267—276 T).—Fourteen samples of transparent sheet celluloid typical of the manufacturers of six countries have been examined. The samples fell into two distinct classes, viz., class *a*, characterised by inferior stability, by acidity, and by high contents of sulphates and mineral matter, and class *b*, characterised by superior stability and the relative absence of the above impurities. The results were confirmed by a preliminary warm-storage deterioration trial. It was shown that the use of inadequately purified nitrocellulose is the main cause of the liability of transparent celluloid to rapid deterioration and to spontaneous inflammation or even explosion under

adverse conditions of storage. The discrimination of celluloids as to stability in storage should be based on tests which indicate with certainty the purity of the nitrocellulose ingredient used. Such tests are described and limits are suggested. Urea has a pronounced stabilising effect on celluloid made from impure nitrocellulose, but by addition of urea such celluloid cannot be rendered as stable as material made from pure ingredients. The behaviour of celluloid in the later stages of its deterioration is discussed. The occurrence of mineral matter in excessive quantity in incompletely stabilised nitrocellulose is explained and the mechanism of the stabilisation process is discussed.

Effect of temperature on the strength of bag-paper. W. HERZBERG (Mitt. Materialprüf. Berlin, 1929, No. 6, 4—6; Chem. Zentr., 1929, i, 3050).—In 24 hrs. at 80° soda-paper gained 22% and sulphite-paper 10% in strength. A. A. ELDRIDGE.

[Test for] paper constituents injurious to metals. W. HERZBERG (Mitt. Materialprüf. Berlin, 1929, No. 6, 18; Chem. Zentr., 1929, i, 3050).—The paper is compared with a standard by wrapping around a polished steel plate and preserving in a moist atmosphere, or by clamping the paper between zinc and copper plates for 1 hr. in a moist atmosphere. A. A. ELDRIDGE.

Influence of atmospheric moisture on the properties of fibres and the measurement of atmospheric moisture. G. HERZOG (Mitt. Materialprüf. Berlin, 1929, No. 6, 24—30; Chem. Zentr., 1929, i, 3052).

Differentiation of viscose and copper-silk. O. RÜNZI (Kunstseide, 1929, 11, 158—160; Chem. Zentr., 1929, i, 3055).

Liquorice root. HOUSEMANN and LACEY.—See XX.

PATENTS.

Washing or cleaning of wool. H. W. FAWCETT (B.P. 319,026, 14.3.28).—Wool-washing liquor is purified by passing through a tank containing a series of inclined plates at a speed below that at which the deposited dirt is remingled with the liquid, and thence to a centrifuge comprising a number of rotating, superimposed, conical plates. F. R. ENNOS.

Treatment of jute to obtain a fibre similar to wool. TEXTILES (NEW PROCESS), LTD., Asses. of J. VIALLET (B.P. 309,021, 15.1.29. Fr., 3.4.28).—Waste jute materials, after treatment in a bath containing 2—3% of an enzyme, e.g., diastase, together with soda in order to remove starch and glue, are submitted to the fermenting action of micro-organisms from virgin jute at 30—35° in the presence of potassium phosphate, and are finally unravelled in a damp state.

F. R. ENNOS.

Treatment of cellulose. A. THIRIET (B.P. 318,868, 8.3.28).—Wood cellulose prepared by the soda or bisulphite treatment is purified by subjecting it while flowing down an inclined gauze to jets of water under pressure. After a thorough oxidation it is then washed with water free from alkaline-earth salts, treated with sodium hydroxide solution containing less than 0.001%

of alkaline-earth salts at ordinary or elevated temperature, washed again, and finally bleached.

F. R. ENNOS.

Acetylation of cellulose. RUTH-ALDO CO., INC., Assees. of H. L. BARTHELEMY (B.P. 303,099, 3.8.28. Fr., 28.12.27. Addn. to B.P. 282,791; B., 1929, 429).—The reaction cylinder, which is mounted on hollow trunnions permitting the introduction of the reagents and also of the inlets and outlets of heating or cooling coils, is made to rotate about an axis which is parallel to but off-set from the plane passing at right angles through the centre of its longitudinal axis.

F. R. ENNOS.

Manufacture of chloroacetic cellulose esters. SOC. DES USINES CHIM. RHÔNE-POULENC (SOC. CHIM. DES USINES DU RHÔNE), H. GAULT, and F. BIDAUD (B.P. 318,908, 12.6.28).—Cellulose is heated with monochloroacetic anhydride and a catalyst, *e.g.*, sulphuric acid, the ester being separated by diluting with a non-solvent such as ether either before or after dissolution of the cellulose.

F. R. ENNOS.

Manufacture of cellulose derivatives and solutions of cellulose. L. LILIENFELD (B.P. 318,088, 22.3.28).—Cellulose is pretreated with alkali and carbon disulphide under such conditions that at least a substantial part of the resulting product is insoluble in water. After filtration and washing, if necessary, the cellulose product is converted into viscose, cuprammonium cellulose, or cellulose esters or ethers by the usual methods.

F. R. ENNOS.

Manufacture of cellulose esters. I. G. FARBENIND. A.-G. (B.P. 292,929, 25.6.28. Ger., 25.6.27. Addn. to B.P. 284,298; B., 1929, 593).—Solubility of the esters is assisted by heating them at a high temperature in an anhydrous liquid with an acid halide (inorganic or organic) and, if desired, an acid, an acid anhydride, a salt of a strong acid with a weak base, or a mixture of these.

F. R. ENNOS.

Manufacture of artificial silk yarns and the like. W. P. DREAPER (B.P. 320,100, 6.6.28).—The "dry"-spun artificial silk yarn is removed from the centrifugal box in the form of a cake and wound directly on to a suitable holder; while passing from cake to holder it may be dried, treated with oil solution or sizing material, or given an extra twist.

F. R. ENNOS.

Manufacture of yarns, filaments, ribbons, fabrics, etc. from organic derivatives of cellulose. H. DREYFUS (B.P. 320,363, 5.4.28).—Materials having less tendency to lose their lustre and become opaque and crinkled when treated with hot water are made by spinning cellulose acetate of acetyl value (calc. as acetic acid) 52.5–56% (especially 54.5%) from solution in aqueous acetone of 97.5–100%, of 88–72%, or of lower concentration, under conditions precluding precipitation of the ester.

F. R. ENNOS.

Production of compound materials containing sheets of cellulose derivative. BRIT. CELANESE, LTD., and J. H. ROONEY (B.P. 320,374, 6.7.28).—Sheets of cellulose acetate which have been treated with a mixture of a non-solvent, *e.g.*, methylated spirit, and a plasticiser, *e.g.*, triacetin, are applied while wet to one or more sheets of glass and subjected to heat and pressure; the

edges may then be sealed by means of a solution containing acaroid resin.

F. R. ENNOS.

Desulphurising artificial products made from viscose. O. Y. IMRAY. FROM HERMINGHAUS & CO. G.M.B.H. (B.P. 319,098, 18.7.28).—The artificial silk is treated with a liquid organic desulphurising agent, *e.g.*, pyridine, or with a solution of one or more desulphurising agents in one or more organic solvents, *e.g.*, phenol or sodium sulphide in ethyl alcohol, which are either free from water or contain insufficient water to have any appreciable swelling action on the product.

F. R. ENNOS.

Surface-treatment of celluloid articles to render the same practically unflammable. A. WEBER, H. HÖPPNER, JUN., and H. WEICH (B.P. 316,276, 25.9.28. Ger., 27.7.28).—The articles are sprayed with a solution of acetate-celluloid in acetone containing amyl acetate, a surface gloss being imparted, if desired, by addition of shellac dissolved in alcohol or varnish lac.

F. R. ENNOS.

Covering articles with non-flammable celluloid. R. J. CARRUTHERS (B.P. 320,504, 20.9.28).—Non-flammable celluloid is softened by steeping in a mixture of petrol, acetone, and methylated spirit, wiped dry, and applied to the article which has previously been coated with an intermediate adhesive consisting of cuttings of the celluloid dissolved in methylated spirit and camphor; the covering is then stretched over the article, allowed to dry, and polished.

F. R. ENNOS.

Manufacture of ethers or esters of carbohydrates of the type $(C_6H_{10}O_5)_x$. I. G. FARBENIND. A.-G. (B.P. 293,316, and Addn. B.P. 293,757, [A] 2.7.28, [B] 5.7.28. Ger., [A] 30.6.27, [B] 11.7.27).—(A) After removal of the associated air by passing a current of gas or vapour capable of being absorbed by alkali, *e.g.*, carbon dioxide, acetaldehyde, through the carbohydrate contained in a vessel which is maintained under a vacuum, the alkali compound is formed by treatment with caustic soda and esterification is completed in the usual manner. (B) A current of gas or vapour, which is absorbed or condensed on access of the esterifying liquid, is passed through a vessel containing the carbohydrate under a vacuum until the associated air is eliminated.

F. R. ENNOS.

Treatment of flax. O. D. LUCAS, ASSR. TO VICKERS, LTD. (U.S.P. 1,733,742, 29.10.29. Appl., 13.1.28. U.K., 5.7.27).—See B.P. 298,178; B., 1928, 889.

Manufacture of textile yarns. ABBEY SYND., LTD., and D. R. NANJI (B.P. 320,860, 20.7.28).

Manufacture of composite yarns and the like. W. P. DREAPER (B.P. 318,531, 5.6.28).

[Pump] apparatus for spinning artificial silk. H. WADE. FROM SYNTHETA A.-G. (B.P. 320,858, 18.7.28).

Drying of films, bands, etc. of cellulose and the like. WOLFF & CO. KOMMANDIT-GES. AUF AKT., and R. WEINGAND (B.P. 300,496, 27.9.28. Ger., 12.11.27).

Manufacture of tubular bodies from cellulose solutions, particularly viscose. KALLE & CO. A.-G. (B.P. 297,103, 26.7.28. Ger., 15.9.27).

Method and apparatus for coating paper, fabrics, etc. C. MÜNCH (B.P. 291,031, 23.5.28. Ger., 24.5.27).

Purification of [zinc chloride] solutions (B.P. 294,259).—See VII. **Cellulose-resin product** (B.P. 305,671).—See XIII. **Leather substitute** (B.P. 320,444).—See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Dyeing of artificial silk. IV. Conditions for uniform dyeing of artificial silks (except acetate silk) with substantive dyes. W. WELTZIEN and K. GÖTZE (Seide, 1929, 34, 136—143; Chem. Zentr., 1929, i, 3038).—For uniform dyeing the degree of dispersion of the dye is of little importance. The temperature of the bath is related to the amount and uniformity of the dye taken up. Simple rules governing the dependence of uniformity on the operating conditions are elucidated. A. A. ELDRIDGE.

Dyeing wool with direct cotton dyes. A. E. PORAI-KOSHITZ (J. Appl. Chem., Russia, 1928, 1, 11—20).—Experiments with benzopurpurin 4B and diamine-violet N show that, unlike cotton, wool adsorbs only sulphonic acids. CHEMICAL ABSTRACTS.

Action of caustic soda on wool. SPEAKMAN.—See V.

PATENTS.

Bleaching of fur skins. H. STEIN, W. E. AUSTIN, and I. LIEBOWITZ (B.P. 318,471—2, 3.5.28).—The skins, e.g., brown moufflons which have been washed with sodium carbonate solution, rinsed, and hydro-extracted, are (A) treated (preferably brushed) with an aqueous solution containing about 3—7.5% of hydrogen peroxide, 0.5—1.5% of concentrated ammonia, and 1—2% of concentrated potassium soap solution, after which they are dried in the air at about 15—38°, or (B) brushed with a solution containing a bleaching agent, preferably 4—7.5% of hydrogen peroxide, and an organic aromatic reducing agent which may (e.g., *o*- or *p*-aminophenol, *p*-phenylenediamine) or may not (e.g., quinol, phenylhydrazine) impart a desired tint to the skin, with the addition, if desired, of an inorganic reducing and accelerating agent, e.g., ferrous sulphate, after which they are air-dried. L. A. COLES.

Removal of ink spots and the like. C. H. KIDWELL, Assr. to KIDWELL & Co., Inc. (U.S.P. 1,732,606, 22.10.29. Appl., 4.1.24).—Ink spots containing an iron compound are removed from paper, cloth, etc. by moistening them with water, soaking with 5% sodium sulphide solution, washing, acidifying with 5% oxalic acid solution, and again washing. H. ROYAL-DAWSON.

Production of coloured woollen and worsted materials in two or more harmonious shades. F. BAUER (B.P. 319,112, 9.8.28).—Two-tone coloured wool piece-goods are obtained by weaving together unbleached white yarn and yarn mordanted by boiling with chromium fluoride and formic acid (this mordant protects the wool during subsequent dyeing) and afterwards dyeing; the mordanted wool dyes to a darker shade; A. J. HALL.

Printing on wool. I. G. FARBENIND. A.-G. (B.P. 298,648, 12.10.28. Ger., 13.10.27).—Wool may be printed with water-soluble ester salts of the leuco-compounds of vat dyes and afterwards fully oxidised, without staining the white unprinted parts, by using acidified persulphate solution as a developer. E.g., wool is printed with Indigosol Orange HR made into a paste with a dilute solution of British gum, then dried, steamed for ½ hr., developed in a bath at 50—70° containing potassium persulphate acidified with hydrochloric acid (*d* 1.16) or its equivalent of sulphuric acid, rinsed, soaped, rinsed, and dried. A. J. HALL.

Multi-colour printing process. A. SARK and J. KOROLEW (B.P. 318,844, 8.5.28).—A multi-coloured printing block is prepared by assembling according to a desired pattern a number of shaped units pressed or moulded from coloured plastic masses prepared by suitably mixing kaolin, chalk, white lead, and similar substances with adhesive, water-soluble, viscous, colloidal substances such as syrups, and colouring matters; the joining edges of the shaped units may be blended or effaced by use of a heated tool. Before printing, the fabric, paper, or other material is moistened with a solvent (e.g., dilute alcohol or glycerin) to facilitate transference of colour from the block to the material. A. J. HALL.

Improvement of artificial fibrous materials. L. LILIEFELD (U.S.P. 1,724,670, 13.8.29. Appl., 22.5.26. Austr., 17.6.25).—To increase its strength, artificial silk is treated with a solution of caustic alkali containing not more than 5% by wt. of alkali hydroxide (calc. as NaOH), while stretching the material during at least a part of the treatment. F. R. ENNOS.

Shrinking and felting of animal fibres. J. H. MARTIN (U.S.P. 1,727,374—5, 10.9.29. Appl., 7.2.28. Renewed 31.1.29).—The process is carried out in the presence of a liquid to which is added the condensation product of an aldehyde with (A) a bisulphite or (B) sulphur dioxide. F. R. ENNOS.

Improving [raising the ironing temperature of] textile and other materials. BRIT. CELANESE, LTD. (B.P. 304,596, 17.1.29. U.S., 21.1.28).—Material composed wholly or partly of cellulose acetate is treated at 70—90° with a solution containing 0.3—0.7 g./litre of caustic soda, the alkalinity being maintained within these limits by additions of soda from time to time, whereby a regular and partial (not more than 6%) hydrolysis of the cellulose ester takes place. F. R. ENNOS.

Rendering material mothproof. LARVEX CORP., Assees. of M. G. MINAEFF (B.P. 301,421, 8.11.28. U.S., 29.11.27).—The material is treated with a 2% solution of thiourea or phenylthiourea in aqueous acetone containing a sulphonated oil to facilitate wetting. F. R. ENNOS.

Treatment of vegetable textile fibres to render them non-putrefiable. J. J. LAMBRECHT (B.P. 294,101, 20.6.28. Fr., 16.7.27).—A mixture of oleic acid, soda lye, and a light-coloured mineral oil in water is agitated to cause saponification of the acid, and is then emulsified with boiling water containing sodium carbonate and soda lye. The textile material is heated with this liquor

either in an open vessel or under 3—4 atm., and afterwards bleached and washed in acidulated water.

F. R. ENNOS.

Dyeing of vat colours on fabrics. J. WAKEFIELD (U.S.P. 1,733,439, 29.10.29. Appl., 21.5.28. U.K., 23.6.27).—See B.P. 307,238; B., 1929, 354.

Washing, bleaching, or dyeing artificial silk on cross-wound bobbins. MASCHINENFABR. SCHWEITER A.-G. (B.P. 304,310, 17.12.28. Ger., 19.1.28).

Treatment with liquids of [twisted] artificial threads prepared from cellulose solutions. W. W. GROVES. From HERMINGHAUS & Co. G.M.B.H. (B.P. 297,063, 13.9.28).

Acidification of fabrics. F. R. GROSSER (B.P. 320,996, 27.8.28).

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

Ammonia oxidation in [relation to sulphuric acid] chamber plants. T. R. OLIVE (Chem. Met. Eng., 1929, 36, 614—617).—The use of nitrogen oxides derived from ammonia is increasing in sulphuric acid manufacture not only on economic grounds, but also owing to the regularity and capability of control of the process. An alloy of platinum and rhodium is more efficient, and has a longer life than platinum as a catalyst and the newer forms of design enable the external heating of the catalyst to be dispensed with. Careful filtering of the gases before they reach the catalyst is necessary, and in the particular converter described the catalyst consists of four layers of 80-mesh gauze made of platinum-rhodium alloy wire (No. 750 alloy; diam. 0.003 in.) with a service life of 6—12 months.

C. A. KING.

Recent developments in the electrochemical production of hydrogen peroxide. L. LOWENSTEIN (Chem.-Ztg., 1929, 53, 821—822).—The electrolysis of sulphuric acid for the manufacture of hydrogen peroxide works with a low current efficiency, but the subsequent distillation of persulphuric acid *in vacuo* is simple and almost automatic. The use of ammonium sulphate solution, with which the efficiency is much better, has hitherto involved difficulties in the distillation, or if it is converted into potassium persulphate the labour cost is much increased. A process has now been worked out in which the anode solution is drawn into tubular distillation vessels in the upper part of which fractionation takes place. This permits direct working from ammonium sulphate. The manufacture of 1 kg. of 30% hydrogen peroxide thus requires only about 4½ kw.-hrs. of direct current and 1 kw.-hr. of alternating current.

C. IRWIN.

Burkheiser ammonium sulphite-bisulphite process. III. Oxidation of ammonium sulphite and bisulphite in aqueous solution. E. TERRES and A. HEINSEN (Gas- u. Wasserfach, 1929, 72, 994—998, 1022—1026, 1050—1054. Cf. B., 1928, 156).—The velocity of oxidation is reduced with increasing concentration of sulphite or bisulphite, whence it may be concluded that it is the dissociated part of the salt which participates chiefly or entirely in the reaction.

