

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUGUST 15 and 22, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Heat transmission: modern methods of expressing convection data. M. FISHENDEN (Gas World, 1930, 92, 632—637).—Mathematical treatment of heat transfer is applied to problems of the coal-gas industry. Data and calculations are given for radiations of varying wave-length, natural or forced convection, and heat transfer in tubes or through partition walls. A number of results, from different observers, have been correlated to give figures for natural convection from horizontal cylinders suspended in gases, and for forced convection in tubes. An example is given of the application of calculations, derived in this way, for the case of hot air flowing through a horizontal tube, with or without insulation.

R. H. GRIFFITH.

Maintenance and working of simple types of autoclaves. A. W. C. HARRISON (Ind. Chemist, 1930, 6, 244—248; cf. B., 1930, 643).—Packing materials, discharge arrangements, pipe-lines, and accessories are discussed. Processes using ammonia entail the addition of 7 or 8 times as much as that theoretically needed, and the greater part of this can be recovered by blowing off and condensing at a suitable point during the cooling down. Two valves should be used on the ammonia connexion to avoid leakage and consequent blockage. Gas-heating installations are described and a detailed description is given of the conversion of a batch of Schäffer acid into Brönner acid by amination with ammonia and ammonium sulphite.

C. IRWIN.

Construction and use of homogenisers. C. H. MÖLLERING (Chem. Fabr., 1930, 239—240).—The principle of all homogenising machines consists in the forcing of the material through fine passages, whereby the particles in suspension or emulsified are brought to a uniform size by friction. Power is economised by working at higher temperatures. Pressures of 150—200 atm. are usual, and are obtained either by a three-stage compressor or by centrifugal force. A machine of each type is described. The pressure obtained in centrifugal machines is lower than with pumps, but as they also act as emulsifiers by mechanical mixing the results are equal. The process of homogenisation was first applied to milk, but it is also employed in the margarine, lacquer, and artificial silk industries, and in connexion with pharmaceutical chemicals and cosmetics.

C. IRWIN.

Porous medium for flotation cells. F. C. DYER and H. L. McCLELLAND (Univ. Toronto Fac. Appl. Sci. Eng., Eng. Res., Bull. No. 8, 1928, 419—424).—A résumé of the defects of diaphragms made of canvas, rubber, or ceramic material is given. Since resistance to abrasion was a highly desired quality, linoleum was con-

sidered most worthy of investigation owing to its known good qualities on concentrating tables. Porous linoleum was unobtainable in a ready manufactured state, and drilling or punching was too slow and unsatisfactory owing to the holes closing up; an encouraging result was obtained by stripping off the canvas backing, cutting into strips, and reassembling with a grooved face to a smooth face. As, naturally, the grooves were of different sizes and the distribution of air was not uniform, other grooves were cut by a multiple saw and the result was good, except that it was impossible to prevent the adhesive (durofix) from closing up some of the holes. Experiments were then tried starting with an uncured mix of cork and oils which was granulated, heated between rolls till soft and sticky, and then rolled, the grooves being formed by ridges on one roll; it was found unnecessary to roll the material in the form of strips because it could be guillotined without closing up the ends of the grooves. The finally adopted roll was then formed with only circumferential ridges about 0.0012 in. high and wide, spaced $\frac{3}{8}$ in. apart; this paired with a smooth roll and formed sheets $\frac{1}{8}$ in. thick, which were then sheared at right angles to the grooves into strips $\frac{3}{8}$ in. wide, the reassembled sheet then being $\frac{3}{8}$ in. thick and having the holes spaced at $\frac{1}{8}$ in. apart one way and $\frac{3}{8}$ in. the other. The natural stickiness was sufficient for adherence, and the curing was effected in an oven for several days. Besides more obvious advantages, scale-forming liquors cannot approach the walls of the holes through the substance of the material, so that they do not choke even though scale may build up harmlessly on the surface between the holes. Although linoleum is resistant to a large number of solutions, the authors suggest that the same method of manufacture may be applied to other materials such as tar products (congoleum), soft metals, glass, rubber, or clay.

B. M. VENABLES.

Bacterial filtration by the Berkefeld filter and the size of the pores. H. HOEK (Chem. Fabr., 1930, 249—250).—These filters consist of ceramic cylinders, and as the pore diameters may vary it is desirable to test their suitability for retaining bacteria. A number may be immersed in water and connected to air under gradually increased pressure, the pressure at which each commences to pass bubbles being noted. From this the diameter of the largest pores can be calculated. Experiments on the filtration of bacterial cultures are described which indicate that satisfactory cylinders should resist an air pressure of at least 0.55 atm. without passing bubbles. The cylinder dimensions are immaterial; it is the maximum pore size, not the average, that is important.

C. IRWIN.

* The remainder of this set of Abstracts will appear in next week's issue.

Pilot-plant fractionating column. R. McCULLOUGH and L. D. GITTINGS (Ind. Eng. Chem., 1930, 22, 584—587).—A fractionating column suitable for semi-works' scale operation is described. It consists of a 3-in. pipe, 9 ft. long, packed with $\frac{1}{4}$ -in. glass Raschig rings. It is fitted with a hot-air jacket, and is itself also electrically heated. The boiler consists of a piece of 12-in. pipe. The upper 2 ft. of the column is air-cooled and acts as a partial condenser. An air-cooled final condenser is also fitted. The column was used to fractionate nitrodiphenyls at 30-mm. pressure which it effected at 3.0 lb./hr., and chlorodiphenyls at atmospheric pressure for which the rate obtained was 2.1 lb./hr.

C. IRWIN.

Laws of motion of particles in a fluid. R. G. LUNNON (Trans. Inst. Min. Met., 1930, 38, 402—420).—The limitations of Rittinger's law are greater than has previously been assumed. Stokes' law (resistance proportional to speed) holds within 5% when vd is between 0 and 0.002 for water and between 0 and 0.03 for air (v = velocity and d = diam. of sphere in c.g.s. units). Similarly, Allen's law (resistance proportional to the $3/2$ power of the speed) holds when vd is between 0.3 and 3 for water and between 5 and 50 for air. Finally, Newton's law (resistance proportional to the square of the velocity) holds when vd is between 10 and 1000 for water and between 150 and 15,000 for air. The equations for the motion of a sphere are applicable to the motion of irregular particles with certain small changes. The only exception occurs when the particles are closely packed together and are just lifted by a rising fluid.

W. E. DOWNEY.

Separation of gases by adsorption with charcoal. K. PETERS and K. WEIL (Z. angew. Chem., 1930, 43, 608—612).—The adsorption isotherms of argon, krypton, and xenon at temperatures from -80° to 0° have been determined. At pressures from 0 to 100 mm. Hg these obey the relationship $a = \alpha^{1/n}$, where a is the mass of adsorbed gas, p the pressure over the adsorbed phase, and α and n are constants. At higher pressures this no longer holds good, but as separation by adsorption is facilitated by low pressure, only this pressure range is considered. From the experimentally obtained isotherms, isobars and "isosteres" (analogous to vapour-pressure curves) for the different gases are obtained. The activated carbon used for these experiments was Bayer A carbon. The optimum conditions for separation are given by these curves, and this may be carried out by either fractional adsorption or fractional "desorption." In the first case due consideration must be given to time of contact, or adsorption will not be complete; if desorption be used it is essential to select the correct temperature for the particular case. It is readily possible to effect a complete separation of the three gases mentioned by these methods and also to separate traces of a less volatile component from the more volatile (e.g., radium emanation from liquid air), when present in quantity too small for chemical separation.

C. IRWIN.

Modern fire extinguishers. E. BIESALSKI (Z. angew. Chem., 1930, 43, 603—608).—Dry fire extinguishers invariably depend on sodium bicarbonate to which a little kieselguhr has been added. This is dis-

tributed by a current of carbon dioxide from a container filled with the liquefied gas. Carbon dioxide snow may also be used, produced by the expansion of the liquefied gas through a jet. Carbon dioxide gas is suitable for permanent sprinkler installations. Liquid fire extinguishers of the acid and soda type are most suitable for extinguishing burning solid materials where penetration is an advantage. Other makers employ water and compressed carbon dioxide or a concentrated solution of sodium or potassium carbonate. The latter solution will extinguish a heavy oil, when burning, by emulsification. "Erkalen" is a water-soluble organic compound marketed for extinguishing fires of lignite and cork dust. Carbon tetrachloride has many advantages, and the alleged danger of phosgene formation is negligible except perhaps in confined spaces. Methyl bromide is an efficient fire extinguisher, but is believed to be dangerous in use. Foam extinguishers employ carbon dioxide with either saponin or other foam producers in aqueous solution. The volume of foam is 8—12 times the volume of the water used. Its electrical conductivity, however, makes it unsuitable in presence of high voltages. The Minimax foam generator employs sodium bicarbonate and aluminium sulphate, or oxalic acid, to generate carbon dioxide, and is connected to a water main.

C. IRWIN.

Determination of humidity. G. ALIVERTI (Nuovo Cim., 1929, 6, cxvii—cxxiii; Chem. Zentr., 1930, i, 1009).—A discussion of the use of various types of hygrometer.

A. A. ELDRIDGE.

Graphical methods in the gas industry. SCHUMACHER.—See II. Measurement of colour and gloss. DESBLEDS.—See V. Silica grinding. CAWOOD.—See VII.

PATENTS.

[Boiler] furnaces. J. J. DESCHAMPS (B.P. 330,200, 1.10.29. Fr., 1.10.28).—The water-tubes forming the walls of a boiler furnace are provided with fins of such a diameter and pitch that ash accumulates on them to such an extent as to cover the whole surface of the tube proper. In the case of nearly horizontal tubes for water-bottoms, the fins are longitudinal blades, radial on the upper parts and curved on the lower.

B. M. VENABLES.

[Boiler] furnace. C. J. HUBER, ASSR. to FLYNN & EMRICH Co. (U.S.P. 1,751,861, 25.3.30. Appl., 2.6.25).—A furnace has a coking shelf adjacent the opening from the supply hopper and an arch immediately above it. Air is drawn through a hollow metallic girder in the arch and between double walls of the hopper into the fire.

B. M. VENABLES.

Furnaces for heating articles. H. A. DREFFEN (B.P. 302,652, 27.11.28. U.S., 19.12.27).—A furnace for heating metal plates and the like is provided with a combustion chamber down a longitudinal side of the goods chamber which near the burner is in unobstructed communication with the latter, but owing to the longitudinal velocity of the flame the heat reaches the goods only by radiation, and there may even be a back-circulation into the combustion chamber at this point. Further along the gases are permitted to enter the goods chamber through ports in a dividing wall.