The rate of oxidation of bisulphite increases with the rise of temperature much more quickly than that of sulphite. Lead peroxide catalyses the former reaction, but no catalyst has been found to accelerate air oxidation of sulphite. The reaction velocity with constant mass of sulphites was determined by agitating a bulb containing the solution together with oxygen and connected to a manometer. The partial pressures of ammonia and sulphur dioxide under reaction conditions are obtained from previous experimental results. Then $K = 1/t \cdot \log [a/(a-x)]$, where a is the barometric pressure less partial pressures, and x the manometer reading in time t . K was found to decrease with decreasing oxygen concentration and (*vide supra*) with increasing sulphite concentration. The rate of oxidation of the solutions used in the Burkheiser process will be quite small, but repeated use of wash-liquors will make the method practical. Concentrated bisulphite solutions are the more readily oxidised. The formation of "Burkheiser salt" is sufficiently explained by these results, and the conditions for its avoidance are established. An acid bisulphite solution should be oxidised to bisulphate and used as wash-liquor. The ammonium sulphate solution remaining after neutralisation should be saturated with sulphur dioxide and used for washing, whereby a sulphite-sulphate solution is obtained which is oxidised by aeration before it returns to process. By the interchange of wash-liquor the deposition of sulphite should be avoided. A temperature of 40—50° is best for oxidation. Experiments with catalysts which led to no very good result are described in detail.

C. IRWIN.

Hyposulphite meter. R. FEIBELMANN and W. MEVES (Chem.-Ztg., 1929, 53, 749).—The meter consists of a glass-stoppered cylinder graduated from 0 to 115%, the zero graduation being some distance from the bottom. The hyposulphite preparation (1 g.) is placed in the cylinder and cold, air-free water is added up to the zero mark. After stirring, a standard solution of ferric sulphate containing thiocyanate is added until, after mixing, the solution remains pink. The percentage of hyposulphite present is then read directly from the height of the liquid above the zero mark.

A. R. POWELL.

Electrolytic production of potassium ferricyanide from potassium ferrocyanide. I. G. SHCHERBAKOV (J. Appl. Chem., Russia, 1929, 2, 155—165).—The use of the liquid mercury cathode is described. The average current density should be 1.5—2 amp./in.² at 20—30°. If electrolysis is continued after all the ferrocyanide has been oxidised acid salts are formed.

CHEMICAL ABSTRACTS.

Electrolytic preparation of hydrogen. II. K. TERANO and H. SHIMOYAMA (Bull. Coll. Eng. Kyushu, 1928, 3, 227—241).—Electrode corrosion diminishes in the order: nickel steel, cobalt steel, steel, nickel-plated steel, nickel. Corrosion is marked with alkali carbonate solutions. The cathode overvoltage is lowest with nickel and with rough surfaces. As electrolyte 20% sodium hydroxide solution is preferably employed with an asbestos diaphragm.

CHEMICAL ABSTRACTS.

Determination of inert gas content of gas mixtures. LEATHERMAN and BARTLETT.—See I.

Magnesium bisulphite solution. VON POSSANNER. See V. **Lime putty.** COWPER and WILLIAMS.—See IX. **Arsenious iodide and its solutions.** COCKING.—See XX.

PATENTS.

High concentration of nitric acid. E. I. DU PONT DE NEMOURS & Co. (B.P. 292,951, 15.6.28. U.S., 27.6.27).—Dilute nitric acid is passed as vapour up a tower in which it is brought in contact with a countercurrent of cold, concentrated sulphuric acid; the vapour issuing from the top is conveyed to a reflux condenser which returns to the top of the tower a portion of the condensed acid (1.5–1.75 lb. per 1 lb. removed from the system at 20°); this cools and further dehydrates the emergent vapours. The spent sulphuric acid is denitrated with just sufficient steam for the purpose, and the dilute nitric acid vapour liberated passes into the bottom of the tower. L. A. COLES.

Manufacture of hydrochloric acid. G. P. ADAMSON, Assr. to GEN. CHEM. Co. (U.S.P. 1,729,431, 24.9.29. Appl., 20.12.27).—A mixture of sodium chloride and sulphuric acid is heated at 113–118° to produce pure, gaseous hydrogen chloride. H. ROYAL-DAWSON.

Conducting chemical reactions [manufacture of hydrogen cyanide]. G. D. BAGLEY, Assr. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,731,331, 15.10.29. Appl., 26.2.25).—Nitrogen is brought into reactive relationship with a bath of hydrocarbon oil, in the vicinity of which is an electric arc maintained under the surface of the bath. H. ROYAL-DAWSON.

Production of ammonium nitrate. APPAREILS ET EVAPORATEURS KESTNER (B.P. 305,121, 20.11.28. Fr., 31.1.28).—Ammonium nitrate solution in a container situated below a tower and provided with an overflow for draining off excess liquor is pumped through a cooler to the top of the tower; after admixture with about 2% of nitric acid, the liquor is neutralised in the tower with a countercurrent of ammonia and the neutral solution, the temperature of which should not exceed 35°, flows back into the container. L. A. COLES.

Preparation of [anhydrous] calcium sulphate. P. SPENCE & SONS, LTD., and S. F. W. CRUNDALL (B.P. 319,228, 14.6.28).—The product is obtained in a finely-divided, amorphous form by treating the hydrated sulphate, *e.g.*, gypsum, at raised temperatures with dehydrating agents, *e.g.*, concentrated sulphuric acid, sulphur trioxide, phosphoric acid, acid sulphates. L. A. COLES.

Manufacture of calcium cyanide. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,733, 15.9.28).—Calcium oxide and/or hydroxide is treated with dry hydrogen cyanide and the water formed during the reaction is withdrawn as vapour or gas by mixing with the hydrogen cyanide dry nitrogen or other inert gas. The process is carried out at 10–40° and at ordinary pressure. (Cf. B.P. 300,348; B., 1929, 53.) W. G. CAREY.

Production of chemically active anhydrous aluminium oxide, and its uses. G. A. BLANC (B.P. 319,850, 6.7.28).—Hydrated aluminium nitrate pro-

duced, *e.g.*, as described in B.P. 181,677 or 246,827 (B., 1923, 402 A; 1927, 75) is heated at 140–180° under atmospheric pressure. This oxide has a special application in the manufacture of aluminium sulphate and the electrolytic preparation of the metal. L. A. COLES.

Production of alkali phosphates and ammonium phosphates. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,318, 8.6.28).—A mixture of dicalcium phosphate and an alkali or ammonium bisulphate is heated below sintering point, *e.g.*, at 50–60°, for $\frac{1}{2}$ –1 hr., and the product is leached with water to extract the desired alkali or ammonium phosphate. L. A. COLES.

Production of lasting, storable carbon dioxide developing powder for foods etc., as, *e.g.*, baking powder. L. WEIL (B.P. 303,353, 15.12.28. Ger., 31.12.27).—A neutral, anhydrous alkaline-earth or magnesium pyrophosphate in quantity sufficient to maintain the gas-forming constituents in a dry state is added as filler to baking powder etc. L. A. COLES.

Generating energy from water or aqueous solutions. T. D. KELLY (B.P. 319,604, 23.6.28).—Water containing acids, alkalis, salts, or gases to render it unstable, *e.g.*, 2–10% of nitric acid, calcium nitrate, or nitrous oxide, is injected, with or without subjection to the action of an alternating or direct electric current, into a hot chamber which is preferably lined with silicon carbide and contains air under pressure. The solution is thereby split up into hydrogen, oxygen, etc., and these gases are subsequently fired, the pressure generated being used for the production of power. Air, steam, lubricating oil, etc. may flow through the apparatus to keep it cool. L. A. COLES.

Purification of metallic salt solutions contaminated by organic substances. I. G. FARBENIND. A.-G. (B.P. 294,259, 20.7.28. Ger., 21.7.27).—The solutions, *e.g.*, spent zinc chloride liquor from the manufacture of parchment paper, are treated with oxygen or gases containing it at about 130° under raised pressure. L. A. COLES.

Removal of halogen ions from metal salt solutions containing same as impurities. METALLGES. A.-G. (B.P. 301,512, 30.11.28. Ger., 1.12.27).—Salt solutions for use, *e.g.*, in the electrodeposition of metals flow through a cell containing mercury or a mercury amalgam as anode, under conditions such that the quantity of mercury dissolved is just sufficient to precipitate halogen ions as mercurous halide. L. A. COLES.

Colloidal iodine preparations in gel form. J. COFMAN-NICORESTI (B.P. 320,719, 24.8.28).—Iodine is dissolved in a hydrocarbon oil or oleic acid, the solution is dispersed in colloidal state by an alcohol, *e.g.*, ethyl, methyl, isopropyl alcohols, or mixtures of these, and is stabilised by fatty acids or their salts, *e.g.*, palmitic or stearic acid, or by agar, gelatin, pectin, gums, resins, tallow, etc.; metallic salts or hydroxides may also be added. W. G. CAREY.

Production of hydrogen from methane. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 319,957, 18.10.28).—Methane is treated with steam in proportions

and under conditions such that the issuing gases contain 2–15% (preferably 4–5%) of unchanged methane, which is subsequently removed, *e.g.*, by combustion with oxygen or, together with carbon dioxide, by extraction with solvents (gasoline) under pressure. Removal of carbon monoxide from the products may be facilitated by converting, prior to the removal of undesired gases, the greater part of it into carbon dioxide and hydrogen by the action of steam and the remainder catalytically into methane. L. A. COLES.

Apparatus for making hydrogen peroxide by distillation of persulphuric acid and persulphate solutions and for the concentration of hydrogen peroxide solutions. OESTERR. CHEM. WERKE G.M.B.H. (B.P. 293,755, 4.7.28. Austr., 11.7.27).—Heated portions of the apparatus, the inner surfaces of which are covered continuously with a layer of the liquid under treatment, are constructed of a metal, *e.g.*, lead, acid-resisting iron alloys, which, although having a catalytic action on persulphuric acid, persulphates, and hydrogen peroxide, is not attacked by the residual liquor or solvent. The portions in contact with the cold liquid and with hydrogen peroxide are constructed of ceramic ware; *e.g.*, the liquid under treatment is drawn by suction from a ceramic container through a metal tube heated externally by steam, and thence into a separator constructed of ceramic ware. L. A. COLES.

Production of dense carbon dioxide snow from liquid carbon dioxide. MIDDEN-EUROPEISCHE OCTROOIMAATSCHAPPIJ (B.P. 314,371, 1.1.29. Switz., 26.6.28).—Liquid carbon dioxide is converted into wet carbon dioxide snow by expansion to a pressure below the triple-point pressure, compression to above the triple-point pressure, and expansion still further, thus freezing the moist snow by sucking the vapour evolved from the bottom of the expansion chamber through a permeable plate upon which the snow rests. W. G. CAREY.

Filtering and extracting sulphur. A. J. CROWLEY, Assr. to HUMBOLDT SULPHUR Co. (U.S.P. 1,731,562—3, 15.10.29. Appl., [A, B] 16.7.27).—(A) Molten sulphur is passed through a filtering medium so that a constant volume is maintained on each side of the latter. (B) Steam is used to superheat a mixture of water and the material containing sulphur, whereby the sulphur meets and continuously enters a tubular passage; the coalesced sulphur and residue pass on to a larger part of the tube in which the sulphur solidifies on cooling. H. ROYAL-DAWSON.

Catalytically manufacturing phosphoric acid and hydrogen. A. MITTASCH and G. WIETZEL, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,732,373, 22.10.29. Appl., 14.11.25. Ger., 22.11.24).—See G.P. 431,504; B., 1926, 916.

Separation of solid salts of ammonium and of the alkalis or alkaline earths. P. OSSWALD and K. SCHAD, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,733,272, 29.10.29. Appl., 10.10.25. Ger., 31.10.24).—See B.P. 242,975; B., 1927, 251.

Production of pure aluminium hydroxide particularly from aluminium-containing raw materials rich in silica. E. L. RINMAN (U.S.P. 1,732,772, 22.10.29.

Appl., 14.11.25. Swed., 22.11.24).—See B.P. 243,356; B., 1927, 74.

Recovery of vanadium [from solutions]. R. H. STEVENS, G. C. NORRIS, and W. N. WATSON, Assrs. to RHODESIA BROKEN HILL DEVELOPMENT Co., LTD. (U.S.P. 1,733,700, 29.10.29. Appl., 24.9.26. S. Afr., 7.4.26).—See B.P. 269,780; B., 1927, 555.

Preparation of agglomerated solid carbon dioxide. W. HESSLING (B.P. 294,584, 26.7.28. Ger., 27.7.27).

Crystallisation (B.P. 301,496).—See I. Copper oxide-ammonia cellulose solution (U.S.P. 1,728,565).—See V. Sulphur from pyrites (U.S.P. 1,731,516).—See X. Electrochemical processes (B.P. 303,027).—See XI. Fertilisers (B.P. 320,708).—See XVI. Oxygen-evolving preparations (B.P. 319,393).—See XXIII.

VIII.—GLASS; CERAMICS.

Determination of fluorine and of silica in glasses and enamels containing fluorine. J. I. HOFFMAN and G. E. F. LUNDELL (Bur. Stand. J. Res., 1929, 3, 581–595).—The method of Berzelius for the separation of silica and fluorine is tedious and gives low results for both substances, but the following method is rapid and gives accurate results for quantities of fluorine between 0.01 and 0.1 g. in the presence of boric, chromic, sulphuric, or phosphoric acid, although it is not satisfactory for the analysis of phosphate rock or of materials with a high magnesia content. None of the usual constituents of enamels or glasses interferes. The sample (0.5 g.) is fused with 5 g. of sodium potassium carbonate and the product is leached with hot water. The residue is collected and boiled with 50 c.c. of 2% sodium carbonate solution to remove the last of the fluorine. The combined filtrates (300 c.c.) are boiled for 1 min. with a solution of 1 g. of zinc oxide in 20 c.c. of 1 : 9 nitric acid to precipitate the greater part of the silica; the precipitate is collected and washed with hot water, and the filtrate nearly neutralised with nitric acid, evaporated to 200 c.c., neutralised exactly with 1 : 9 nitric acid until methyl-red indicator becomes a very faint pink, and boiled with a solution of 1 g. of zinc oxide in aqueous ammonia and ammonium carbonate until the odour of ammonia disappears. The precipitate is collected, washed with hot water, and, together with those previously obtained, evaporated with 25 c.c. of hydrochloric acid on the steam bath. The dry residue is moistened with 10 c.c. of hydrochloric acid and 150 c.c. of hot water are added. The silica is collected after 15 min., washed, dried, and ignited at 1200°; the filtrate is evaporated with 10 c.c. of sulphuric acid until copious fumes are evolved, water is added, and the remaining silica collected and added to that previously obtained. The usual purity test is applied. The filtrate from the zinc oxide precipitation is treated with 2 drops of a 0.4% solution of bromophenol-blue and 3 c.c. of 10% sodium chloride solution, the volume adjusted to 250 c.c., and dilute nitric acid added until the colour changes to yellow followed by dilute sodium hydroxide solution until the blue is just restored. The solution is treated with 2 c.c. of 1 : 1 hydrochloric acid and 5 g. of solid lead nitrate, heated until the latter

dissolves, and for a further 30 min. after addition of 5 g. of sodium acetate crystals, and set aside overnight. The precipitate of lead chlorofluoride is collected on a close-textured filter paper, washed once with cold water, five times with a cold saturated solution (0.325 g./litre) of lead chlorofluoride, and again once with cold water. Precipitate and paper are then stirred with 100 c.c. of 5% nitric acid on the water-bath, an excess of 0.2*N*-silver nitrate is added to precipitate the chlorine, and the excess is titrated with thiocyanate, using iron alum as indicator (1 c.c. of 0.2*N*-silver nitrate = 0.00380 g. of fluorine). For good results the above details must be followed exactly.

A. R. POWELL.

Tenacity of the system clay-water. O. BARTSCH (Ber. Deut. keram. Ges., 1929, 10, 146—184; Chem. Zentr., 1929, i, 3025).—Measurements of the deformation-resistance were made for various clay-water mixtures, to some of which electrolytes had been added.

A. A. ELDRIDGE.

Clay slip. I. Electrolytes, fluidity, and hydrogen-ion concentration. T. YOSHIOKA and T. IRIE (J. Japan. Ceram. Soc., 1928, 36, 312—323).—Addition of alkali (within certain ranges) increases, and of acids decreases, fluidity; sodium silicate or hydroxide (*e.g.*, 0.16—0.24%) has a greater deflocculating action than the carbonate. The fluidity is slightly increased by ageing.

CHEMICAL ABSTRACTS.

Ceramic coatings. MORRELL and FARAGHER.—See II.

PATENTS.

Manufacture of compound [unsplinterable] glass. RÖHM & HAAS A.-G. (B.P. 297,413, 11.7.28. Ger., 21.9.27).—Sheets of glass are united by polymerisable, unsaturated organic compounds, *e.g.*, methyl cinnamate, ethyl crotonate, which may be polymerised, *e.g.*, by the action of heat or light, before or after application to the glass and may, if desired, be applied in solution, the solvent being subsequently expelled. Colouring and/or strengthening material may also be added.

L. A. COLES.

Colouring glassware [for electric lamp bulbs]. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of M. PIPKIN (B.P. 300,179, 7.11.28. U.S., 7.11.27).—Bulbs frosted on the interior are dusted inside with a dry powder comprising tungstic oxide and other suitable pigments, *e.g.*, zirconium oxide, cadmium sulphide, ultramarine, with the addition, if desired, of red phosphorus.

L. A. COLES.

Apparatus for treating clay. J. C. BLACK, Assr. to CONTACT FILTRATION Co. (U.S.P. 1,731,702, 15.10.29. Appl., 2.8.27).—Clay is heated and agitated with acid, in tanks provided with separate acid and water supplies, and is then transferred to water wash-tanks, in which it is again heated and agitated, the wash water being removed by means of siphons. After passing to a steam-heated receiver, the product is filtered, dried, and pulverised. It has clarifying and decolorising properties.

F. G. CLARKE.

Manufacture of [ceramic] abrasive wheel. H. R. SIMONDS, Assr. to A. A. SIMONDS-DAYTON Co. (U.S.P. 1,732,287, 22.10.29. Appl., 1.7.25).—An abrasive is

bonded with compounds of the alkali and alkaline-earth metals and boric oxide in such quantity that, when fused together into the completed wheel, the expansion of the wheel in use will be negligible.