B. M. VENABLES.

[Heating] furnaces [for metal bars etc.]. INTERNAT. CONSTRUCTION CO., LTD., E. P. JONES, and J. H. MIDDLETON (B.P. 330,160, 2.7.29).—A single chamber for heating bars and two chambers for heating the plates rolled therefrom are combined with two fireplaces in one structure, the bar chamber lying transversely between the fires and the plate chambers.

B. M. VENABLES.

Apparatus for heating [metal sheets etc.]. E. M. FREELAND and F. MCGOUGH, ASSTS. to FOLLANSBEE BROS. CO. (U.S.P. 1,750,884, 18.3.30. Appl., 31.5.27).—A reverberatory furnace for heating sheets etc. has the hearth formed of live rolls which are themselves heated from below by the products of combustion passing through flues, though their shafts may be water-cooled.

B. M. VENABLES.

Heat exchanger. (A) W. M. CROSS, (B) W. B. PHILLIPS, ASST. to W. M. CROSS (U.S.P. [A] 1,751,725 and [B] 1,751,757, 25.3.30. Appl., [A] 7.1.26, [B] 14.1.28).—In (A) corrugated sheets are arranged ridge to ridge in pairs forming longitudinal passages for the inner fluid. The pairs are kept apart by spacers embossed on the sheets at the ends and intermediately, thus forming transverse passages for the outer fluid. (B) In an exchanger as above, separate spacers are provided and the preferred arrangement of the adjacent sheets of different pairs is ridge to valley, so that the transverse passages have parallel but sinuous walls.

B. M. VENABLES.

Drying apparatus. A. T. OAKLEY and P. LUPKE, JUN., ASSTS. to ESSEX RUBBER CO. (U.S.P. 1,750,813, 18.3.30. Appl., 10.11.26).—A rotary drum dryer for materials which are sensitive to heat, such as pulverised reclaimed rubber, is provided with interior stationary conduits for drying and moist gases, respectively, perforated throughout their length and supported on a structural steel bridge extending right through the drum in such a manner that they may expand independently of each other.

B. M. VENABLES.

Drying ovens. S. L. GROOM (B.P. 329,972, 24.1.29).—A mechanism is described by which plate-like objects which can be handled only on one side are transferred from the upward run of a vertical conveyor to the downward run without turning over.

B. M. VENABLES.

Dryer. C. J. BLEIL (U.S.P. 1,749,525, 4.3.30. Appl., 12.4.28).—The apparatus comprises a number of superposed circular hearths, inclined alternately to the centre and circumference, with rakes driven by an axial shaft; the transfer passages through the hearths are provided with double doors operated out of phase by the stirrers, so that they form air-locks permitting different conditions to be maintained in different chambers. Some chambers may be heated by wet steam in coils, others by dry steam mixed with the material, and some chambers may be subjected to a vacuum.

B. M. VENABLES.

Drying process and apparatus. A. A. CRIQUI, ASST. to BUFFALO FORGE CO. (U.S.P. 1,751,472, 25.3.30. Appl., 1.5.26).—The material is subjected alternately to strong blasts of air in a direction which holds it on its support, then to feebler currents in an opposite direction; e.g., it may be placed on a pervious conveyor

and exposed to concentrated downward and diffused upward currents of heated air. B. M. VENABLES.

Pulverising machine. W. J. ARMSTRONG, ASST. to JEFFREY MANUF. CO. (U.S.P. 1,751,611, 25.3.30. Appl., 2.10.26. Renewed 12.6.29).—A disintegrator is provided with a pocket in advance of the feed hopper and out of the path of the hammers, which pocket is fitted with an abutment and a shelf in addition to two walls at right angles, all of which surfaces are intended to deflect and crush material flung by the hammers.

B. M. VENABLES.

Pulverising machine. W. K. LIGGETT, ASST. to JEFFREY MANUF. CO. (U.S.P. 1,751,000, 18.3.30. Appl., 20.5.18. Renewed 30.7.29).—A disintegrator of the hammer type is fed from the upper circumference; alongside the hopper, but outside the zone swept by the hammers, is a crushing zone in which particles that are too large to enter the hammer zone are swept round like an eddy.

B. M. VENABLES.

Pulveriser mill. R. O. GROMAN, ASST. to FULLER-LEIGH CO. (U.S.P. 1,750,636, 18.3.30. Appl., 11.2.27).—The invention relates to methods of obtaining even distribution of air to a mill which may be of the ball-and-ring type.

B. M. VENABLES.

Abrading and crushing machine. P. L. CROWE (U.S.P. 1,750,573, 11.3.30. Appl., 27.7.28).—The machine has an electric motor built round it and the draught that carries away the ground coal or other material has previously cooled the motor. The abrasion is effected inside a conical hopper with apertures for the exit of ground coal and with an internal worm which tends to raise any material sliding along it. The moving unit comprises a number of double conical elements assembled on a vertical shaft, the largest diameter of one of them nearly closing the bottom of the hopper. The ground material passes mainly through the walls of the hopper to a fan surrounding it.

B. M. VENABLES.

Machine for kneading, crushing, and similar operations. B. ZETTERGREN (U.S.P. 1,750,558, 11.3.30. Appl., 2.10.28. Swed., 23.12.27).—A mill in which a roller is dragged round inside a cylindrical casing by means of a crank and pair of links is described.

B. M. VENABLES.

Crusher heads for homogenising apparatus. A. P. ANDERSEN (B.P. 312,351, 3.5.29. Denm., 26.5.28).—Each of the grinding surfaces (with central feed) is provided with concentric grooves which are not opposite each other.

B. M. VENABLES.

Determination of moisture in aggregate [by unskilled persons]. R. E. ROBB (U.S.P. 1,749,606, 4.3.30. Appl., 17.8.28).—In a vessel suitable for suspending from a weighing-beam, water is placed to a definite level, determined by a lower draw-off cock. The lower cock is closed and a definite weight of dry aggregate of the same quality as that to be tested is added to the vessel and the water that runs out of a higher cock is collected in a measuring cylinder. The gross weight of the weighing vessel is then counterpoised by adjusting a sliding weight on the scale beam and the weight is clamped in that position. The standard aggregate is then discarded and the weighing vessel refilled with water up to the lower cock, then moist

aggregate is added (the overflow being caught) until the vessel again balances; there will then be present the same weight of dry aggregate as was taken of the standard, but the volume of water that has overflowed out of the upper cock will be greater by exactly the amount of original moisture in the sample, so that the height of water in the measuring cylinder will indicate directly on a scale moisture percentage of dry weight. No calculation and no weighing other than the occasional dry standard are necessary. B. M. VENABLES.

Material classifying devices. H. G. LYKKEN (B.P. 330,145, 14.6.29. U.S., 5.12.28).—An air separator is placed above a disintegrator and comprises a number of cylindro-conical walls which are co-axial, but not parallel, and adjustable relatively to each other. A bladed element to produce whirling is also provided which telescopes on the cylindrical part of the inner baffle to vary the effective area of the passages between the blades. B. M. VENABLES.

[Compressed air] atomisers and powder blowers. A. S. FISHER & NIELSEN, and P. A. FISHER (B.P. 330,022, 12.3.29).—For a paint or powder sprayer, a trigger valve is described which prevents dribbling at the paint jet. B. M. VENABLES.

Separation of materials of different sp. gravities. T. M. CHANCE and H. O. STAPLES (U.S.P. 1,750,090, 11.3.30. Appl., 26.3.29).—It is known to separate coal from rock by flotation in a fluid medium composed of sand and water kept in a state of agitation sufficient for it to behave as a liquid of intermediate density. The principle is extended to elevate the coal alone to any desired point, *e.g.*, from the mine to the surface, leaving the waste matter behind. An apparatus for effecting the separation and transport of coal or other materials is described. B. M. VENABLES.

Separation of solids from gases and liquids. J. E. POLLAK. From F. ARLEDTER (B.P. 329,992, 24.1.29).—The fluid is admitted through the wall, at an intermediate level, of a funnel-shaped tank which is provided with an outlet for solid matter at the bottom and peripheral overflow for clear fluid at the top. The fluid may be admitted tangentially to produce a swirling motion. [Stat. ref.] B. M. VENABLES.

Releasing the pressure of mixture of solids and liquids existing under high pressure. W. R. TATE, H. P. STEPHENSON, H. P. DEAN, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 330,106, 9.5.29).—The pressure of an abrasive liquid is released in a tandem hydraulic ram, the other piston of which operates on clean liquid, which may be caused to do useful work. B. M. VENABLES.

Filter. F. B. LEOPOLD, Assr. to McKENNA BRASS & MANUF. Co., Inc., and L. DE MARCUS (U.S.P. 1,750,879, 18.3.30. Appl., 10.11.26).—A non-porous or sand filter is combined with a porous or paper filter; the latter forms a check on the condition of the former, and for convenience of inspection is placed in the cover of the apparatus. Connexions are provided so that, when cleaning, the sand filter only receives a back-flush. B. M. VENABLES.

Filtering device. C. A. and P. X. MCGEEHAN (U.S.P. 1,750,185, 11.3.30. Appl., 22.8.27).—A number

of disc filter elements are mounted with spacers on an outlet pipe which can be rotated by hand. Fixed but flexible scrapers are attached to the outer casing which forms the inlet chamber and serves to remove collected sludge and/or filter aid (if used). B. M. VENABLES.

Filter device. F. L. M. KENNEY (U.S.P. 1,749,730, 4.3.30. Appl., 22.6.28).—A simple device for insertion in a pipe-line or attachment to a tap is described; a filter disc is clamped between inlet and outlet chambers. B. M. VENABLES.

Centrifugal machine. W. H. BATH, Assr. to SHARPLES SPECIALTY Co. (U.S.P. 1,750,154, 11.3.30. Appl., 5.12.28).—A cushioned gyrating bearing for a supercentrifuge is described. B. M. VENABLES.

Clarifier bowl. L. D. JONES and A. U. AYRES, Assrs. to SHARPLES SPECIALTY Co. (U.S.P. 1,749,538, 4.3.30. Appl., 4.9.25).—For a centrifuge, a form of neck is described by which the depth of liquid retained may be easily varied by changing a weir ring. B. M. VENABLES.