H. ROYAL-DAWSON.

Manufacture of abrasive wheels. CARBORUNDUM Co., LTD. From CARBORUNDUM Co. (B.P. 319,221 and 319,200, 15.5.28).

Manufacture of bricks and operation of brick kilns. J. S. WHITEHOUSE (B.P. 321,023, 26.9.28).

IX.—BUILDING MATERIALS.

Cellular cement mortars. C. R. PLATZMANN (Cement, 1929, 2, 263—269).—The results of two processes (U.S.P. 1,087,098 and G.P. 327,907; B., 1914, 355; 1921, 180 A) which aim at the production of cellular building material by means of the generation of gas in a cement mixture during the initial setting stage are compared. In the former process, in which 0.1—0.25% of powdered aluminium is incorporated, fineness of grain of the metal and of any filling material, *e.g.*, coke, is necessary to prevent segregation. Increases in volume up to 130% have been obtained without sand, which reduces the expansion to 60—75%, though up to 30% of sand improves the strength. The German method, which employs zinc in a similar manner, showed less satisfactory results with only 30—50% increase in volume, and then only when extraneously heated; better results were obtained when calcium chloride was also introduced. Other gas-forming chemicals, *e.g.*, carbide, were tried, but with negative results; it is considered that the lime in the cement furnishes a valuable third component to the reaction.

C. A. KING.

Influence of aluminium on mortar strength. H. W. LEAVITT, J. W. GOWEN, and L. C. JENNESS (Proc. Nat. Acad. Sci., 1929, 15, 740—742).—A consistent increase (up to 8%) in tensile strength is developed by mortar on the 28-day test as the aluminium content of the sand increases. The breaking strength also increases in the same way. The aluminium content does not affect the compressive strength to such a marked extent.

A. J. MEE.

Joint influence of iron and aluminium in native [Maine] sands on mortar strength. H. W. LEAVITT, J. W. GOWEN, and L. C. JENNESS (Proc. Nat. Acad. Sci., 1929, 15, 742—743; cf. B., 1927, 483).—Iron and aluminium jointly account for about 16% of the variation in the strength developed by mortars made from native sands containing these substances. In general, the chemical condition of the sand used in cement mortars materially affects the strength of the hardened mortars.

A. J. MEE.

Influence of sugar on the setting and hardening of Portland cement. H. W. GONELL (Zement, 1929, 18, 372—375, 437—441, 472—474; Chem. Zentr., 1929, i, 3026).—In presence of sugar, the gel formation in the setting and hardening of Portland cement takes place at the expense of crystal formation. Addition of sodium carbonate favours crystal formation, considerable separation of calcium carbonate taking place.

A. A. ELDRIDGE.

Volume-yield and density of lime putty. A. D. COWPER and J. F. WILLIAMS (J.S.C.I., 1929, 48, 276—279 T; cf. B., 1928, 194).—A rapid method for determining "volume-yield" of lime putty in standard tests of building limes is described, involving only determination of density. The density of dispersed wet calcium hydroxide is found to be constant ($= 2.12$) over a wide range of conditions.

Heat-insulating materials. STILES.—See I. **Effect of stored coal on concrete.** WOLF.—See II. **Liquor-ice root.** HOUSEMAN and LACEY.—See XX.

PATENTS.

Manufacture of quick-setting lime products. C. C. LOOMIS, and A. D. MACDONALD, Assrs. to COLLOIDAL LIME PLASTER CORP. (U.S.P. 1,732,409, 22.10.29. Appl. 5.4.26).—Quicklime, pulverised to pass through a 5-mesh/in. sieve, is partly converted into carbonate by intimate contact with carbon dioxide, such process being continued until the increase in weight is within the range 3—40% of the original weight of the lime.

H. ROYAL-DAWSON.

Manufacture of [coloured] cement. H. C. BADDER (B.P. 320,597, 26.4.28).—Mixtures of finely-ground material comprising a pure base of Portland or aluminous cement, metal oxides, carbonates or salts, or earths or pigments adapted to produce the desired colour, and a flux, are heated to fusion under oxidising conditions.

L. A. COLES.

Manufacture of cellular concrete. K. P. BILLNER, Assee. of N. E. BROANDER (B.P. 306,444, 13.8.28. U.S., 20.2.28).—The use is claimed of finely-ground mixtures of hydraulic cement, puzzuolanic material (blast-furnace slag, lava, trass), and material for producing a cellular structure, e.g., aluminium powder, lime and gypsum being added, if necessary. [Stat. ref.] L. A. COLES.

Production of asphalt-like road building and the like material, from coal tar or pitch. W. SCHROTH (B.P. 291,749, 7.6.28. Ger., 7.6.27).—The phenol content of tar or pitch is adjusted to about 8—10% by suitably blending tars of high and low phenol content, or by adding phenols to pitch, before the material is treated with sulphur and/or oxidising agents, with or without the addition of contact material, for the production of asphalt substitutes.

L. A. COLES.

Brick kilns. J. S. WHITEHOUSE (B.P. 320,741, 26.9.28).

Compositions for road making etc. HEWITT (DARLSTON), LTD., and A. HEWITT (B.P. 319,904, 4.9.28).

Making bituminous emulsions and distributing same on road surfaces. H. WADE. From SOC. PROVIA (B.P. 320,919, 19.6.28).

Treatment of crude oils, residues, etc. (B.P. 319,673 and 319,718).—See II. **Conversion of smelts** (B.P. 294,175).—See X.

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

Hadfield steel. Correlation of physical properties with microstructure and thermal treatment. V. N. SVETCHNIKOV (Rev. Mét., 1929, 26,

401—408).—Measurements of the resistance to shock on a Charpy pendulum machine, and to wear by loss of weight when rubbed on moist carborundum, were carried out on a Hadfield manganese steel containing 1.23% C, 12.67% Mn, 8.35% Si, and 0.1% P. Specimens were used (1) as cast, (2) forged but not thermally treated, (3) forged and quenched in water at 850°, 950°, 1050°, and 1250°, and the results obtained with the treated specimens were plotted against the quenching temperatures. The curves for shock and wear tests both show a sharp change in direction for an optimum quenching temperature of 1150°, whilst the curves for Shore hardness, Brinell hardness, and sp. gr. show a similar but less well-defined change in direction at the same temperature. The cast steel consists of austenite and the eutectic surrounded by a network of carbide. After forging, the metal consists of austenite and a network of a granular constituent. Quenching the forged specimens at increasingly high temperatures results in a progressive diminution in quantity of the granular constituent, which is accompanied by an increase in the viscosity and the resistance to wear. The last traces of the granular constituent disappear at a quenching temperature of 950°, whilst the maximum viscosity is reached only at 1150°. Quenching at 1050° produces a fine-grained structure of pure austenite, slip-bands being very prominent; at 1150° the structure is similar but the grains are larger and more irregular, whilst at 1250° another constituent appears, partly present as a network and partly in the form of rounded grains which treatment with Stead's reagent shows to be rich in phosphorus; the decrease in shock-test value and wearing properties of test-pieces quenched at this temperature is probably due, therefore, to the presence of a network of this phosphoric constituent. The superiority of Hadfield steel over other steels of equal wearing properties is due to its high viscosity, whilst its high resistance to wear is due to the rapidity with which it undergoes cold-hardening. When quenched at 1150° and reheated for 2 hrs. at 570°, it loses much of its viscosity, but its resistance to wear is not affected. The micrographic results can all be attributed to a high phosphorus content.

M. E. NOTTAGE.

Hot aqueous solutions for quenching of steels. H. J. FRENCH and T. E. HAMILL (Bur. Stand. J. Res., 1929, 3, 399—418).—The rates of cooling from 875° of the surface and central portions of steel (0.96% C) cylinders 0.5 in. diam. and 2 in. high in two oils, water, 5% sodium hydroxide, and 5% sodium chloride solutions at 20—100° have been determined and cooling curves plotted and correlated with the microstructures and hardness obtained. The rate of cooling was reduced by increase in temperature of the cooling medium, but was regular in the central zones with aqueous solutions up to 80°, above which it became irregular. The results indicate that warm salt or alkaline solutions are suitable quenching media when rates of cooling intermediate between those obtained in water and oils are required.

A. R. POWELL.

Determination of manganese in steel and iron by the persulphate-arsenite method. A. A. BRIGHT and C. P. LARRABEE (Bur. Stand. J. Res., 1929, 3,

573—579).—The following modified procedure gives good results with up to 20 mg. of manganese. The iron or steel (1 g.) is heated with 30 c.c. of a mixture of 100 c.c. of sulphuric acid (*d* 1.84), 125 c.c. of 85% phosphoric acid, and 250 c.c. of nitric acid (*d* 1.4) diluted to 1 litre. When red fumes cease to be evolved, 100 c.c. of cold water, 10 c.c. of 0.8% silver nitrate solution, and 10 c.c. of 25% ammonium persulphate solution are added and the solution is boiled briskly for 30 sec., cooled, diluted with 75 c.c. of water, and titrated with 0.03*N*-sodium arsenite to a clear yellow end-point. The procedure is the same for cast iron except that the graphite is filtered off before oxidation of the manganese. Chromium-tungsten steels (0.5 g.) should be dissolved in 50 c.c. of 1:9 sulphuric acid and 3 c.c. of 85% phosphoric acid; 40 c.c. of water are then added and the solution is boiled with 10 c.c. of 1:1 nitric acid until the tungsten carbide is all dissolved. The analysis is then finished as described above.

A. R. POWELL.

Composition of ϵ -bronze. W. HUME-ROTHLEY (Phil. Mag., 1929, [vii], 8, 114—121).—It is shown that under ordinary conditions of examination the composition of ϵ -bronze can vary between the limits 24.5—25.1 at.-% Sn, but there is no conclusive evidence to show whether ϵ -bronze is a solid solution within these limits or whether there exists the compound Cu_3Sn lying in this range, in which there is slight solubility of copper on the one hand and of tin on the other. It is suggested that the ultimate answer to the question will be provided by a detailed X-ray examination, and the precautions to ensure adequate annealing for this purpose are indicated.

A. E. MITCHELL.

Relation between adsorption, wetting power, and flotation. W. LUYKEN and E. BIERBRAUER (Metall u. Erz, 1929, 26, 197—202; Chem. Zentr., 1929, i, 3030—3031).—Experiments with apatite, calcite, wollastonite, magnesite, celestine, and galena in relation to the adsorption of sodium palmitate are described. The adsorption, which is attributed to the exceptional affinity of the sodium palmitate for the alkaline-earth metals, takes place only when the adsorption compound is less soluble than the adsorbent. The sodium palmitate becomes concentrated at the air-water interface, and the individual molecules become oriented. After adsorption the minerals show a diminution of wetting power. The flotation of cassiterite is discussed from the authors' point of view.

A. A. ELDRIDGE.

Extraction of zinc from sulphide ores. V. ILJINSKI and A. SAGAJDITSCHNI (Trans. Sci. Inst. U.S.S.R. Sci. Tech. Dep., Russia, 1928, No. 259, 187—193; Chem. Zentr., 1929, i, 2915).—At 650—700° in an atmosphere of sulphur dioxide conversion into sulphate is complete. Unroasted zinc sulphide ores yield up to 95% of the zinc to sulphuric or hydrochloric acid; under the conditions described only small amounts of sesquioxides pass into solution. Treatment of the roasted ore with alkali dissolves 80% of the zinc, no iron, and only traces of copper.

A. A. ELDRIDGE.

Effect of oxidising conditions on accelerated electrolytic corrosion tests. H. S. RAWSON and W. A. TUCKER (Bur. Stand. J. Res., 1929, 3, 375—390).—The tests were made on sheet copper and nickel, both

anode and cathode being of the same metal, in dilute sodium chloride solution, and the results are expressed in the form of potential-loss in weight curves. The effect of partial immersion as compared with complete immersion was to increase appreciably the anode losses, especially when the overlying atmosphere was oxygen. A nitrogen atmosphere reduced the losses considerably in both cases, showing that the presence of oxygen reduces the potential required to produce a given rate of corrosion. The effect of differential oxidation (aeration), whereby those parts having free access to the air are rendered cathodic and those having less access anodic, originates within the cell and is additional to that resulting from the applied external *E.M.F.*; it therefore tends to increase the corrosion losses, the oxygen acting as a cathode depolariser. In practically all cases the observed corrosion losses were greater than those corresponding with a 100% anode efficiency as calculated in accordance with Faraday's laws. These irregularities show that the accelerated electrolytic corrosion test as at present conducted cannot be considered a satisfactory routine test for the behaviour of a metal under corrosive conditions.

A. R. POWELL.

Electrodeposition of silver from argentocyanide solutions. S. GLASSTONE and E. B. SANIGAR (Trans. Faraday Soc., 1929, 25, 590—598).—The silver deposits formed on prepared sheet cathodes by electrolysis of alkali argentocyanide solutions containing an excess of cyanide and one of the following anions: formate, acetate, hydroxide, phosphate, sulphate, carbonate, chloride, borate, for 1 hr. with a cathode current density of 0.35 amp./dm.² and using pure silver anodes were investigated. Carbonate or an excess of free cyanide decreased the hardness of the deposits, whilst the other anions each increased the hardness, especially borate and chloride. The presence of the anions investigated caused an increase in the polarisation during deposition, but it was very small in the case of solutions containing a large excess of cyanide. Sodium solutions produced slightly more polarisation than the corresponding potassium solutions. Cathodic polarisation and the quality of the silver deposits did not appear to be inter-related, the former appearing to be connected with the alkali metal ions and the latter with the anions. The silver concentration and not the conductivity of the electrolyte determines the limiting current density at which the metal can be deposited with 100% efficiency.

H. T. S. BRITTON.

[Catalyst for] ammonia oxidation. OLIVE.—See VII. Soils and iron. HOLLER.—See XVI. Liquorice root. HOUSEMAN and LACEY.—See XX.

PATENTS.

Conversion of smelts [molten blast-furnace slag] into foaming highly porous masses. T. GILLER, Assee. of P. OERT (B.P. 294,175, 13.7.28. Ger., 19.7.27).—The slag is poured successively into a series of small containers arranged on the periphery of a vertical revolving wheel which automatically empties the granulated slag from the containers and refills them with the regulated quantity of water sufficient to convert the slag into a dry porous mass.

A. R. POWELL.

Employment of cerium in the production of steel and iron. I. G. FARBENIND. A.-G. (B.P. 302,574, 21.11.28. Ger., 17.12.27).—The cerium is added as a scavenger in the form of an alloy with iron and another metal, *e.g.*, vanadium, aluminium, silicon, calcium, magnesium, or titanium, the reaction products of which increase the fluidity of the reaction products of the cerium. A. R. POWELL.

Production of metals [iron, by the carbonyl process]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,177, 13.8.28).—The gases obtained by the thermal decomposition of iron carbonyl at 280° contain about 2% CO₂, which is removed by means of quicklime, and the residual carbon monoxide is passed over metallic iron produced by the reduction of iron oxide to regenerate iron carbonyl. The yield is increased by 40% as a result of the purification of the gas. A. R. POWELL.

Soldering of iron and steel parts. F. VON WURSTEMBERGER (B.P. 309,032, 2.4.29. Ger., 3.4.28).—The parts are first nickel-plated then soldered with a copper alloy. A. R. POWELL.

Alloy for high-speed steel. M. S. CLAWSON (U.S.P. 1,729,154, 24.9.29. Appl., 26.6.20).—The alloy contains 13–40% W or Mo, 5–20% Cr, 14–75% Fe, 0.5–3% V, 4–20% Co, and 0.85–3.5% C. H. ROYAL-DAWSON.

Hard-metal alloy for [cutting] tools. J. BERTRAM (B.P. 320,991, 23.8.28. Addn. to B.P. 232,591; B., 1925, 962).—In alloys of the type described in the prior patent, the nickel is replaced up to one third of its amount by cobalt. F. G. CROSSE.

Alloy of aluminium, silicon, and iron. B. D. SAKLATWALLA, Assr. to VANADIUM CORP. OF AMERICA (U.S.P. 1,732,915, 22.10.29. Appl., 2.2.28).—A densifying and cleaning alloy for steel contains 5–30% Al, 60–90% Si, and the remainder is chiefly iron; not more than 0.4% C is present. H. ROYAL-DAWSON.

Welding rod. J. R. DAWSON, Assr. to ELECTRO-METALLURGICAL Co. (U.S.P. 1,728,174, 17.9.29. Appl., 8.3.24).—The rod consists of an iron alloy containing about 0.5% V, less than 0.2% C, and about 1.2% Cr. H. ROYAL-DAWSON.

Aluminium-welding rod. H. GILBERT (U.S.P. 1,728,052, 10.9.29. Appl., 30.7.28).—The rod consists of 88.74% Al, 2.42% Cu, 8.06% Cd, and 0.78% Bi. H. ROYAL-DAWSON.

Magnetic alloys. SIEMENS & HALSKE A.-G. (B.P. 290,658, 20.4.28. Ger., 20.5.27).—Alloys having high permeability with low magnetic forces and containing 30–80% Ni, 20–70% Fe, and 5.5–20% Si, the nickel in which may be wholly or partly replaced by cobalt, are claimed. The magnetic properties of such alloys are increased by heating for 1 hr. at 900°, slowly cooling to about 625°, keeping at that temperature for 15–30 min., and finally cooling in air. F. G. CROSSE.

[Non-magnetic] ferrous alloys. S. E. DAWSON (B.P. 320,332, 4.4.28).—A non-magnetic alloy highly resistant to corrosion and having an austenitic structure comprises iron with 0–2% Si, 0.2–0.35% C,

3–14% Mn, 3–12% Ni, and 5–15% Cr. The preferred composition is 0.2% C, 7% Mn, 7% Cr, 7% Ni, and 79% Fe. [Stat. ref.] A. R. POWELL.

Hard metal composition [tungsten-cobalt alloy]. BRIT. THOMSON-HOUSTON Co., LTD., Asses. of E. G. GILSON (B.P. 289,477, 27.4.28. U.S., 28.4.27).—A mixture of tungsten, carbon, and cobalt, all in powdered form, is pressed into a carbon mould and heated to 1300–1450° (the sintering temperature of the mixture) between electrodes while subjected to a pressure of 1000 lb./in.²; the temperature should be maintained for several minutes. A suitable mixture contains 5–25% of cobalt and an amount of carbon equal to about 6% of the tungsten content. F. G. CROSSE.