Viscosimeters. A. E. ROBINSON (B.P. 330,042, 2.4.29).—A viscosimeter of the falling-ball type is provided with electric contacts at the bottom to indicate the time of arrival of the ball when used in an opaque fluid. B. M. VENABLES.

Method and apparatus for crystallisation. E. W. KOPKE, Assr. to F. L. ALLEN (U.S.P. 1,749,588, 4.3.30. Appl., 19.1.27).—The apparatus comprises a horizontally elongated vessel with stirrers spaced on a horizontal shaft. Alternating with the stirrers are vertical, stationary, flat cooling coils extending across substantially the whole transverse area of the vessel; these are supplied in parallel with regulable quantities of cooling medium so that a rapid initial cooling and slower final cooling may be effected. The liquid, *e.g.*, mastic, is supplied continuously. B. M. VENABLES.

Fractionating apparatus. E. H. HARRIS, Assr. to B. M. HERR (U.S.P. 1,750,306, 11.3.30. Appl., 6.8.26).—A fractionator in which the gradation of temperature is continuous and not stepped is constructed of a bundle of inclined tubes for the vapour being treated, around which are passed a number of circulations of heating or cooling fluid, the temperatures of which are thermostatically controlled; a heater, cooler, and pump are included in each circuit. To prevent sudden changes of temperature at the walls dividing one circulation from the next, baffles transverse to the tubes are provided which are spaced in such a manner to give a varying velocity (and hence heat transmission) of the outer fluid, the spacing being precalculated according to the laws of fractionation and of heat transmission. B. M. VENABLES.

Evaporator. W. SIECK, JUN., Assr. to W. GARRIGUE & Co. (U.S.P. 1,750,434, 11.3.30. Appl., 16.1.28).—A bundle of inclined tubes is supplied with heating medium inside so that vapour produced outside can immediately leave the heating surface and thus improve the transmission of heat. Definite circulation is provided for by partitioning off a lower, inclined, longitudinal compartment for the return flow of liquid. Means are also provided for the removal of precipitate. B. M. VENABLES.

Chemical apparatus. [Precipitation tanks.] W. H. ALLEN, Assr. to PARKER RUST PROOF CO. (U.S.P. 1,750,617, 18.3.30. Appl., 14.3.27).—Precipitates are removed from the bottom of a tank by a worm or other type of conveyor working in a sediment chamber below the tank. This chamber communicates with a stand-pipe normally extending to above the level of the liquid in the tank, but it is made telescopic or flexible so that to discharge sediment it may be lowered below liquid level. A perforated false bottom may be placed in the tank. B. M. VENABLES.

Dust separators and collectors. DORMAN, LONG & Co., LTD., and M. R. KIRBY (B.P. 330,163, 5.7.29).—An inner whirling chamber is situated within an outer settling chamber. The former has a cylindrical outer surface with tangential slots to permit exit of concentrated dust-laden air to the outer settling space, but it (the inner chamber) is formed from one or a number of intercoiled, helical passages; alternatively, it may be cylindrical and the whirling produced by a paddle-wheel. B. M. VENABLES.

Apparatus for filtering air. J. C. HINES (U.S.P. 1,751,999, 25.3.30. Appl., 26.5.27).—A method of distributing oil or viscous liquid over labyrinth plates is described, the edges of the plates being bent back to prevent oil being blown off. B. M. VENABLES.

Thermal-conductivity gas-analysis apparatus. J. C. PETERS, JUN. (U.S.P. 1,751,715, 25.3.30. Appl., 27.2.26. Renewed 4.2.30).—The sample chamber of an apparatus that compares the heat loss from two electric-resistance wires (in the sample and a standard gas) by means of a Wheatstone bridge (cf. U.S.P. 1,504,707) is lined with non-corrodible material, and the heat conductivity between the lining and wall is maintained at a high definite value by the interposition of solder, varnish, or the like. B. M. VENABLES.

Light filter. I. SHOJI, Assr. to SANKYO KABUSHIKI KAISHA (U.S.P. 1,751,220, 18.3.30. Appl., 3.8.26. Jap., 24.12.25).—The use of hexamethylenetetramine-triphenol as a film of less than 0.25 mm. is claimed as a filter for ultra-violet light; colouring matter to filter visible light may be added, and the film may be placed between two glass plates or applied to bottles or electric bulbs or even used without a rigid base. A method of manufacture is described in which phenol and formaldehyde are condensed in the presence of ammonia. B. M. VENABLES.

Light filter. C. S. McNAIR (Austr.P. 9581, 27.9.27).—A solution of alum, copper sulphate, and, preferably, an iron salt, together with free sulphuric acid to prevent the formation of basic salts, is contained in a red glass vessel or, alternatively, contains red colouring matter, e.g., a cobalt salt, to neutralise its colour, and is used to cut off heat rays. J. S. G. THOMAS.

Heat-treatment process and apparatus in which a hot liquid mass is employed. C. F. HAMMOND, Assr. to W. SHACKLETON (U.S.P. 1,765,148, 17.6.30. Appl., 1.4.27. U.K., 15.4.26).—See B.P. 278,768; B., 1927, 928.

Heat exchanger. J. HEIZMANN (U.S.P. 1,767,794, 24.6.30. Appl., 31.3.28. Fr., 28.2.28).—See B.P. 297,643; B., 1928, 878.

Heat interchanger. A. E. LEEK (U.S.P. 1,767,989, 24.6.30. Appl., 22.4.27. U.K., 8.12.26).—See B.P. 279,663; B., 1928, 39.

Air- and gas-washing apparatus. E. M. BASSLER (U.S.P. 1,766,588, 24.6.30. Appl., 20.8.23).—See B.P. 296,159; B., 1928, 774.

Apparatus for collecting suspended dust. H. WARING, Assr. to ASSOCIATED LEAD MANUFACTURERS, LTD. (U.S.P. 1,761,377, 3.6.30. Appl., 9.3.27. U.K., 28.8.26).—See B.P. 282,849; B., 1928, 216.

[Centrifugal] fluid-mixing device. P. JANES (U.S.P. 1,766,643, 24.6.30. Appl., 1.3.28. Austr., 30.7.27).—See B.P. 305,877; B., 1929, 498.

Filter for absorbing ultra-violet light. T. SUZUKI and S. SAKURAI, Assrs. to ZAIDAN HOJIN RIKAGAKU KENKYUJO (U.S.P. 1,766,411, 24.6.30. Appl., 8.1.27. Jap., 20.12.23).—See B.P. 242,721; B., 1926, 76.

Production of chemically stable articles. J. K. WIRTH (U.S.P. 1,767,421, 24.6.30. Appl., 19.11.25. Ger., 11.4.25).—See B.P. 292,334; B., 1928, 606.

[Suspension-type] furnace roofs. SCHEIDHAUER & GRESSING A.-G. (B.P. 313,175, 7.6.29. Ger., 8.6.28).

Process and apparatus for manufacture of shaped objects from fusible materials. I. G. FARBERIND A.-G. (B.P. 308,614, 22.3.29. Ger., 23.3.28).

Pulverising apparatus (B.P. 328,437).—See II. **Dehydration** (U.S.P. 1,749,455).—See VII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Origin of coal and oil. E. TERRES and W. STECK (Gas- u. Wasserfach, 1930, 73, Sond.-Heft, 1—5).—Recent views on the origin of coal are briefly outlined. Peat moss (sphagnum) has been transformed into peat in the laboratory by subjecting it to bacterial decay for 9—10 months; during the process only the cellulose appeared to have suffered complete decomposition. By heating the product at 280—300° under pressure it was converted into brown coal containing 35.2% of a bituminous oil. By similar treatment of the sludge formed by the putrefaction of yeast or egg albumin, artificial coals possessing swelling and caking properties have been obtained. These products also contained an oil similar in composition to that extracted from the artificial brown coal. It is concluded that whereas the lignites and brown coals are transformation products of lignin, the bituminous coals originated in protein-containing substances. A. B. MANNING.

Determination of oxygen in coal. F. SCHUSTER (Gas- u. Wasserfach, 1930, 73, 549—551).—The significance of the results obtained in the ultimate analysis of coal is discussed with reference to the interpretation of the oxygen content as determined "by difference." It is shown that combustion produces chemical changes in the mineral constituents of the coal, and that consequently the percentage of ash determined is not necessarily identical with the content of mineral constituents. This affects the percentage of oxygen as determined by difference. Experiments were made to determine the oxygen in coal by a method similar to that used by

Ter Meulen and Heslinga ("Neue Methoden der organisch-chemischen Analyse," Leipzig, 1927). The apparatus and manipulative details are described. The method consists essentially in heating the coal in a combustion tube through which a stream of pure dry hydrogen is passing. The gases produced pass over a heated column of asbestos fibre, where hydrogen and any oxygen unite, and the products, containing also carbon monoxide and dioxide, pass over a column of a mixture of asbestos and nickel freshly reduced from nickel oxide. This column is maintained at 420° and catalytically induces the reactions $\text{CO} + 3\text{H}_2 \rightarrow \text{CH}_4 + \text{H}_2\text{O}$, $\text{CO}_2 + 4\text{H}_2 \rightarrow \text{CH}_4 + 2\text{H}_2\text{O}$. The mixture of gases passes over a short plug of nickel-asbestos mixture maintained at 150°. Sulphur and chlorine are here abstracted from the gas. Finally the gases pass through three weighed U-tubes containing, respectively, (a) a solution of silver sulphate acidified with sulphuric acid, (b) anhydrous calcium chloride, (c) soda-lime and anhydrous calcium chloride. Ammonia and chlorine which has escaped reaction are retained by (a) and are determined by distillation and titration with potassium thiocyanate, respectively; the water produced is calculated from the increase in weight of the tubes. The last U-tube serves to absorb any unchanged carbon dioxide. The percentage of oxygen is calculated from the water produced. It is pointed out that the presence of oxides and sulphides in the mineral constituents leads by this method to oxygen percentages higher than the true value, and to low oxygen content as determined by difference. Experiments were made to determine the differences in the oxygen content as found by the two methods. With three dried coals of ash percentage 7.6, 9.7, 10.3, the differences were +0.7, +1.8, +2.5% O respectively, the hydrogenation method giving the highest results. Coal ash was prepared from the three coals by the usual method and was reduced in the apparatus described. Appreciable quantities of reducible oxygen were shown to be present. It was concluded that the hydrogenation method of Ter Meulen and Heslinga, although quite applicable to pure organic substances, gives results as open to criticism as are those of the difference method when applied to coal, especially when the oxygen content of the coal substance is under consideration.