Extraction of copper-nickel matte. W. J. HARSHAW and G. L. HOMER, Assrs. to HARSHAW CHEM. Co. (U.S.P. 1,729,423, 24.9.29. Appl., 28.4.26).—The matte after being ground is leached with acid, lightly roasted, and leached again with acid. H. ROYAL-DAWSON.

Manufacture of shaped pieces consisting of homogeneous alloys of lead with alkali or alkaline-earth metals. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 300,129, 5.11.28).—The alloy of lead with one or more of the metals is extruded at a temperature between 200° and the m.p. of the alloy. Thus, a lead-sodium alloy (20% Na) is extruded at 260–330° under 800–3000 kg./cm.² M. E. NOTTAGE.

Alloy of magnesium. J. A. GANN, Assr. to DOW CHEM. Co. (U.S.P. 1,729,339, 24.9.29. Appl., 27.10.21).—The alloy contains 90–95% Mg and 10–5% Cd. H. ROYAL-DAWSON.

Solder. J. SILBERSTEIN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,728,772, 17.9.29. Appl., 9.9.27).—A lead-thallium alloy is claimed containing 99–80% Pb and 1–20% Tl. H. ROYAL-DAWSON.

Treatment of zinc ores. SOC. MINIERE & METALLURG. DE PENARROYA (B.P. 313,430, 29.6.28. Fr., 11.6.28).—The finely-divided zinc ore is mixed with cellulosic organic material such as sawdust, chaff, or paper pulp, the charge being artificially humidified if the necessary degree of humidity does not already exist in the cellulosic material. The subsequent calcination of oxide ores or desulphurisation of sulphur ores may be effected, if desired, simultaneously and in the same apparatus. M. E. NOTTAGE.

Recovery of precious metals by amalgamation. D. ENZLIN and J. A. EKLUND (B.P. 320,185, 20.8.28).—The finely-ground ore is mixed with a dilute solution of zinc chloride, mercuric chloride, hydrochloric acid, and chlorine, and the pulp is passed over iron or nickel plates coated with zinc or tin amalgam wherein the precious metals collect. The tailings are dewatered, the solution is electrolysed to regenerate free chlorine, mercuric chloride is added, and the liquor returned to the circuit. A. R. POWELL.

Thermostatic element. W. M. CHACE, Assr. to W. M. CHACE VALVE Co. (U.S.P. 1,729,245, 24.9.29. Appl., 19.12.27).—A bimetallic thermometer consists of two metal parts welded together, the composition of which, respectively, is: 0.35% C, 2% Si, 40% Ni,

57.65% Fe; and 0.25% C, 1.75% Si, 13.25% Cr, 2.5% Ni, 5% Cu, and 77.25% Fe (wrought).

H. ROYAL-DAWSON.

Production of metal plates and sheets for offset printing. I. G. FARBENIND. A.-G. (B.P. 293,834, 9.7.28. Ger., 13.7.27).—The plates or sheets are made of magnesium or a high-grade magnesium alloy, e.g., 5.2% Al, 1% Zn, 93.8% Mg, which is grained before use by immersion in 1–3% nitric acid for 10–100 sec. according to the fineness required. A. R. POWELL.

Electrolytic manufacture of iron. E. KELSEN (B.P. 306,151, 15.2.29. Austr., 16.2.28).—The conductivity of the usual ferrous chloride electrolyte is increased by diluting it slightly and adding a mixture of two other chlorides, e.g., potassium and sodium or potassium and calcium chlorides. A suitable mixture is FeCl₂ 122.5 g., CaCl₂ 109 g., KCl 110 g., and H₂O 525 g.

A. R. POWELL.

Apparatus for electrolytically depositing chromium. SOC. CHIM. DE LA SEINE, and V. SZIDON (B.P. 320,440, 28.7.28).—The articles to be plated are supported upon a horizontal buss-bar which is mounted so as to be capable of vertical movement in the bath, and which is provided with a brush moving over an electrical resistance so that the current is adjusted automatically to the area immersed. Movable anodes shaped to correspond with the shape of the articles to be treated are mounted in a frame supported inside the bath so that the distance between the anodes and cathodes is kept uniform. A. R. POWELL.

Electrolytic preparation or purification of metals [beryllium, tungsten, tantalum, or molybdenum]. L. ST. C. BROUGHAL (B.P. 320,818, 31.12.28).—A solution of a salt of the metal in liquid ammonia is electrolysed at a low temperature or under pressure between electrodes of non-rusting steel. A. R. POWELL.

Heat treatment of steel. S. MAKITA, ASSR. to KABUSHIKI KAISHA NIHON SEIKOSHO (U.S.P. 1,733,669, 29.10.29. Appl., 19.4.27. Jap., 22.6.26).—See B.P. 273,248; B., 1928, 451.

Improving the strength qualities of steel wire. W. PÜNGEL, ASSR. to VEREIN. STAHLWERKE A.-G. (U.S.P. 1,732,615, 22.10.29. Appl., 1.6.27. Ger., 4.6.26).—See B.P. 272,240; B., 1928, 820.

Reduction of ores and the like. W. BUDDÉUS (U.S.P. 1,732,788, 22.10.29. Appl., 14.1.27. Ger., 20.1.26).—See B.P. 264,834; B., 1927, 942.

Treatment of ores for recovery of titanium. A. W. GREGORY (U.S.P. 1,734,034, 5.11.29. Appl., 16.10.26. U.K., 20.10.25).—See B.P. 251,527; B., 1926, 591.

Recovery of zinc oxide. F. RÜSBERG and P. SCHMID, ASSRS. to KALI-CHEMIE A.-G. (U.S.P. 1,732,662, 22.10.29. Appl., 24.10.28. Ger., 11.11.27).—See B.P. 300,271; B., 1929, 718.

Aluminium solder. A. J. LINE (U.S.P. 1,732,727, 22.10.29. Appl., 7.6.26. U.K., 8.9.25).—See B.P. 262,192; B., 1927, 80.

Preparation of thorium alloys. H. S. COOPER, ASSR. to KEMET LABS. CO., INC. (U.S.P. 1,732,326,

22.10.29. Appl., 4.5.25).—See B.P. 279,274; B., 1928, 20.

Moulds for casting metals. A. E. WHITE. From ALUMINIUM, LTD. (B.P. 320,947, 27.7.28).

Making of metal wool. BRILLO MANUFACTURING CO., INC., ASSEES. OF C. FIELD (B.P. 292,473, 18.6.28. U.S., 18.6.27).

Polishing preparation for metal and other surfaces and its manufacture. A. VAN DER BRUGGEN (B.P. 309,853, 16.6.28. Fr., 16.4.28).

Furnaces (U.S.P. 1,725,879 and 1,727,898). Reverberatory furnaces (B.P. 318,748).—See I. Washing of minerals (B.P. 296,775).—See II. Anhydrous aluminium oxide (B.P. 319,850).—See VII. Separation of minerals (B.P. 302,725).—See XI.

XI.—ELECTROTECHNICS.

Dielectric losses at high frequencies. G. E. OWEN (Physical Rev. 1929, [ii], 34, 1035–1039).—The power loss in very small pieces of dielectrics in high-frequency alternating fields was measured by a calorimetric method in which the heat produced in the dielectric was compared with that produced in a resistance carrying a measured direct current. For vulcanised fibre, celluloid, rubber dam, and glass the loss was found to be proportional to the frequency and to the square of the applied voltage. N. M. BLYTH.

Chemistry in incandescence lamp manufacture. W. J. BARTLETT (Ind. Eng. Chem., 1929, 21, 970–973).—Chemical processes used in the manufacture of such lamps are briefly reviewed, with special reference to the preparation of tungsten for filaments, gases for filling the lamps, getters, basing cements, inside frosting, and lamp coatings. C. J. SMITHELLS.

Rugged type of calomel electrode vessel. L. V. SORG (Cereal Chem., 1929, 6, 344–346).—A new type of calomel electrode intended for routine work and giving reasonable accuracy combined with the prevention of diffusion into and out of the cell is described. The liquid junction is easily renewable. The whole cell, constructional details of which are shown by diagram and photograph, can easily be made in the laboratory. E. B. HUGHES.

Coking of pitch. KOETTITZ.—See II. **Potassium ferricyanide.** SCHERBAKOV. **Hydrogen.** TERANO and SHIMOYAMA. **Hydrogen peroxide.** LÖWENSTEIN.—See VII. **Electrolytic corrosion tests.** RAWDON and TUCKER.—See X. **Electrodeposition of silver.** GLASSTONE and SANIGAR.—See X. **Automatic pH recorders.** HOLVEN.—See XVII. **Sodium glutamate.** HAN.—See XIX. **Purification of water.** BLOCHER. **Chlorination of drinking water.** BAILEY.—See XXIII.

PATENTS.

Separation of materials of different physical qualities, especially of raw mining products. F. KRUPP GRUSONWERK A.-G. (B.P. 302,725, 13.12.28. Ger., 21.12.27).—In two oscillating electric circuits, tuned to the same frequency and each actuating an amplifying valve, a phase displacement is produced by

entrance of material to be treated into the field of an induction coil and/or condenser in one of the circuits, and is used to generate current or voltage which is used, either directly or after amplification, to control the separating apparatus. J. S. G. THOMAS.

Electrochemical [oxidation and reduction] processes. E. CITOVICH (B.P. 303,027, 24.12.28. Ger., 24.12.27).—The processes are conducted with a dilute electrolyte at a temperature between 100° and its critical temperature, and with the use of oxidation or reduction electrodes consisting of solid conductors, *e.g.*, metals, alloys, metal oxides or sulphides, solid fuels, the electrodes being activated by the gases generated during the process. A suitable galvanic battery is built on the filter-press system, and is so constructed that gases may be manufactured under high pressure and that the conveyance of the reacting materials and the regulation of the temperature are effected by the circulation of the electrolyte or the gas. Examples are given of the accumulation of energy by the decomposition of water and subsequent recombination of the high-pressure gases, the generation of pure high-pressure hydrogen, the recovery of pure oxygen and nitrogen from the air, and the generation of working energy from substances of different potential. Other possibilities cited are the production of chlorine, bromine, and iodine, and the reduction of nitrobenzene to aniline by the process.

L. A. COLES.

Apparatus for measuring ultra-violet radiations. A. K. CROAD. From HANOVIA CHEM. & MANUF. Co. (B.P. 320,794, 8.11.28).—Ultra-violet radiation and visible light from a source of controllable intensity, after incidence on a fluorescent material and a non-fluorescent reflector placed in an opaque chamber, are transmitted through a colour filter. J. S. G. THOMAS.

Electrolytic rectifier. E. P. LUNDEEN, Assr. to WILLARD STORAGE BATTERY Co. (U.S.P. 1,729,429, 24.9.29. Appl., 14.4.27).—The breakdown voltage of an electrolytic cell having a film-forming electrode is increased by being heated prior to the formation of the film thereon. Such an electrode having a homogeneous structure of amorphous and crystalline metal thoroughly intermixed and containing less than a normal amount of amorphous metal is claimed.

H. ROYAL-DAWSON.

Electric battery. SCHMID-PATENT CORP., Assees. of A. SCHMID (B.P. 293,299, 15.6.28. U.S., 2.7.27).—A cathode composed largely of aluminium is immersed in a flowing electrolyte, *e.g.*, a solution of sodium chloride, containing substances such as sodium chlorate and sulphuric acid, and a mixture of sodium chloride and hydrochloric acid, respectively, for depolarising the anode and for preventing polarisation of the cathode. A porous carbon anode, through which the electrolyte is forced, may be used.

J. S. G. THOMAS.

Electric discharge tube. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 320,783, 1.11.28).—The wall of an electric discharge tube containing helium and using the positive-column light is composed partly or wholly of a material, *e.g.*, yellow glass, which absorbs violet light.

J. S. G. THOMAS.

Getter for vacuum devices. L. McCULLOCH, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,729,888, 1.10.29. Appl., 14.10.24).—For thermionic discharge devices a combination of calcium oxide and the product obtained by baking nickel and magnesium oxalates is used.

H. ROYAL-DAWSON.

Manufacture of electron-emitting material. W. B. GERO, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,731,244, 15.10.29. Appl., 29.6.26).—A mixture of pure powdered tungsten, molybdenum, and thorium is subjected to pressure. The intimate mixture of particles is made to coalesce by heating, while maintaining a high vacuum.

F. G. CLARKE.

Tungsten electrode for determining [hydrogen] ion concentration. J. R. BAYLIS, Assr. to LEEDS & NORTHRUP Co. (U.S.P. 1,727,094, 3.9.29. Appl., 16.7.24).—The electrode comprises an ordinary tungsten-filament lamp with a hole cut in the top for admission of the solution, the p_H of which is to be determined. The second electrode comprises a calomel half-cell, external to the lamp, having a capillary connexion tube passing just below the surface of the liquid in the lamp, the lead-in wires of which are connected to the usual electrical apparatus and galvanometer.

A. R. POWELL.

Manufacture of filament supports [for electric incandescence lamps]. C. J. SMITHELLS, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,733,076, 22.10.29. Appl., 25.4.27. U.K., 14.5.26).—See B.P. 275,735; B., 1927, 786.

Recovery of condensates from coal-distillation gases (B.P. 294,106).—See II. **Hydrogen cyanide** (U.S.P. 1,731,331). **Removal of halogens from salt solutions** (B.P. 301,512).—See VII. **Colouring glass** (B.P. 300,179).—See VIII. **Magnetic alloys** (B.P. 290,658). **Iron** (B.P. 306,151). **Chromium** (B.P. 320,440 and U.S.P. 1,730,349). **Purification of metals** (B.P. 320,818). **Insulating materials** (B.P. 320,370).—See XIV.

XII.—FATS; OILS; WAXES.

Detection and determination of carbon disulphide and sulphur in fluids [e.g., olive oil]. J. A. PIERCE (Ind. Eng. Chem. [Anal.], 1929, 1, 227—228).—Five c.c. of a solution of equal volumes of the oil to be examined and pure chloroform are placed in a test-tube, 2 c.c. of a reagent (freshly prepared by dissolving 1 g. of copper sulphate, nitrate, or chloride in a small amount of water, adding 4 c.c. of ammonia solution followed by 3 g. of hydroxylamine hydrochloride, and making up to 50 c.c. with water) are added, and the tube is stoppered and tilted to and fro for $\frac{1}{2}$ min. The reagent reacts with carbon disulphide instantly with the formation of an opaque, chocolate-coloured aqueous solution which quickly clears, and a heavy slimy precipitate of the same colour floats at the interface of the two liquids. The reaction is sensitive to solutions containing 0.0033% CS₂. With dissolved sulphur no preliminary colouring of the solution occurs, but a dense, deep black metallic-lustred precipitate of copper sulphide instantly forms. With oils containing both carbon disulphide and sulphur, microscopical examination shows the presence of a brown and a black precipitate both clearly distinguishable.

H. S. GARLICK.

Oil content of Malayan estate copra. C. D. V. GEORGI (Malayan Agric. J., 1929, 17, 335—340).—Copra obtained from six representative plantations lost on the average 6.9% of moisture when heated at 100° for 6 hrs. and the moisture-free material contained 65.6% of oil.

E. HOLMES.

Malayan lumbang oil. T. H. BARRY (J.S.C.I., 1929, 48, 289—290 T).—The examination of a recent sample of the Malayan lumbang oil (from *Aleurites moluccana*) indicates that the oil is equal to that obtained from other sources, and of similar chemical and physical properties. Preliminary experiments on its use in paints, however, are not promising owing to the softness of the film and the slowness of drying. It is shown that tung oil can be adulterated with nearly 20% of lumbang oil before the "Brown heat test" exceeds the maximum limit of the present American specifications for tung oil.

Composition of neem oil. So-called margosic acid. A. C. ROY and S. DUTT (J.S.C.I., 1929, 48, 333—335 T).—The fatty acids from neem oil are shown to contain no "margosic acid," which is proved to be a mixture of stearic, palmitic, oleic, linoleic, and small quantities of lower fatty and unsaturated resinous acids, together with a very small quantity of arachidic and lignoceric acids.

Fatty acids and glycerides of kusum oil. D. R. DHINGRA, T. P. HILDITCH, and J. R. VICKERY (J.S.C.I., 1929, 48, 281—286 T).—The properties and constitution of kusum (macassar) fat from the seeds of *Schleichera trijuga*, which grows abundantly in various parts of India, have been studied. The fat is semi-solid and yellow or cream in colour, and is accompanied by unsaponifiable matter and resinous compounds, the properties of which render its suitability for general edible purposes somewhat uncertain. The mixed fatty acids from the fat have setting point 50.5—51°, and are composed approximately as follows: oleic (60%), linoleic (3—4%), arachidic (20—25%), palmitic (5—8%), stearic (2—6%), and myristic acid (1%). These acids are apparently distributed more or less evenly in the form of mixed glycerides; fully-saturated glycerides (triarachidin) occur only in minute amounts, but there is probably some free triolein present. Arachidic acid is present in abnormally large quantities, and the oil is a suitable source of vegetable arachidic acid. The presence of arachidic acid in large proportions renders the soap from kusum fat somewhat hard and granular, although its lathering power is fair; the qualities of the soap are reminiscent of those of a hydrogenated fish oil. Employed in conjunction with other oils consisting largely of combined oleic acid, it should find use as a soap-making material. Of other acids which have been reputed to be present in the oil, lauric acid has been definitely shown to be absent, and no evidence of the presence of butyric acid is forthcoming; small amounts of acetic acid are present, but this acid is probably not in combination as glyceride, and arises from some of the non-fatty compounds which invariably accompany the fat.

Viscosity of glycerin solutions. L. V. COCKS (J.S.C.I., 1929, 48, 279—280 T).—Tables compiled from literature and experimental determinations are given

for the viscosity of glycerin solutions from 10% to 99.2% over a temperature range 1—100°. Attention is directed to similarity between the viscosities of 99.2% glycerin and castor oil. Small quantities of soap, represented by sodium oleate, have the effect of increasing the viscosity of glycerin solutions, the influence of the addition of quantities of the order 0.1% and 0.5% being similar on the average to increasing the glycerin concentration of 80% solution by 3.6% and 7.4% of glycerin, respectively. At higher concentrations of glycerin the effect of small amounts of soap is less marked.