H. E. BLAYDEN.

Selection of coal for gas producers. E. J. C. BOWMAKER and J. D. CAUWOOD (J. Soc. Glass Tech., 1930, 14, 16—29 T).—Determinations of the percentage of ash, volatile matter, Fe_2O_3 in ash, softening temperature of ash, and aniline solubility were made on samples of 11 coals. The solubility in aniline was determined by Soxhlet extraction of 0.5 g. of powdered dried coal until the aniline in the siphon tube was almost colourless. From the data so obtained the suggested specification for a suitable coal was: ash not above 5%, volatile matter 35—38%, solubility in boiling aniline not above 6%, Fe_2O_3 (in ash) not above 15%, of the dry coal. It was stressed that this applied to Kerpely producers, and might need modification for conditions widely differing from those obtaining with this type of plant.

M. PARKIN.

Functions of coke ovens. E. G. STEWART (Gas World, 1930, 92, 651—659).—Coke-oven developments

are followed with reference to the steel and gas industries, and the function of this type of plant in England, Germany, and America is discussed. The circumstances under which coke ovens can successfully compete with horizontal and vertical retorts for gasworks purposes are described, and it is shown that they are only advantageous when a capacity of about 1000 tons, or more, of coal per day is required. The coke ovens in course of construction at Beckton are described in full, and the arrangements for handling coke and for purification of gas are discussed.

R. H. GRIFFITH.

Activated carbons. G. SIMONIN (Caoutchouc et Gutta-Percha, 1929, 26, 14,357—14,359, 14,468—14,470, 14,516—14,519, 14,551—14,553, 14,594—14,595, 14,632—14,636, 14,701—14,702, 14,742—14,744, 14,778, 14,821; 1930, 27, 14,866—14,868, 14,903—14,904, 14,940—14,941, 14,983—14,985, 15,022—15,023, 15,064—15,065).—After indicating the chief physical properties of activated carbons, it is shown that the activity is not appreciably influenced by the nature of the gas from which the vapour is removed. If care is taken to avoid too high temperatures it is possible to recover methyl and ethyl alcohols, acetone, ether, ethyl acetate, and carbon disulphide unaltered from active carbon in which they have been adsorbed from air. The most rapid method for expulsion of the adsorbed vapours from the carbon without chemical alteration is by treatment with steam; below 130° the steam may be adsorbed to some extent, but above this temperature it is possible substantially to avoid such adsorption. Investigation of the tarry matter retained in the carbon after removal of benzol from coal gas revealed the presence of surprisingly little real tar, probably because of rapid resinification of the major portion of such small quantities as may be present in the gas, by contact with the charcoal. Inactivation of carbon in the removal of benzol from coal gas is attributed to the effect of sulphur, which converts the active carbon molecules into a carbon disulphide. Re-activation by calcination before and after treatment with hydrochloric acid is tentatively explained by the decomposition of the disulphide by iron naturally present; the acid not only decomposes the ferrous sulphide formed in the first calcination, but effects a uniform and intimate dispersion of the regenerated iron salt in readiness for the second calcination. Similarly, it is possible to remove the inactivating sulphur and so to revivify carbon by impregnation with ferric chloride solution followed by heating, e.g., for 2 hrs. at 900°.

D. F. TWISS.

Flash point of activated charcoals. M. SWIDEREK (Rocz. Chem., 1930, 10, 365—384).—Two constant flash points are obtained for activated charcoals, according to the rate of heating; the value obtained when the temperature is raised slowly is higher than when the rate of heating exceeds a certain characteristic value for each type of charcoal. The lower flash point is not characteristic of the given type of charcoal, as its value depends on various factors, such as the temperatures of activation and of drying, content of activating substance, moisture content, etc. The value of the upper point is a function of the temperature at which the charcoal was activated, but is independent of the content of

activators, of various secondary processes which might have taken place during the preparation of the charcoal, and of the initial velocity of heating, provided only that the rate of heating at temperatures close to the flash point is sufficiently low (about $3^{\circ}/\text{min.}$). The adsorptive power of the charcoal stands in no relation to its flash point; this observation militates against the probability of the theory that activated charcoal is a chemisorbent. Charcoals reheated at temperatures above those used for their activation have a lower flash point than have those activated originally at the higher temperature, and this difference is directly proportional to the difference between the two temperatures of activation. The flash point has a maximum value for charcoals the granules of which have a mean diameter of about 1.3 mm.

R. TRUSKOWSKI.

Benzol recovery, with special reference to gas-works' practice. W. H. HOFFERT (Gas World, 1930, 92, 667—671).—The cost of benzol recovery from coal gas, and its refining, is partly dependent on the amount of unsaturated hydrocarbons in the crude liquid. The value of the benzol as a component of town's gas is correspondingly affected. Special methods adopted for the treatment of benzols which deposit gums on storage are mentioned.