Iodine values. Report of Committee of Society of Leather Trades' Chemists on Oils and Fats. IV. V. KUBELKA, J. WAGNER, and S. ZURAVLEV (J. Soc. Leather Trades' Chem., 1929, 13, 437—442; cf. B., 1929, 924).—The Hanus, Margosches, and Rosenmund-Kuhnemann methods of determining the iodine value have been used on olive, castor, arachis, and dark fish oils, oleic acid, and coconut oil fatty acids. The Hanus method proved the most useful and convenient. Difficulty was experienced with the Margosches method because the mixture of fat with alcohol had to be heated, and this affected the analytical results; also the result was dependent on the weight of fat used. In the other methods the result was unaffected by excess of fat where the iodine value was below 100, but with fats having iodine values above 100, the Rosenmund method proved unreliable with excess of fat. The Margosches method was very sensitive to the effect of increase in the time of reaction.

D. WOODROFFE.

Transformation of fatty acids. STADNIKOV and WEIZMANN.—See II.

PATENTS.

Production of [hard] soap. G. PETROV (U.S.P. 1,732,656, 22.10.29. Appl., 16.1.26. Ger., 21.12.25).—Drying and semi-drying oils are treated with a water-insoluble metal chromate in the presence of a mineral acid, other than chromic acid, capable of forming an insoluble salt with the metal radical of the chromate.

L. A. COLES.

Production of alkali soaps, including ammonium soap, and obtaining fatty acid therefrom. V. GRAUBNER (B.P. 320,331, 4.4.28).—The soap is made by passing a mixture in molecular proportions of fat, alkali, and water continuously in thin streams under pressure through a saponifying apparatus where it is in contact with a heated body, a zig-zag path being formed by the alternate arrangement of projecting baffle-ribs on the heated body and surrounding casing. The saponified mass is forced in a finely-divided state by means of a fine nozzle into a hot saturated salt solution to separate the glycerin from the "soap core"; alternatively, the soap may be distributed into a continuously acidified solution of the corresponding salt in order to separate the fatty acids.

E. LEWKOWITSCH.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Iodine value of turpentine oil. C. KRANZ, V. HRACH, and I. FRANTA (Chem. Obzor, 1928, 3, 365—367).—The maximum value, 370—380, is obtained by Hübl's

method when about 0.1 g. of oil is treated with about 0.4*N*-iodine solution. CHEMICAL ABSTRACTS.

Determination of the opacity [of pigments] by painting methods. F. SCHMID and K. WEISE (*Farben-Ztg.*, 1929, 35, 229—230).—The limitations of the method of comparing opacity of pigments by conversion into paint and ascertaining the weight required for obliteration of a striped undercoat are indicated. The standard undercoat for white paints recommended by the Reichsausschuss für Lieferbedingungen in this connexion comprises a central black stripe over a white background, the three stripes thus formed having equal width. This is criticised in that on such an undercoat there is a tendency to apply more white paint over the central stripe than over the remainder of the panel, and that the error due to losses at the edge are accentuated. A further source of error is the use of paints of equal consistency but containing different amounts of thinners, leading to inequality in the thickness of the paint coats which are being compared. In an improved undercoat (which should be flat) the edges of the panel are occupied by black stripes, and these preponderate in the striping scheme. Similar considerations apply to undercoats for other colours. With this improved type of undercoat the obliteration method yields results concordant with those attained by exact physical methods. S. S. WOOLF.

Low-temperature tar and the artificial resin industry. EHRMANN.—See II. **Viscosity of nitro-cellulose solutions.** PAM.—See V. **Malayan lumbang oil.** BARRY.—See XII.

PATENTS.

Manufacture of [fire- and water-proof] paint, adhesive, etc. R. J. CARRUTHERS (B.P. 320,735, 20.9.28).—Celluloid, preferably cuttings or waste (4 pts.), camphor, boric acid, sodium bicarbonate, and zinc chloride (1 pt. of each) are dispersed in 1 pt. of methylacetone, and the whole is thinned if desired with amyl acetate or other suitable thinner. Suitable dyes may be added. S. S. WOOLF.

Production of palette [“stopping”] materials. G. TRÜMLER (B.P. 294,262, 20.7.28. Ger., 21.7.27).—Quick-drying, non-porous, and non-clogging surfacing or stopping pastes for coating metal, wood, or other surfaces comprise solutions, preferably highly concentrated, of one or more cellulose esters and/or ethers, containing resins if desired, in which solutions one or more finely-divided metals or alloys, and, if desired, filling substances, *e.g.*, kieselguhr, mica, are incorporated. S. S. WOOLF.

Coating of surfaces. BRIT. THOMSON-HOUSTON Co., LTD., A. T. WARD, and G. R. R. BRAY (B.P. 320,649, 18.7.28).—A coating composition made from a solution of a phenol (preferably di- or poly-hydric, *e.g.*, resorcinol) in furfuraldehyde is applied to a surface and hardened by exposure to the action of a suitable catalyst, *e.g.*, gaseous hydrochloric acid. The addition of plasticisers, *e.g.*, tolyl phosphate and/or toughening agents, *e.g.*, cellulose acetate, improves the characteristics of the film. S. S. WOOLF.

Manufacture of a white-pigment base. A. E. MARSHALL, ASS. to MARYLAND PIGMENTS CORP. (U.S.P.

1,728,296, 17.9.29. Appl., 1.12.23).—Iron is removed from ilmenite sand containing silica by phosgene, the remaining silica and titania are fused with alkali, and the oxides are precipitated. H. ROYAL-DAWSON.

Manufacture of synthetic resins. G. T. MORGAN and H. A. HARRISON (B.P. 319,444, 23.7.28).—A condensed hydrocarbon of the type containing one or more unreduced benzene rings and also a five-membered hydrocarbon ring containing one or more methylene groups, *e.g.*, acenaphthene, fluorene, indene, or a monohalogen derivative of such hydrocarbon, is condensed with formaldehyde, or a substance yielding formaldehyde, in the presence of an acid catalyst (sulphuric acid). S. S. WOOLF.

Cellulose[—artificial] resin product. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of H. C. ROHLFS (B.P. 305,671, 8.2.29. U.S., 9.2.28).—Layers of cellulose material are cemented together by means of an “alkyd” (glyptal-type) resin in the intermediate (“B”) stage, and the composite material is subjected to pressure, *e.g.*, 1000 lb./in.², at a temperature sufficiently high to soften the resin, *e.g.*, 125—140°. These conditions are maintained for a sufficient time, *e.g.*, 14—16 hrs., to “cure” the resin without affecting the character of the sheet material. S. S. WOOLF.

Production of paint or waterproofing material. F. W. McRAE (B.P. 320,886, 19.7.28).—See U.S.P. 1,684,593; B., 1928, 903.

Synthetic resins and the like. NOBEL INDUSTRIES, LTD., Assees. of C. E. BURKE and H. H. HOPKINS (B.P. 289,784, 30.4.28. U.S., 28.4.27).—See U.S.P. 1,667,189; B., 1928, 457.

Grinding mill (B.P. 318,873).—See I. **Low-temperature tar and distillates** (B.P. 318,116).—See II. **Colour-binding agents** (B.P. 289,053).—See XVII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Preparation and properties of aqueous rubber dispersions. H. L. TRUMBULL (*Coll. Symp. Mon.*, 1928, 6, 215—224).—Aqueous dispersions of rubber, obtained by mastication with protective colloids, are free from pear-shaped particles and give a dried film of controllable properties. CHEMICAL ABSTRACTS.

Vulcanisation without sulphur. **Vulcanisation by heat or ultra-violet light with the aid of trinitrobenzene and picric acid.** F. KIRCHHOF (*Gummi-Ztg.*, 1929, 44, 252—253).—Contrary to its behaviour in vulcanisation by sulphur, litharge does not assist hot vulcanisation by trinitrobenzene or picric acid; lamp-black aids such vulcanisation, but the products are of poor mechanical quality and resemble regenerated rubber in physical character. Vulcanisation of rubber in ultra-violet light by such compounds, especially trinitrobenzene, yields better results comparable with those of cold vulcanisation; these products, like ordinary vulcanisates, also show strong absorption of ultra-violet radiation suggestive of reduced unsaturation. D. F. TWISS.

Ageing of rubber. B. V. BYZOV (*J. Appl. Chem.*, 1928, 1, 6—11).—Properly vulcanised rubber undergoes smaller changes on ageing than does uncured rubber.

The changes are probably due to a continuation of the vulcanising process at the temperature (*e.g.*, 70°) used in artificial ageing tests, and differ from those which occur in natural ageing.

CHEMICAL ABSTRACTS.

Carbon black [in rubber]. C. M. CARSON and L. B. SEBRELL (*Ind. Eng. Chem.*, 1929, **21**, 911—914).—The absorptive power of a carbon black for iodine is a measure of the rate of cure of a rubber mix, low absorptive power corresponding with rapid curing rate. The effect of heating blacks over the temperature range 500—1200° is to increase their absorptive power and, at the same time, to increase both the rate of cure of a rubber mix made from them and also the modulus of the product. The stiffening action of a carbon black may be estimated by measuring the amount of the substance which is retained in a thin rubber cement after centrifuging for a given time. Carbon blacks when boiled with sulphur and zinc oxide in xylene liberate a substance which serves to accelerate the rate of cure of a pure gum mix.

H. INGLESON.

PATENTS.

Production of articles substantially made of organic materials from aqueous dispersions containing such materials. DUNLOP RUBBER CO., LTD., G. W. TROBRIDGE, and E. A. MURPHY (B.P. 320,387, 12.4.28).—Substantially adhering fluid layers of an aqueous dispersion of organic materials, such as rubber, are applied to non-porous, inert surfaces by repeated dipping, spreading, and/or pouring; the setting and drying of each layer are effected by heat applied to the side of the layer adjacent to the deposition surface prior to the deposition of the next fluid layer.

D. F. TWISS.

Coating or plating apparatus. ANODE RUBBER CO., LTD., Assees. of G. F. WILSON (B.P. 298,650, 13.10.28. U.S., 13.10.27).—In order to prevent flocculation or agglomeration, *e.g.*, by frictionally sliding surfaces, in colloidal dispersions such as of rubber during the deposition of material such as rubber from colloidal dispersion on to a rotating article, the latter is so supported that the liquid is excluded from the rotating or moving parts.

D. F. TWISS.

Manufacture of artificial compositions, especially those resembling rubber. I. G. FARBENIND. A.-G. (B.P. 294,474, 6.7.28. Ger., 23.7.27).—Artificial compositions, especially such as resemble rubber, are obtained from organic esters or ethers of vinyl alcohol by polymerisation and incorporating therewith, before, during, or after polymerisation, such nitrogenous organic compounds and/or aromatic hydrocarbons as are solid at ordinary temperature. Polymerisation may be effected by known methods, *e.g.*, heating or irradiation, or a combination of such methods, in the presence or absence of accelerators, the resulting products being finally compounded and vulcanised as required.

D. F. TWISS.

Electrical insulating materials. W. S. SMITH, H. J. GARNETT, J. N. DEAN, B. J. HABGOOD, and H. C. CHANNON (B.P. 320,370, 4.7.28 and 28.3.29).—By hydrolytic treatment, *e.g.*, by the action of water at an elevated temperature, in the presence of glycerin or other hydroxy-compounds, the ordinarily water-insoluble impurities of raw rubber are rendered remov-

able by extraction with water. Heated glycerin effects a swelling of the rubber protein, and thereby aids the hydrolytic influence of the water. Alternatively, the raw rubber may be caused to absorb a solvent, *e.g.*, about 10% of benzene, and then be subjected to hydrolytic treatment with superheated water and steam.

D. F. TWISS.

Manufacture of synthetic rubber. R. P. DINSMORE, ASSR. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,732,795, 22.10.29. Appl., 13.9.27).—See B.P. 297,050; B., 1929, 829.

XV.—LEATHER; GLUE.

Pine bark and the manufacture of rich pine bark extracts. P. JAKIMOV (*Collegium*, 1929, 334—356).—The tannin content of pine bark varies from 6.5% to 11.5%, due to climatic conditions, method of harvesting, and mould attacks. Only the extract can be obtained of standard composition. Cold leaching of the bark extracts all the non-tans, but only 60—65% of tannin. The best extract was obtained by extracting pieces of bark, 2—3.5 mm. thick, at 95—96° in a battery of 8 leaches, allowing 2 hrs. in each. A crude, evaporated extract (*d* 1.180) contained tans 35.5%, non-tans 49.3%, sugars 11.3%, insoluble matter 15.2%, and tans: soluble matter 41.3%. This ratio was low, and was improved by subjecting the unground bark to a slow current of water for 24 hrs., pressing it to remove excess water, cutting it into small pieces, and leaching in the usual manner. The maximum amount of insoluble matter was rendered soluble by treatment of the improved extract for 20 hrs. at 96° with 35% (on the weight of insoluble matter) of a mixture of sodium sulphite and bisulphite. The solubilised extract contained tans 55.7%, non-tans 41.3%, and insoluble matter 2.98%. The greater content of non-tans in the untreated extract renders the viscosity higher, yields a greater amount of acid on fermentation, hinders the salting out of the tannin by sodium chloride, and adversely affects the speed of tannage with this material.

D. WOODROFFE.

Tannin analysis: Report of Committee of Society of Leather Trades' Chemists. J. G. PARKER (*J. Soc. Leather Trades' Chem.*, 1929, **13**, 412—421).—The Koch extractor is too small and frequently chokes; materials occasionally separate into two layers. The degree of concordance is bad and reinstatement of the Procter extractor is advocated. For the extraction 4 hrs. is ample time. Different results were obtained in the determination of the moisture by vacuum oven, electric oven, water oven, and combined evaporator and drier, respectively. The moisture should be determined by drying a small quantity of the powdered extract in a small weighing bottle in an oven at not above 100°. Lower percentages of total soluble matter were obtained by the filter-paper method than by use of the Berkefeld filter-candle, and the former method possesses no advantage in manipulative detail or accuracy over the latter. The filter-candles do not vary in degree of porosity beyond the limits of ordinary experimental error. The chroming of the hide powder with chrome alum was found to be highly satisfactory and better than the old method.

D. WOODROFFE.

Action of neutral salts on the enzyme activity of tryptic bates. I. V. KUBELKA and J. WAGNER (Collegium, 1929, 328—334).—The activity of tryptic enzymes on casein was increased by the addition of ammonium salts, and at low salt concentrations the increase was almost proportional to the salt concentration. Maximum activity was attained with an amount of ammonium salt 2—4 times that of standard bating material present. This corresponds to 33% of ammonium salt in a commercial bating material, and since this amount is usually exceeded, the enzyme activity of the bating material is always at its maximum, hence the author's method of analysis (B., 1929, 140, 755) is free from objection on this ground. With enzyme preparations which contain less than 33% of ammonium salt it will be necessary to add a further supply of the salt sufficient to bring the proportion present above 33% in testing them by the casein substrate method.

D. WOODROFFE.

Vegetable-tanned sole leathers. A. COLIN-RUSS (J. Soc. Leather Trades' Chem., 1929, 13, 443—462).—As a result of the analysis of a considerable number of such leathers it is suggested that they fall into three well-defined groups of chemical composition and quality with hide substance, water-soluble matter—hide substance ratio, leather substance, and ashed water-soluble matter, respectively, as follows: grade I $> 42\%$, < 0.3 , $> 72\%$, 0.9% ; grade II $42-32\%$, $0.3-0.9\%$, $72-56\%$, 1.65% ; grade III $< 32\%$, > 0.9 , $< 56\%$, 3.1% . All leathers should pass the thymol-blue test for free acid. Leathers containing less than 36% of hide substance are liable to spue. Photomicrographs are given of sections of a number of different grades and manufactures of sole leather. The quality of leathers of grade I is the best, and of grade III the worst.

D. WOODROFFE.

Effect of heat on wetted vegetable-tanned leathers. IV. W. J. CHATER (J. Soc. Leather Trades' Chem., 1929, 13, 427—437).—The effect of various buffer solutions on the shrinkage temperature of calf pelt has been determined, and also shrinkage curves for a number of insole leathers, and various other leathers including sulphur-tanned formaldehyde leather and several defective leathers. The initial shrinkage temperatures of chrome-tanned leathers stripped with Rochelle salt, $0.1N$ -sodium hydroxide, and pyridine, respectively, were 81° , 65° , and above 95° .

D. WOODROFFE.

PATENTS.

Tanning substances. J. R. GEIGY, SOC. ANON. (J. R. GEIGY, A.-G.) (B.P. 305,013, 12.9.28. Ger., 28.1.28).—Tanning agents are prepared by condensing in strongly acid solution water-soluble sulphonation products of phenol or its derivatives with formaldehyde or material yielding it, in the presence of urea, or material yielding urea, or of previously prepared urea-formaldehyde condensation products, and subsequently neutralising the products with alkalis.

L. A. COLES.

Tanning-material products. R. AISTRUP (B.P. 320,344, 7.7.28).—Extracts of tanning materials are concentrated in a triple-effect apparatus to a water content of 60—70%, then spray-dried to produce a

thread-like product of 1—5% water content which may be briquetted.

D. WOODROFFE.

Manufacture of a leather substitute. J. M. GONNISEN (B.P. 320,444, 31.7.28).—Celluloid waste, e.g., scrap film, is dissolved in acetone and ethyl acetate, kneaded together with leather dust, compressed, and dried.

D. WOODROFFE.

Production of tough transparent foils from gelatin. O. KLOTZ (B.P. 320,509, 24.9.28).—On a backing or an endless running band, gelatin and rubber latex are alternately applied to form a film of superimposed gelatin and rubber layers. The outer layers consist of gelatin. The product is dried, stripped off, and wound.

D. WOODROFFE.

Bleaching of skins (B.P. 318,471—2).—See VI. **Vegetable-glue base** (U.S.P. 1,726,824).—See XVII.

XVI.—AGRICULTURE.

Soil genetics. I. H. T. JONES and J. S. WILLCOX (J.S.C.I., 1929, 48, 304—308 r).—Work which has been carried out on seven Millstone Grit profiles from various parts of Yorkshire is described. The results of analyses of these profiles are given and evidence is adduced to show that the soils belong to the "podsol" class, the nature of which is explained. The various current theories of iron-pan formation are briefly discussed. Experiments are described on which a further explanation is based. It is shown that tartaric acid and oxalic acid extracts of soil deposit compounds of iron on keeping. It is suggested that sesquioxides are dissolved by soil organic acids, the iron and aluminium entering into the electronegative portion of the molecule; these compounds are then leached through the soil in solution and are ultimately precipitated as basic salts, thus giving rise to a zone of sesquioxide accumulation and finally to a pan.

Adsorbing soil complex and adsorbed cations as a basis for genetic classification of soils. K. K. GEDROIZ (Kolloidchem. Beih., 1929, 29, 149—260).—The following classification of soils is proposed. (1) Soils containing no hydrogen in the adsorbing complex (soils saturated with bases). These are divided into two sub-groups: (a) Soils which contain adsorbed calcium or magnesium in the adsorbing complex. The adsorbing complex is in the form of aggregates of the primary colloidal particles, the aggregates having, in general, a diameter greater than 0.1μ ; the aggregates are broken down, dissolved, or chemically decomposed by water only with difficulty. (b) Soils containing adsorbed sodium in addition to calcium and magnesium; the sodium salts may be present in an easily soluble excess or may be present in small amount strongly adsorbed; in this case the ability of water to break up the aggregates and to dissolve and chemically decompose the constituents of the complex is greater than for any other type of complex. (2) Soils containing adsorbed hydrogen in the adsorbing complex. In regard to the loosening and decomposing action of water, these complexes occupy an intermediate position between those containing only adsorbed calcium and magnesium and those partly or completely saturated with sodium.

E. S. HEDGES.

Hypothesis of "unfree water" in soils. A. N. PURI (Agric. J. India, 1929, 24, 318—324).—Four dry soils tested were unable to remove any water from sugar solutions of concentrations 4—8%. At higher concentrations there was evidence that a more dilute solution was taken up by the soil, leaving the sugar solution more concentrated, but it is considered unlikely that any "unfree water" existed in these soils.

E. HOLMES.

Reaction between soils and metallic iron. H. D. HOLLER (J. Washington Acad. Sci., 1929, 19, 371—378).—Evolution of hydrogen from equal weights of soil and steel turnings or pulverised cast iron in water serves as a convenient guide to the relative corrosiveness of different soils on iron. The amount of hydrogen evolved is not dependent on p_H alone, but if a soil has a low p_H value together with material capable of buffering the solution at this p_H , then a high rate of evolution may be expected. Clays and silt loams are therefore more active than sands. The activity of a soil on iron as measured by hydrogen evolution bears a direct relation to soil acidity as measured by titration. In the absence of air or oxygen, hydrogen evolution takes place up to p_H 9.4.

E. HOLMES.

Dispersing soils for mechanical analysis. A. N. PURI (Agric. J. India, 1929, 24, 330—331).—It is claimed that the dilute hydrochloric acid used for the preliminary treatment of soils for mechanical analysis, by the method recommended by the International Society of Soil Science, may be replaced with advantage by *N*-sodium chloride.

E. HOLMES.

Composition of commercial acid lead arsenate and its relation to arsenical injury. H. S. SWINGLE (J. Agric. Res., 1929, 39, 393—401).—At low concentrations (less than 0.01% As_2O_5) arsenic and arsenious acid solutions were equally injurious to peach foliage. Arsenic acid is the more injurious at the higher concentrations, the increased toxicity corresponding to p_H 2.6 in the solution. The minimum concentration of As_2O_5 harmful to peach leaves was 0.0012%. Acid lead arsenate containing less than 0.25% of soluble arsenic produced minimum leaf injury. Reduction of soluble arsenic in this substance sufficiently to prevent injury appears impossible. The initial concentration of soluble arsenic (within the limits of ordinary spray preparations) had little or no effect on the toxicity of lead arsenates to insects.

A. G. POLLARD.

Effect of external growth conditions on germination of cereals in sugar solutions. K. MEYER (J. Landw., 1929, 77, 97—138; cf. B., 1928, 421).—The germination of individual species of cereals varies with their origin, age, and method of storage. The significance of the suction power of seeds as a measure of their soundness is therefore restricted. It is necessary to differentiate between the osmotic power and germinative capacity of seeds in the application of these methods.

A. G. POLLARD.

Reduction of soil nitrates during the growth of soya beans. E. P. DEATRICK (J. Amer. Soc. Agron., 1928, 20, 947—958).—Nitrates in soils under maturing soya beans are very low. A long period for nitrification between harvesting and replanting is desirable.

CHEMICAL ABSTRACTS.

Sugar beet top silage. A. W. OLDERSHAW (J. Min. Agric., 1929, 36, 634—640).—Good silage may be made from sugar beet tops. Two analyses record: starch equivalent 5.6, 6.8; nutritive ratio 1:6.7, 1:7.7. The inclusion of adhering soil in the silo must be minimised. Pit- and trench-silos must be well drained, and clamp-silos made on the ground level should be covered with straw and banked with soil to a depth of 1 ft.

A. G. POLLARD.

Low-temperature tar as fungicide. EHRMANN.—See II. **Liquorice root.** HOUSEMAN and LACEY.—See XX.

PATENTS.

Manufacture of fertilisers containing ammonium nitrate. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 320,708, 7.8. and 21.9.28).—Finely-divided calcium carbonate, other than that obtained in the manufacture of ammonium sulphate from ammonium carbonate and calcium sulphate, is mixed intimately with fused ammonium nitrate, or with a hot, concentrated solution of it, and the product is solidified into granules by spraying or by compression between cooled rollers.

L. A. COLES.

Insecticide. Parasiticide. R. G. MEWBORNE, Assr. to CONSUMERS TOBACCO Co. (U.S.P. 1,731,677—8, 15.10.29. Appl., [A] 6.10.25, [B] 23.10.25. Renewed [A] 13.1.28, [B] 6.8.27).—(A) Tobacco is treated with an alkali in the presence of moisture, and with sufficient dehydrating agent to give a dry, readily pulverised product. (B) Tobacco is mixed with bentonite. The product yields free nicotine on mixing with water, and may be used as a dip.

F. G. CLARKE.

Insecticidal solution. A. W. C. MENZIES (U.S.P. 1,732,240, 22.10.29. Appl., 7.2.27).—An aqueous solution containing about 0.5% of sodium fluoride and about 0.2% of sodium taurocholate and sodium glycocholate is claimed.

H. ROYAL-DAWSON.

(A) **Extirpation of insects noxious to plants.** (B) **Insecticide.** E. VON AMMON and K. SZOMBATHY (B.P. 294,646—7, 10.7.28. Ger., 29.7.27).—(A) Insects which are protected by enveloping webs may satisfactorily be sprayed with mixtures of the alkaloid lupanine (0.3%), an alcohol such as amyl alcohol (1.5%), and an alkali, e.g., caustic potash (0.2%). (B) Insects which are normally protected by a layer of waxy secretion, particularly *Schizoneura lanigera*, may be killed by spraying with an emulsion containing lupanine (0.25%) and resinic salts and free resinic acid (1.2%) which dissolve the wax layer.

E. HOLMES.

Juices from plants (B.P. 320,369).—See XX.

XVII.—SUGARS; STARCHES; GUMS.

Colloids in molasses. A. VON BRODOVSKI (Kolloid-chem. Beih., 1929, 29, 261—353).—An investigation has been made of the physico-chemical properties of beet-sugar molasses, and the various colloidal constituents have been isolated and analysed. The surface tension of the solution of molasses at first falls with increasing concentration, then passes through a minimum, and thereafter rises. The surface tension is highest for neutral solutions. The relative viscosity of the solution of molasses is lower than that of a sucrose solution of

the same concentration, and little effect is produced by altering the hydrogen-ion concentration, although the viscosity rises in strongly alkaline solutions. The colour of the solutions deepens with increasing p_H . The colloid content of the molasses amounts to 1.07%, and the isolated colloids are divided into two groups according to whether they are soluble or insoluble in water (reversible or irreversible). The greater part of the colloidal material is insoluble in water, ether, alcohol, or benzene. In the irreversible part of the colloidal material can be distinguished three fractions with acid reaction and three fractions which behave amphotERICALLY. The reversible part consists of at least two substances, but it was not possible to fractionate it; about 25% consists of araban, and the presence of hexoses in the products of hydrolysis was established. The irreversible acid fractions are soluble in pyridine, whilst the amphoteric fractions and the water-soluble colloids are not. All the isolated fractions contain nitrogen (about 7.5–8.7% in the irreversible colloids and 4% in the reversible colloids). The irreversible colloids are also richer in carbon. Phosphorus is also present. One of the acid fractions appears to be similar to the fuscazinic acid isolated by von Staněk (cf. B., 1917, 935); it is tri- or tetra-basic. It has not been possible to recognise the presence of albumins, and it is suggested that these may have suffered degradation during the manufacturing process. Salts of most heavy metals precipitate the colloids, but mercuric chloride is a notable exception; the salts of the alkaline earths have only a small precipitating power. In feebly acid solution the reversible colloids lower the surface tension of water more than do the irreversible colloids, but the reverse is the case in weakly alkaline solutions; the viscosity of the reversible colloids is greater than that of the irreversible colloids. The colouring matter in molasses is in the irreversible colloidal part. When the isolated colloids are added to sucrose solutions the surface tension of the latter is reduced, but practically no effect is produced on the viscosity.

E. S. HEDGES.

Advantageous zone of p_H for [beet sugar] refinery liquors. M. I. NAKHMANOVICH (Nauch. Zapiski Sak. Prom., 1929, 7, 265–281).—Filtration is best effected at p_H 7.0–7.5. Destruction of sugar begins below p_H 7.0, whilst colour is developed by heating at p_H 8.

CHEMICAL ABSTRACTS.

Automatic p_H recorders for sugar-refinery alkalinity control. A. L. HOLVEN (Ind. Eng. Chem., 1929, 21, 965–970).—Various methods for the automatic determination of p_H have been investigated in order to control the addition of lime in the refining of sugar products, but none has proved entirely satisfactory. The quinhydrone, the antimony, and the tungsten-manganese sesquioxide electrodes were found to be unreliable, owing to failure to hold the calibration (cf. Parker, B., 1927, 634; 1928, 627). The bare-wire tungsten electrode in conjunction with a calomel half-cell (cf. Balch, B., 1928, 461; 1929, 32) proved more suitable, but required elaborate compensatory devices owing to the variations of temperature, viscosity, density, and chemical character of the products to be tested. The temperature coefficient of the electrode is about 2 millivolts, and was compensated by a

thermopile of 50 copper-constantan couples giving the same current but in opposition to the electrode. The difference in the readings for two different products at the same p_H was found to be constant, so that the zero of the electrode could be adjusted for each product by means of a cell and potentiometer. Screening of the liquors also presented difficulty as the electrode is very susceptible to poisoning. It is concluded that, although more sensitive, the method offers no advantage over the colorimetric method.

H. J. DOWDEN.

Application of the Selivanov reaction [for detection of lævulose]. S. MALOWAN (Chem.-Ztg., 1929, 53, 800; cf. Ofner, B., 1929, 832).—The colour given by lævulose in the Selivanov test is attributed not to the sugar itself but to its aldehyde and ketone decomposition products; the coloured compounds are in the nature of xanthene derivatives.

A. R. POWELL.

Acetic and lactic acids. ALLGEIER and others.—See XVIII. **Sodium glutamate.** HAN.—See XIX.

PATENTS.

Sugar manufacture. D. STEWART & CO., LTD., and J. B. TALBOT-CROSBIE (B.P. 320,826, 14.1.29).—In the diffusion process where carbonation is employed, the liquor from exhausted pulp is mixed with press cake, thereby reducing acidity of the liquor, then filtered, and used again at any suitable stage in the diffusion.

E. B. HUGHES.

Recovery of sucrose from cane molasses. H. DE F. OLIVARIUS, Assr. to CALIFORNIA PACKING CORP. (U.S.P. 1,730,473, 8.10.29. Appl., 22.12.25. Renewed 25.2.29).—Invert sugar present is eliminated by fermentation, an alkaline-earth oxide or hydroxide and alcohol are added to precipitate organic impurities, and the liquor is filtered. Sucrose is then precipitated by means of an alkaline-earth oxide or hydroxide, after distilling off the alcohol.

F. G. CLARKE.

Treatment of starch. INTERNAT. PATENTS DEVELOPMENT Co. (B.P. 294,979, 3.7.28. U.S., 3.8.27).—Modified starches (from thin-boiling starch to highly-soluble dextrin) are prepared by drying to 2% of moisture, then treating with chlorine gas (0.03–0.12% of the weight of starch) at 115–135° for at least 2 hrs.

E. B. HUGHES.

Manufacture of dry colour-binding agents soluble in cold water. HENKEL & CIE., G.M.B.H. (B.P. 289,053, 12.4.28. Ger., 21.4.27).—Gelatinated starch preparations soluble in cold water, obtained by heating starch with water or by treating it with sodium hydroxide, with magnesium, zinc, or calcium chloride, or with ammonium thiocyanate, are slightly disintegrated by treatment with, e.g., hydrogen peroxide, per-salts, hypochlorites, or chloroamines, in the presence of alcohol as diluent, and the products are dried and ground. Alternatively, disintegration can be effected before or during gelatinisation.

L. A. COLES.

Vegetable-glue base. E. H. HARVEY, Assr. to PERKINS GLUE Co. (U.S.P. 1,726,824, 3.9.29. Appl., 26.1.23).—Dry starch substantially free from gluten is mixed with not more than 5% of a non-hygroscopic oxidising agent, e.g., barium dioxide, which does not convert the starch on admixture, but renders the finished glue suitably fluid on addition of water or caustic alkali solution.

F. R. ENNOS.

Carbohydrate esters or ethers (B.P. 293,316 and 293,757).—See V. Yeast (U.S.P. 1,732,922).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Nitrogen requirements of yeast. A. A. D. COMRIE (J. Inst. Brew., 1929, 35, 541—547).—The author has reviewed the data published on the nitrogenous nutrients of yeast. From 0.025—0.026% of the total nitrogen is assimilated by yeast during fermentation of wort under brewery conditions. Data relating to the most propitious state of combination of the nitrogen are contradictory, and although leucine, asparagine, and ammonium sulphate are quoted as substances favourable to yeast, on occasion, they are condemned by other workers as detrimental to fermentation. Substances in which the nitrogen is not combined either as amino-nitrogen or ammonia-nitrogen, such as alkaloids or pyridine, are of no value. Protein, nitrates (other than ammonium), and, according to one authority, acid amides, excepting formamide and those of a complex nature, are non-assimilable. Peptones, aliphatic amines, aromatic amines with side-chain nitrogen, vitamins, diastase, and amino-acids, in general, are stated to be assimilable. The examination of the published work has also yielded a great number of compounds of known chemical constitution with possible nutrient properties which have been classified according to their assimilability by yeast and their action on fermentation.

C. RANKEN.

Methylene-blue studies [in the staining of yeast]. J. FUCHS (Woch. Brau., 1929, 46, 437—440).—By observation of individual cells of yeasts 8—30 days old in water suspension it was noted that certain cells remained unstained for as much as 30 min. and then stained dark blue in a few seconds. In other cases staining occurred gradually after several hours. These two types of resistant cell were characterised by bright plasma and well-defined vacuoles. The proportion of cells stained at once by methylene-blue in concentrations of 0.001, 0.0002, and 0.0001% differs little, but the difference increases and is marked at 15 min. Stained cells may become decolorised on keeping owing to reductase action, and this is hastened by slight acid reaction of the medium. Certain weakly stained cells are capable of reproduction; these are considered to be stained only on the surface. In testing yeast a 0.0001% solution of methylene-blue should be employed, and only sufficient added to impart a greenish-blue colour to the suspending medium. Only deeply stained cells should be considered as dead (cf. B., 1929, 792).

F. E. DAY.

Brewers' pitch [for lining casks]. F. KUTTER (Woch. Brau., 1929, 46, 357—361, 372—376).—A cask-lining composition was heated at 170° and 200° under various conditions of aeration. The loss in weight was noted at intervals, also the softening and melting points as determined by the Hoffmann-Herbst and Klinger-Lampe methods, and the viscosity. At 170° in a closed or open vessel without aeration the physical constants were little changed, with 0.6 litre of air per hr. there was comparatively slight change, but with 1.2 litres/hr. a decided raising of the m.p. and viscosity took place after 67 hrs. This was much more marked

with 1.0 litre/hr. of air at 200°, the m.p. (Hoffmann-Herbst) rising from 50.7° to 76.5°, and the viscosity at 140° from 1.80 to 23.25 (Engler). Correspondingly the cooled material became darker, harder, and more brittle. In the chemical examination the neutral and acid constituents were determined (cf. B., 1929, 34), and in place of the "oxidation value" a bromine value was determined by treating 0.7 g. of the material dissolved in 10 c.c. of glacial acetic acid for 15 min. at 70° with 27—30 c.c. of a solution (N) of bromine in glacial acetic acid. The reaction flask was closed with a ground-in two-bulbed absorption funnel containing 8 c.c. of 0.5N-potassium iodide. This solution prevented escape of bromine, and on cooling it was drawn back into the flask; the iodine liberated by the excess of bromine was titrated with 0.5N-thiosulphate. The bromine value was found to fall during heating with aeration in a manner corresponding to the changes in the physical properties. The proportions of neutral and acid constituents showed more complex changes, the latter at first decreasing, then increasing in amount. It is suggested that this is possibly due to loss of volatile acids in the early stages, followed by oxidation of neutral to acid substances later, accompanied by rise of m.p. and viscosity. An apparatus suitable for carrying out the hot aeration test is figured.

F. E. DAY.

Mechanism of enzyme action and its action in breadmaking. F. F. NORD (Z. angew. Chem., 1929, 42, 1022—1025).—Various theories of the mechanism of the action of yeast-cell enzymes on hexoses are discussed. From experiments on the action of yeast on dough under various physical conditions, a theory of the mechanism of enzyme action is developed based on the equilibrium at and adsorption on the yeast-cell wall. This theory serves to explain the observed phenomena of stimulation and depression of fermentation.

A. A. GOLDBERG.

Production of acetic and lactic acids [by fermentation] from mill sawdust. R. J. ALLGELER, W. H. PETERSON, and E. B. FRED (Ind. Eng. Chem., 1929, 21, 1039—1042).—Fir, spruce, and pine sawdust hydrolysed by sulphuric acid gives wood-sugar liquors fermentable by a unique lactic acid organism capable of fermenting both pentoses and hexoses (cf. Marten and co-workers, B., 1927, 889). Fermentation is carried out in presence of 5% (wt./vol.) of malt sprouts as a source of nitrogen and excess calcium carbonate, which may be added at the commencement or at intervals, as neutralising agent. The presence of added colloids or phosphates has no accelerating effect on the fermentation. Liquors of 7—10% concentration of sugar are fermented in 7—9 days with destruction of about 85% of the sugar, which is almost quantitatively recovered as products. Dilution of solutions aids fermentation. The mixed acid obtained contains, in small- and large-scale experiments, respectively, about 10 and 5% of acetic and about 90 and 95% of inactive lactic acid. Soft-wood liquors are more readily fermented than those from hard woods. The dextrose tetrameride produced by the Rheinau process (cf. J.S.C.I., 1926, 45, 267T) after first hydrolysing with water is as readily fermented as are the liquors from the sulphuric acid process, and yields similar products.

D. W. HILL.

Recalculation of the densities of water-alcohol mixtures from the experimental data of Mendeléev. A. N. GEORGIEVSKI (J. Appl. Chem., Russia, 1928, 1, 207—219).

Densities of water-alcohol mixtures. B. M. KOYALOVICH (J. Appl. Chem., Russia, 1928, 1, 219—260; cf. also Vredski, *ibid.*, 1928, 1, 160).

PATENTS.

Production of yeast. J. R. WHITE, ASSR. to H. LEEDS (U.S.P. 1,727,847, 10.9.29. Appl., 29.4.27. Renewed 31.1.29).—Seed yeast is successively propagated in a series of sacchariferous solutions, each containing 0.5—5% of sodium chloride.

H. ROYAL-DAWSON.

Manufacture of diastatic composition. J. TAKAMINE, JUN., ASSR. to TAKAMINE FERMENT CO. (U.S.P. 1,731,400, 15.10.29. Appl., 20.8.18).—The composition is precipitated from a diastatic solution containing sodium bicarbonate, by means of magnesium chloride.

F. G. CLARKE.

Cooling of worts during fermentation. F. E. LICHTENTHAELER (U.S.P. 1,731,073, 8.10.29. Appl., 14.3.25).—The carbon dioxide produced during fermentation is drawn off, compressed, cooled, and passed again into the wort.

A. COUSEN.

Aerobic fermentation. G. S. BRATTON, ASSR. to ANHEUSER-BUSCH, INC. (U.S.P. 1,732,921, 22.10.29. Appl., 6.3.26).—A stream of air is circulated over and over again through the fermenting material, and the loss of oxygen is compensated by replacing part of the escaping exhaust air by an equal quantity of fresh air.

C. RANKEN.

Production of citric acid by fermentation. MONTAN U. INDUSTRIALWERKE VORM. J. D. STARCK (B.P. 302,338, 7.9.28. Czechoslov., 16.12.27).—Mashes such as are used in the production of alcohol are fermented by moulds which have been cultivated on solid vegetable culture media containing citric or tannic acid and vitamins. Nutrients which include a certain proportion of organic amino-acids are added in amount so that the growth of the moulds is restricted to a minimum necessary for the optimum conversion of sugar into citric acid. Destruction of the citric acid is prevented by "protective" substances, *e.g.*, hydrolysed glue, which unite with the acid to form complexes of high mol. wt. and which do not readily penetrate the cells of the fungi.

C. RANKEN.

Manufacture of yeast. R. HAMBURGER, S. KAESZ, and F. HARTIG, ASSRS. to STANDARD BRANDS, INC. (U.S.P. 1,733,962, 29.10.29. Appl., 11.11.24. Ger., 3.3.24).—See B.P. 230,050; B., 1924, 896.

Sucrose from cane molasses (U.S.P. 1,730,473).—See XVII.

XIX.—FOODS.

Determination of carotin in flour. C. G. FERRARI and C. H. BAILEY (Cereal Chem., 1929, 6, 347—371; cf. Coleman and Christie, B., 1927, 396).—The carotin content of flour is determined by spectrophotometric examination of a gasoline extract of the flour. The extract should be perfectly clear or clarified by filtration through selected alundum thimbles or by means of a

capillary siphon. Filter paper and unglazed porcelain adsorb carotin. The amount of carotin extracted by gasoline from flour depends on the ratio of solvent to sample; for 100 c.c. and 20 g. about 10% of the carotin remains unextracted. Results are given for the carotin content of some varieties of Canadian wheat and also for the distribution of carotin in the various fractions of flour in milling.

E. B. HUGHES.

Loaf volume as produced by different flours under prolonged fermentation. R. J. CLARK (Cereal Chem., 1929, 6, 338—344).—An extension of the experimental baking test on flour is described in which a series of doughs made from the same flour are fermented for varying periods. In curves connecting loaf volume and fermentation period, a major peak is obtained corresponding with the best-quality loaf; certain minor peaks also appear, the significance of which is discussed.

E. B. HUGHES.

Composition of milk. O. R. OVERMAN, F. P. SAMMANN, and K. E. WRIGHT (Univ. Ill. Agric. Exp. Sta. Bull., 1929, No. 325, 174 pp.).—A statistical study of results obtained with 1998 samples shows that the ratio protein:fat is higher than is usually reported, and that the lactose is nearly constant up to 5% of fat and decreases for higher percentages of fat.

CHEMICAL ABSTRACTS.

Influence of acidity on the coagulation of milk. C. PORCHER (Rev. gén. Colloid., 1929, 7, 260—270).—The times of clotting of milk with different rennins are studied under varying conditions of acidity with hydrochloric, acetic, and lactic acids. Carbon dioxide assists clot formation by forming hydrogen ions and by its effect on the solubility of the calcium salts present in the milk, the increase in the concentration of hydrogen and calcium ions favouring coagulation; in this way it is possible to coagulate "lazy," bad, or alkaline milks.

F. R. ENNOS.

Salts in milk. Heat coagulation of evaporated milk. H. H. SOMMER (Butter Cheese J., 1929, 20, No. 11, 44—48).—The effect of salts present in milk on its coagulation by heat is discussed. The amounts of disodium hydrogen phosphate or sodium citrate necessary to remove excess of calcium do not exceed 0.007% (as P_2O_5) and 0.092% (as citric acid), respectively. The effect of sodium hydrogen carbonate is to convert sodium dihydrogen into disodium hydrogen phosphate.

CHEMICAL ABSTRACTS.

Volatile constituents of "miso." R. TAKATA (J. Soc. Chem. Ind. Japan, 1929, 32, 628—629).—The average percentages of the volatile constituents of "miso" are: total volatile material (chiefly water) 49.38, alcohol 1.38, carbon dioxide 0.034, acetone 0.013, acetic acid 0.011, and volatile esters 0.039%. The sweet varieties of "miso" contain larger amounts of alcohol, acetic acid, and esters than the salty varieties, but the amount of acetone increases as the ripening of "miso" proceeds.

K. KASHIMA.

Dried fruit grubs. Ethylene dichloride-carbon tetrachloride fumigation process. J. E. THOMAS (J. Council Sci. Ind. Res., Australia, 1929, 2, 128—133).—A mixture of ethylene dichloride (3 vols.) and carbon tetrachloride (1 vol.) used at the rate of 14 lb. per 1000

cub. ft. at temperatures above 21° for not less than 22 hrs. in airtight compartments sterilises dried fruit as regards *Plodia interpunctella* and presumably also as regards *Ephestia cautella*.
W. J. BOYD.

Low-temperature preservation of foodstuffs. T. MORAN (J.S.C.I., 1929, 48, 245—251 T).—The part played by temperature in the preservation of foodstuffs is reviewed. With living foodstuffs recent work has emphasised the importance of a limited temperature range, and in turn the concept of an optimum temperature for storage. With dead foodstuffs temperature is important in restricting the activities of moulds, bacteria, and enzymes. An account of recent work on the freezing of tissues is also given.

Monosodium glutamate as chemical condiment. J. E. S. HAN (Ind. Eng. Chem., 1929, 21, 984—987).—Monosodium glutamate has a decided meat-like taste and is used largely for flavouring vegetarian dishes. For the cheap commercial production of this condiment vegetable materials such as gluten, soya bean, and beetroot molasses are hydrolysed by hydrochloric acid. Separation of the products may be effected by electrolysis in a three-chambered cell. Amphoteric and neutral products collect in the centre, whilst alkaline and acid products (*e.g.*, glutamic and aspartic acids) collect in the respective outer compartments, which also contain the electrodes. In the usual method for manufacturing the monosodium salt, gluten obtained from Canadian wheat and concentrated hydrochloric acid are heated in stone vessels fitted with reflux condensers until the protein has dissolved, whereon the temperature is raised to boiling and maintained until hydrolysis is complete. The acid is then neutralised with soda and the glutamate is crystallised, dried, and powdered. In an improved method the protein is dissolved in hydrochloric acid at a temperature below the coagulating point, metallic tin is introduced to catalyse the hydrolysis and to remove arsenic, and the glutamate is separated by precipitation with alcohol. The neutralised glutamic acid hydrochloride, when evaporated, gives a product containing 26% of sodium chloride, and to avoid this contamination the neutralised solution is concentrated until most of the sodium chloride has crystallised, or, alternatively, the alkali equivalent of the hydrochloric group is added, whereon the sparingly soluble glutamic acid crystallises.
H. J. DOWDEN.

Carbon dioxide in [mineral] water. ANON.—See VII. **Enzyme action and breadmaking.** NORD.—See XVIII. **Liquorice root.** HOUSEMAN and LACEY.—See XX.

PATENTS.

Manufacture of cereal meals. W. N. BOYES (B.P. 310,372, 27.3.29. N.Z., 24.4.28).—Cereal meals requiring no cooking are prepared from oats, wheat, etc. by treatment of the clean grain in a digester with steam at 6—15 lb. for about 2 hrs., cooling to 15·5° to condition, and then adding about 6% of sodium chloride; the product is dried, baked at about 60°, and again conditioned.
E. B. HUGHES.

Preparation of milk, skimmed milk, or butter-milk, free of milk sugar [lactose] for sufferers

from diabetes. J. POHLMANN and J. R. F. RASSERS (B.P. 320,497, 13.9.28).—The casein is precipitated by slight acidification with the addition also of a small quantity of a decoction of carrageen. The precipitate is washed free from lactose, suitable mineral salts are added, and blood-serum (*e.g.*, of the cow) and water are added to bring the whole into suspension. Cream washed with water may also be added.

E. B. HUGHES.

Treatment of sour cream. Treatment of soured or curdled milks for recovery of butter fat therefrom. A. L. RUSHTON, M. M. SIMPSON, and H. C. BECKMAN, ASSRS. to CREAM PROCESSES, INC. (U.S.P. 1,731,868—9, 15.10.29. Appl., [A] 27.2.28, [B] 6.6.28).—The cream or milk is rendered alkaline by alkali hydroxide and the fat is then separated.
E. B. HUGHES.

Preservation of peanut butter. J. H. BEATTIE and A. M. JACKSON (U.S.P. 1,731,647, 15.10.29. Appl., 18.5.28).—To prevent separation of oil, peanut butter is sealed in airtight containers and subjected to the action of steam under pressure.
E. B. HUGHES.

Cleansing and preparing for storage and market fruit after harvesting. I. F. LAUCKS, H. P. BANKS, and H. F. RIPPEY, ASSRS. to LAUCKS LABS., INC. (U.S.P. 1,732,816, 22.10.29. Appl., 11.10.26).—Fruit bearing residue from spraying is pretreated with trisodium phosphate solution.
E. B. HUGHES.

Production of [concentrated] non-alcoholic fruit juices. G. SCHICHT A.-G. (B.P. 299,043, 9.10.28. Czechoslov., 21.10.27).—Fruit juices are sterilised and clarified by sterilising filters and then concentrated in high vacuum at temperatures not affecting colour, taste, or vitamins.
E. B. HUGHES.

Preparation of fresh cocoa beans. T. ZELLER (B.P. 304,181, 29.6.28. Ger., 16.1.28).—Cacao beans are prepared in one operation by arresting the usual alcoholic fermentation before the commencement of acetous fermentation by heating for 24—30 hrs. at a temperature (optimum range 55—65°) high enough to destroy yeasts, bacteria, and moulds, and to kill the bean entirely, whilst not high enough to destroy the enzymes. The pulp is then removed by washing, after which the beans are left for 24 hrs. at 60° in moist air and then dried at 55—65°.
E. B. HUGHES.

Refrigeration of products such as foodstuffs. J. E. W. REEH (B.P. 300,209, 8.11.28. Fr., 8.11.27).

[Brine tank] freezing of fish. GOVERNOR & COMPANY OF ADVENTURERS OF ENGLAND TRADING INTO HUDSON'S BAY, and C. TOWNSEND (B.P. 320,578 and 321,117, 16.1.29).

Carbon dioxide developing powder for foods, *e.g.*, baking-powder (B.P. 303,353).—See VII. **Juices rich in vitamins** (B.P. 320,369).—See XX.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Arsenious iodide and its solutions. T. T. COCKING (Quart. J. Pharm., 1929, 2, 409—410).—An aqueous solution of arsenious and mercuric iodides (Donovan's solution), after keeping for 14 months in a well-stoppered vessel and being tested at intervals,

contained no arsenic in the arsenious state. The solution should either be freshly prepared before use or stored out of contact with air. E. H. SHARPLES.

Stability of extracts of ergot. F. WOKES (Quart. J. Pharm., 1929, 2, 384—395).—Effects of variations in the conditions of preparation and of storage on the deterioration of ergot extracts have been studied. Under the best conditions half the activity of liquid extracts is lost in from two to three months. This loss occurs in extracts made with hydrochloric acid (cf. Prybill and Maurer, B., 1928, 912), or with citric or tartaric acid, and with alcohol contents varying from 35 to 75%. The rate of deterioration is about twice as rapid at room temperature as at 0°, and still more rapid at 37°. Concentrated (soft) extracts may retain half their activity after storage for 4—9 months at 0°. E. H. SHARPLES.

Determination of "free nicotine" in tobacco: apparent dissociation constants of nicotine. H. B. VICKERY and G. W. PUCHER (J. Biol. Chem., 1929, 84, 233—241).—The fact that part of the nicotine of tobacco is "free" (*i.e.*, can be removed by steam-distillation without addition of alkali) is due to hydrolysis of the nicotine salts, and the amount of such free nicotine depends therefore on the p_H of the sample. The electrometric titration curve of nicotine is given, from which the proportion of the total nicotine which is free in a tobacco extract of known p_H can be deduced.

C. R. HARRINGTON.

Liquorice root in industry. P. A. HOUSEMAN and H. T. LACEY (Ind. Eng. Chem., 1929, 21, 915—917).—Glycyrrhizin is contained in the roots of the plant, which after being dug are cured in the sun and baled. In the extraction the roots are shredded and subjected to the action of water under low-pressure steam. The extract is settled in cascade tanks, the liquor being concentrated under vacuum to d 1.14. This solution is then either treated in drum dryers for the preparation of powdered liquorice, or is further concentrated to a paste containing 20—25% of moisture; on cooling it sets to the characteristic semi-plastic mass. The extract may also be treated with dilute sulphuric acid in slight excess, the precipitated glycyrrhizic acid being washed, dissolved in ammonia, and dried for sale as ammoniated glycyrrhizin. A typical Spanish liquorice mass contains glycyrrhizin 10%, sugars 12%, starch and gums 25%, moisture 15—25%, and ash 5%, whilst Oriental liquorice contains 18% of glycyrrhizin. The bulk of the liquorice is used in the tobacco industry, the remainder being converted into pharmaceutical and confectionery products. Part of the spent roots are subjected to further extraction with 5% caustic soda at 100 lb. steam pressure, and this extract is used as a foam stabiliser in fire extinguishers, as a foaming agent in froth flotation, and as a spreader for insecticides. The tough fibrous residue is utilised in the manufacture of boxboard and wallboard, the latter possessing low thermal conductivity, high water-resistance, and excellent mechanical properties.

H. J. DOWDEN.

Determination of glycyrrhizic acid in liquorice root and extract. R. EDER and A. SACK (Pharm. Acta Helv., 1928, 4, 23—48; Chem. Zentr., 1929,

i, 3017).—Methods are reviewed, and details of the determination are given. A. A. ELDRIDGE.

Examination of medicinal substances containing anthraglucosides. P. VAN DER WIELEN (Pharm. Weekblad, 1929, 66, 877—879).—The proposals for determination of hydroxyanthraquinone compounds in pharmaceutical preparations are discussed, and a standardised colorimetric method is described.

S. I. LEVY.

Boiling points of binary mixtures of ethereal oils. E. BRAUER (Ber. Schimmel, 1929, 151—161).—The b.p./10 mm. of a large number of binary mixtures of various composition of ethereal oils have been determined. In all the cases studied, with exception of the pairs cineole-*p*-cymene, and menthenone-safrole, the b.p. of the binary mixture deviates from the value calculated by the method of mixtures (Δt). Maxima are found in the following pairs, the figure in parentheses being the mol.-% of the first component: carvacrol-carvone (70), carvacrol-menthenone (70.2), carvacrol-pulegone (70.2), cineole-phenol (13.2), and menthenone-thymol (49.6), the b.p. being, respectively, 116.5°, 119.1°, 115°, 74.2°, and 116.5°/10 mm. Minima occur with the pairs: benzyl alcohol-safrole (93.1), citronellol-eugenol (90.4), citronellol-safrole (30.8), *p*-cymene-phenol (62.1), and safrole-terpineol (9.6), the b.p. being, respectively, 91.7°, 107.1°, 102.3°, 55.8°, and 96.4°/10 mm. The greater the difference in the b.p. of the two components the greater is the possibility of the existence of maxima and minima, providing the difference is not too great, since the pair *p*-cymene-terpineol which exhibits the largest deviation (Δt 17.3°, b.p. 60°/10 mm., for a mixture containing 53.5 mol.-% of *p*-cymene) shows no minimum on the curve. Menthenone has b.p. 103°/10 mm. (lit. 98°) and terpineol, b.p. 96°/10 mm. (lit. 104°).

J. W. BAKER.

Terpene- and sesquiterpene-free ethereal oils. T. KLOPPER (Ber. Schimmel, 1929, 167—178).—Various physical constants, solubility data in aqueous alcohol, and saponification and esterification values of the terpene- and sesquiterpene-free fractions of a number of essential oils have been determined and compared with the corresponding values of the natural oils. The values of d_{45}^{25} and α_D (? in 1-dm. tube) for such fractions of the following oils are, respectively: Japanese valerian oil 0.990, -20°; Réunion basil oil 0.980, +1.5°; birch-bud oil 0.980, -13°; tarragon oil 0.9754, slightly dextrorotatory; fennel oil 0.985, +3° (solidification point +17°); galbanum oil 0.985, slightly laevorotatory; helichrysum oil, 0.9294, +0.9°; Roman camomile oil 0.914, slightly dextrorotatory; pine-needle oil 0.960, -20°; niaouli oil 0.937, slightly laevorotatory; origanum oil 0.937, -6°; parsley-seed oil 1.125, 0°; American tansy oil 0.932, +38°; West Indian sandalwood oil 0.985, +4°; sassafras wood oil 1.095, slightly dextrorotatory; star anise oil 0.982, slightly laevorotatory; wine-yeast oil 0.879, slightly dextrorotatory.

J. W. BAKER.

Testing of blossom extracts. I. H. WALBAUM and A. ROSENTHAL (Ber. Schimmel, 1929, 187—202).—Various physical and chemical methods for the detection of impurities in commercial blossom extracts are

described and the mean values of the various physical constants of characteristic samples are tabulated. Solid extracts (extracted with light petroleum) are best characterised by their solidification points and their saponification values, the following being, respectively, the mean values of these constants for various essences: *jasmin* 49.8°, 106.2; *Bulgarian rose* 44.4°, 37.3 (the presence of light petroleum lowers both values); *orange blossom* —, 97.07; *lavender* 15.4°, 111. Fluid extracts (extracted with alcohol) are best characterised by their saponification values, density, and solubility in dilute alcohol. Adulteration with ethyl phthalate is determined by hydrolysis with 0.5*N*-alcoholic potassium hydroxide, filtering, drying at 110°, and weighing the insoluble potassium phthalate; this weight multiplied by 0.917 gives the weight of ethyl phthalate in the weight of extract taken. A reflux apparatus is described for the determination of waxes, resins, etc. in extracts, in which the whole of the essential oil from a known weight of extract mixed with salt is steam-distilled, using a controlled air-bath at 182° (boiling tetralin), and continuously extracted with ether. The weight of residue from the dried ethereal extract is the amount of pure essential oil present. For detailed constants of various extracts the original must be consulted.

J. W. BAKER.

Japanese peppermint oil. H. WALBAUM and A. ROSENTHAL (Ber. Schimmel, 1929, 203—207).—The presence of Δ^{β} -hexenol, b.p. 55—56°/9 mm., d^{15}_4 0.8508, n^{20}_D 1.48030, in Japanese peppermint oil (cf. A., 1918, i, 302) is confirmed by its catalytic reduction with nickel and hydrogen to *n*-hexyl alcohol. Hydrolysis of the ester fraction of high b.p. yields phenylacetic acid, which is separated from liquid acids by centrifuging. Fractionation of the latter gives Δ^{α} -hexenoic acid, b.p. 98°/5 mm., m.p. 34—35°, identical with a specimen synthesised by condensation of *n*-butaldehyde with malonic acid in the presence of pyridine and purification through its ethyl ester, and is converted into $\alpha\beta$ -dibromohexenoic acid by the action of bromine in carbon disulphide. From the acid fraction, b.p. 160°/4 mm., is obtained a *methyl* ester, b.p. 112—113°/4 mm., d^{15}_4 0.9719, n^{20}_D 1.44129, corresponding with an acid $C_{11}H_{22}O_2$, and an *acid* $C_{12}H_{18}O_2$, b.p. 156—157°/4 mm. The fraction, b.p. 170—180°/4 mm., crystallises, the equivalent corresponding with an *acid* $C_{15}H_{24}O_2$.

J. W. BAKER.

PATENTS.

Manufacture of juices rich in vitamins, from plants. J. KORSSELT (B.P. 320,369, 2.7.28).—Raw plant juices rich in vitamins may have their chlorophyll, albumin, and oxalic acid removed, and the resultant juices rendered sterile, by treatment with calcium hydrogen citrate, lactate, or tartrate etc., followed by heating to 100° in an inert atmosphere such as nitrogen. [Stat. ref.]

E. HOLMES.

Manufacture of preparations having an action resembling that of tuberculin. I. G. FARBENIND. A.-G. (B.P. 295,629, 27.7.28. Ger., 15.8.27. Addn. to B.P. 285,087; B., 1929, 661).—The process of the prior patent is applied to preparations of *B. coli*.

F. G. CROSSE.

Artificial ageing of tobacco. T. H. KELLER (U.S.P. 1,729,482, 24.9.29. Appl., 30.12.27).—The tobacco is moistened with an extract of cereal grass, packed, and sweated.

H. ROYAL-DAWSON.

Curing of tobacco. W. E. LILIENFIELD, Assr. to LILIENFIELD BROS. & Co. (U.S.P. 1,731,018, 10.8.29. Appl., 17.3.27).—Loosened tobacco leaves, after being aged and fermented, are subjected to artificial ultra-violet rays for $\frac{1}{4}$ —2 hrs. to produce the desired mildness.

F. G. CLARKE.

Manufacture of synthetic camphor. L. DUPONT (U.S.P. 1,733,218, 29.10.29. Appl., 5.6.26. Fr., 12.6.25).—See B.P. 253,542; B., 1927, 828.

Complex metallic compound of pyrocatechol. H. SCHMIDT, Assr. to WINTHROP CHEM. Co., INC. (U.S.P. 1,718,492, 25.6.29. Appl., 30.4.27. Ger., 6.2.24).—See B.P. 295,734; B., 1928, 799.

Manufacture of sterols. A. GAMS and F. LOCHER, Assr. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,733,009, 22.10.29. Appl., 7.6.28. Switz., 14.6.27).—See B.P. 292,133; B., 1929, 737.

Preparation of α -hydrazino- β -nitropyridine. C. RÄTH (U.S.P. 1,733,695, 29.10.29. Appl., 1.2.26. Ger., 5.2.25).—See B.P. 255,811; B., 1927, 29.

Decreasing the toxic action of cocaines. R. ECKERMANN (U.S.P. 1,733,298, 29.10.29. Appl., 26.6.26. Swed., 13.3.26).—See B.P. 267,463; B., 1927, 974.

Colloidal iodine (B.P. 320,719).—See VII. Milk for diabetics (B.P. 320,497).—See XIX.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Panchromatic silver salt sensitisers and their relationship to the theory of hypersensitisation and sensitisation. A. STEIGMANN (Z. wiss. Phot., 1929, 27, 117—118).—Panchromatic sensitising agents are prepared by treating eosins mixed with sodium sulphide with a mixture of sulphuric and nitric acids, and then reducing the product with hyposulphite. These substances sensitise silver chloride directly, but do not sensitise negative emulsions unless chloride is present (cf. A., 1928, 602).

R. CUTHILL.

Desensitisers. II. (MISS) F. M. HAMER (Phot. J., 1929, 69, 409—415; cf. B., 1929, 872).—An investigation of various *p*-dialkylaminoanils of quinoline-2- and -4-aldehyde methiodides and ethiodides shows that the desensitising capacities of these compounds is comparable with that of safranin. Although this property is somewhat decreased in the presence of sulphite-carbonate solutions, they are, however, very efficient when used in a preliminary bath, owing presumably to the stable silver halide adsorption complexes formed. Their breakdown products, however, attack the latent image. The *p*-dimethylaminoanil of benzthiazole-aldehyde ethiodide, a strong desensitiser, gave chemical fog, whilst the similar anils of pyridine-2-aldehyde methiodide and ethiodide lost their desensitising capacity almost completely in sulphite-carbonate solutions, and could not be used in a preliminary bath. The substitution of acidic, basic, and acylamino-groups in various

aminoanils of quinoline-2-aldehyde methiodide cause no marked changes in the desensitising capacity, but introduction of acid substituents appeared to cause a fall. Examination of a number of cinnamylidene derivatives and 2-methylquinoline alkyl salts gave only desensitisers inferior to safranin, whilst the desensitising capacities of a series of styryl compounds were shown to be completely destroyed by the sulphite in the developer. It is therefore concluded that at present there are no useful desensitisers to be derived from quinoline or related bases.

J. W. GLASSETT.

Diffuse illumination of photographic plates and photographic photometry. A. DENISOV (*Z. wiss. Phot.*, 1929, 27, 128—144).—The effect on the relative blackening and contrast of a photographic plate of fogging by exposure to diffuse light of the same quality before, during, or after the normal exposure has been investigated. For densities not greater than unity diffuse illumination increases the relative density, and for densities of 0.05 and below the contrast is increased. These facts may be turned to advantage in photographic photometry when very small amounts of light are being dealt with. Prolonged exposure to diffuse light or exposure to intense diffuse light reduces the contrast.

R. CUTHILL.

Relationship between the resolving power of a photographic material and the wave-length of the light. O. SANDVIK and G. SILBERSTEIN (*Z. wiss. Phot.*, 1929, 27, 119—127).—Examination of a number of kinds of photographic plates has shown the resolving power over the range of wave-lengths 380—725 m μ to be maximal in the violet or ultra-violet and minima at about 535 m μ for orthochromatic plates, and about 485 m μ for plates sensitised with dyes (cf. A., 1929, 402).

R. CUTHILL.

PATENTS.

Manufacture of light-sensitive materials. KALE & Co. A.-G. (B.P. 294,247, 16.7.28. Ger., 21.7.27).—A combination of boric acid and an organic acid (*g.*, citric, tartaric) is found to be more efficient than an organic acid alone in preventing the premature formation of colouring matter in diazo papers in which the coupling agent and the diazo compound are applied together. A suitable coating mixture is: stannic chloride double salt of 4-diazo-1-dimethylaniline 22 pts., boric acid 20 pts., tartaric acid 50 pts., chloroglucinol 2 pts., and water 1000 pts. J. W. GLASSETT.

Developers for photographic purposes. ØVAERT PHOTO-PRODUKTEN, NAAML. VENN. (B.P. 98,953, 17.10.28. Ger., 17.10.27).—Images of varying colour may be obtained by direct development of ordinary silver halide emulsions by the addition of sodium selenite or other selenium compound to the developer; sodium thiosulphate may also be added. Violet to blue tones are obtained in the presence of a restrainer (potassium bromide), whilst the addition of an accelerator (sodium carbonate) or an increase in the quantity of thiosulphate gives brown to red tones.

J. W. GLASSETT.

Production of printing surfaces [on celluloid etc. from gelatin reliefs]. E. SAUER (B.P. 296,065, 23.8.28. Ger., 24.8.27).—Gelatin reliefs, after drying

by washing with amyl acetate or amyl alcohol, wiping off the liquid, and subsequently removing residual moisture with a blower or by suction, are coated with a liquid capable of softening celluloid but not gelatin, and a celluloid sheet is pressed on to the surface with rollers etc.

L. A. COLES.

XXIII.—SANITATION; WATER PURIFICATION.

Activated sludge process of sewage treatment [at Bangalore]. M. SWAMINATHAN (*J. Indian Inst. Sci.*, 1929, 12A, 131—151).—After four years' continuous operation, a plant, designed to treat the sewage from a population of 200 on a basis of 30 gals. per head per day, in 1926, was treating the sewage from 400 people, using only 18 gals. per head. No trouble has been experienced with the diffusers, except that due to occasional deposits of silt. It is stated that a rather high consumption of air (5.3 cub. ft.) per gal. of sewage treated has been found necessary to obtain a suitable circulation in the aeration tank (3 ft. deep), and to prevent the development of numerous insect larvae and worms and also "bulking" due to filamentous growths, principally *Sphaerotilus natans*. The effluents obtained throughout have been entirely satisfactory; a low dissolved oxygen content is shown to be due to the oxygen demand of the live population of the sludge. Biological observations indicate seasonal variations in the predominant types of organisms present. Sterilisation experiments indicated that these higher organisms utilised the bacteria, present as a source of food, but elimination of them did not appreciably increase the rate of nitrification.

C. JEPSON.

Sewage purification by intermittent land irrigation at Lublino (Moscow), 1914—1923. K. K. BARSOV and K. N. KOROLKOV (*Rep. Moscow Sewage Res. Comm.*, 1928, 10, 120 pp.).—A general description of the works, the results of operation, and analytical returns covering this period is given.

C. JEPSON.

Comparison of the dilution and absorption methods for determination of biochemical oxygen demand [of sewage]. G. E. SYMONS and A. M. BUSWELL (*Ind. Eng. Chem. [Anal.]*, 1929, 1, 214—215).—A modification of Sierp's procedure for the determination of the oxygen demand of sewage by the direct absorption method (B., 1928, 318) is outlined, and the results are compared with those of the dilution method. The former obviates the use of the large number of subsamples, is shorter and more convenient, and the condition of decreasing oxygen concentration is absent. The amount of agitation necessitated by the apparatus apparently causes some slight variation in readings, and it must be kept at constant temperature when the readings are taken. Anomalous results are not obtained by the two methods when buffered dilution water is used in the latter. Both methods show two-stage oxidation.

E. H. SHARPLES.

Improvements in the Imhoff sewage-settling tank. O. MOHR (*Eng. News-Rec.*, 1929, 103, 647—648).—Difficulties met with in the operation of Imhoff tanks, *e.g.*, foaming, spitting, sludge deposition on the sides of the sedimentation compartment, and accumulations of floating scum, are eliminated by causing the

sedimentation compartments to be submerged. According to the analytical returns quoted, this form of tank permits a better separation of colloidal matter than the older type. No arrangement is indicated for gas collection. C. JEPSON.

Purification of water by ultra-violet radiation.

J. M. BLOCHER (J. Amer. Water Works' Assoc., 1929, 21, 1361—1372).—The river is used by the town of Berea, Ohio, as the source of its drinking-water supply, the water being treated with aluminium sulphate followed by rapid sand-filtration and sterilisation by means of ultra-violet rays. The water is passed through four copper boxes in series, each of which contains a mercury-vapour lamp surrounded by a quartz cylinder. Two such sets are installed and may be used separately, in series, or in parallel. The plant is capable of dealing with 2 million gals. per day, and results so far obtained indicate that the sterilisation is effective, particularly with gas-forming micro-organisms, and that after-growths are negligible. There is no danger of over-dosage or of the production of disagreeable tastes. The current consumption is approx. 200 kw./million gals.

C. JEPSON.

Electrolytic cells in chlorination [of drinking water] for the destruction of algæ.

W. T. BAILEY (J. Amer. Water Works' Assoc., 1929, 21, 1388—1394).—Chlorine has been successfully used to prevent algal growths in settlement basins etc. at Council Bluffs, Iowa, with consequent improvement in the quality of water supplied. Experiments to determine whether electrolytic chlorine was a more efficient bactericidal agent than liquid chlorine yielded negative results. The cost of production of electrolytic chlorine compared very unfavourably with the cost of using liquid chlorine.

C. JEPSON.

De-aeration of water in relation to character of water supply.

J. R. McDERMET (J. Amer. Water Works' Assoc., 1929, 21, 1339—1344).—The efficiency of either de-aeration or de-activation of water with a view to corrosion prevention is largely dependent on the nature of the supply. It is generally sufficient to reduce the amount of oxygen present to 0.5 c.c. per litre, and, with waters containing lime or softened by the lime-soda process, protection is assisted by the deposition of a film of scale. Waters free from lime can be given useful but not complete protection if the p_H value is not below 6.0. Zeolite-softened waters which when heated are frequently very corrosive over a narrow zone of attack are rendered non-corrosive by de-aeration, provided the p_H value is not less than 9.5. When water is treated by the de-activation process, the dissolved and colloidal forms of iron taken up are difficult to remove unless p_H 9.3 can be maintained. To accomplish this with varying conditions and rates of flow a calcined and partially sintered magnesium carbonate has been successfully employed, although in exceptional cases additional adjustment is necessary. C. JEPSON.

High-manganese effluents from idle [drinking-water] filters.

A. C. JANZIG and I. A. MONTANK (J. Amer. Water Works' Assoc., 1929, 21, 1319—1328).—The presence of manganese in potable waters causes

stains and incrustation of pipes. The maximum permissible amount of manganese and iron together is quoted as 0.3 p.p.m. Periodic increase in the manganese content of the water at Columbia Heights was traced to the effluent from stand-by filters. Manganese previously deposited on the sand grains was redissolved under anaerobic conditions, probably by biological agency, and the effluents obtained when the filters were recommissioned after washing for 15 min. contained more than the normal amount for nearly 12 hrs. Similar filters which had been treated with copper sulphate for algal prevention were found to deliver a smaller quantity. Manganese interferes with the *o*-tolidine test for residual chlorine, but no direct relation was found.

C. JEPSON.

Errors in the Clark method for determining hardness [in water].

G. G. TOWN (J. Amer. Water Works' Assoc., 1929, 21, 1395—1404).—In this method of determining hardness, any iron or magnesium present is considered to have the same soap-consuming power as calcium, and no allowance is made for the requirements of the water itself. It is proposed that the amount of soap solution required for a volume of distilled water equivalent to that of the sample and the soap-consuming power of any iron present (determined separately) should be deducted from the amount required to produce the false end-point, the remainder being considered as the amount required by the calcium. The quantity now required to convert the false end-point into a final one is a measure of the magnesium present, and results may be quoted in terms of the carbonates of iron, calcium, and magnesium, respectively.

C. JEPSON.

Determination of hardness in water by means of soap solution.

T. VON ESTREICHER (Chem.-Ztg., 1929, 53, 800).—The method described by Bruhns (B., 1929, 662) for determining separately the calcium and magnesium hardness of water was originally described by Olszewski (Abhandl. Krakauer Akad. Wiss., Math.-natur. Kl., 1881, 9, 173).

A. R. POWELL.

Dissociation of water. FELLOWS.—See I. Low-temperature tar as disinfectant. EHRMANN.—See II.

Determination of phenol in presence of salicylates. HAMILTON and SMITH.—See III.

PATENTS.

Oxygen-evolving preparations and their application for air purification.

DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER, W. ZISCH, and E. HERZOG (B.P. 319,393, 19.6.28).—The preparations comprise mixtures of alkali trioxides with alkali hydroxides prepared by heating at 50—120° mixtures of alkalyl hydroxides and alkali peroxides, obtained as described in B.P. 280,554 (B., 1928, 447) or by mixing alkali peroxides with alkali peroxide dihydrates, the reaction proceeding thus: $\text{NaO}_2\text{H} + \text{Na}_2\text{O}_2 = \text{Na}_2\text{O}_3 + \text{NaOH}$. Inert material, alkali hydroxides, and catalysts may be incorporated with the products. L. A. COLES.

Insecticide (U.S.P. 1,732,240). Insecticide and parasiticide (U.S.P. 1,731,677—8).—See XVI.