R. H. GRIFFITH.

Importance of benzol recovery for the German gas industry. H. KEMMER and G. BAUER (Gas- u. Wasserfach, 1930, 73, 509—519. Cf. Firth, B., 1930, 129).—Nomograms are constructed showing the gain or loss, expressed as a variation in the price of gas, resulting from the recovery of benzol from coal gas, as a function of the prices of coal, coke, and crude benzol. Other methods of calculating the effect of benzol recovery are discussed, and it is concluded that, at the prices now ruling in Germany, such recovery would be economically advantageous. In addition to the direct financial gain, the removal of benzol from the gas would result in improved operation of the purifying plant, the distributing system, etc.

A. B. MANNING.

Application of graphical methods in the gas industry. SCHUMACHER (Gas- u. Wasserfach, 1930, 73, 494—499, 524—529).—Curves and diagrams are constructed to illustrate various relationships and operations of importance in the gas industry, *e.g.*, calculation of the calorific value of coke from its ash and moisture content, flue-gas losses as a function of the temperature and composition of the gas, gas-analysis procedure, regulation of automatic gas-heated water-circulating apparatus, operation of refrigerators, distribution and utilisation of the water supply in a gasworks, etc.

A. B. MANNING.

[Coal-]gas dehydration. E. W. SMITH (Gas World, 1930, 92, 639—648).—Practical results from works where gas dehydration is practised are reviewed, and alternative methods for partial drying of gas are discussed. It is found necessary to apply oil films to the water of the ordinary wet gasholder, and to substitute some other suitable liquid for the water in wet gas meters, when dried gas is prepared. American procedure depending on compression is compared with other types of plant, and alterations in operation due to seasonal changes of temperature are mentioned. Obser-

vations by nine different companies, including actual or potential financial advantages, are recorded.

R. H. GRIFFITH.

Naphthalene [in coal gas]. F. W. J. BELTON (Gas J., 1930, 190, 884—886).—Measures adopted to lessen the amount of naphthalene in town's gas are described, and the gradual diminution of troubles due to blockage of service pipes is traced, following the introduction of new plant.

R. H. GRIFFITH.

Experiences with large "double-gas" [mixed coal- and water-gas] generators. W. SCHROTH (Gas- u. Wasserfach, 1930, 73, Sond.-Heft, 18—26).—The plant that has been in operation at Dresden during the last five years is described. It consists of three generators, each 3 m. in diam. in the gasifying zone and 2 m. in diam. in the carbonising zone. The "blow" gases pass through a dust separator to a regenerator, wherein any combustible constituents are burned with secondary air, and thence to a waste-heat boiler. The sensible heat of the water-gas produced during the "run" is utilised in carbonising the coal in the upper part of the generator. The coal used should have an ash of high m.p. It must, moreover, while passing through the plastic stage, offer little resistance to the passage of gas. The suitability of a coal for this process is judged by (a) a crucible coking test; (b) a determination of caking power by Kattwinkel's method (B., 1926, 257, 905), but using only 4 pts. of sand to one of coal; and (c) measurement of the maximum pressure required to force a current of nitrogen through a column of the coal which is heated over the plastic range (cf. Layng and Hathorne, B., 1925, 195). The blow gases are relatively high in hydrogen and carbon monoxide because of the high reactivity and volatile matter content of the coke entering the gasifying zone. For efficiency of operation the steam used should be highly superheated, otherwise an undesirable amount of combustible matter is discharged with the ash. The losses during stoppages are greater than in a water-gas generator, and it is, therefore, desirable to operate the plant continuously, controlling the gas output by regulating the rate of supply of steam during the "run." Loss of gas during the clinkering period is avoided by the provision of a special exhaustor, which is operated during that period, and adds the gas then evolved to the total gas. Some experimental results illustrating different methods of running the plant are tabulated; they show the possibility of raising the rate of production from 30,000, the guaranteed rate, to 50,000 m^3 of gas per 24 hrs. The thermal efficiency of the plant averages 65%, the calorific value of the gas being about 4000 kg.-cal./ m^3 .

A. B. MANNING.

Law of flame speeds. II. Law applied to mixtures of combustibles having different theoretical flame propagation temperatures. Y. NAGAI (J. Soc. Chem. Ind., Japan, 1930, 33, 120—125 B; cf. A., 1930, 1000).—Generally, the law previously proposed does not hold, but it can be applied to those gaseous mixtures containing a sufficient amount of one combustible, so that the second combustible has the same theoretical flame propagation temperature as the first. This application of the law is supported by the work of Bone (A.,

1927, 26, 630) and Payman (B., 1923, 436 A), and is further demonstrated by experiments to determine the concentrations of hydrocarbons and lead tetraethyl in gaseous mixtures containing air having uniform flame speeds of 65 cm./sec. and of 35 cm./sec.

S. K. TWEEDY.

Determination of viscosity on small samples of tar. F. M. POTTER and J. S. SACH (Roads, 1930, 8, 231—232).—The apparatus described by Mallison (B., 1921, 197 A) is shown to provide a rapid means of checking the viscosities of tars, and the precautions necessary to obtain consistent results are discussed.

D. G. MURDOCH.

Origin and formation of acetic acid of pyroligneous liquor. II. C. PADOVANI and A. MARIOTTI (Annali Chim. Appl., 1930, 20, 205—210).—Experiments similar to those on Libyan esparto (B., 1929, 309) have been made on beech wood of the percentage composition: moisture 12.95, ash 1.49, fats and waxes 2.132, cellulose 52.94, lignin 30.49, methoxyl 1.45. Ordinary destructive distillation gave: charcoal 29.5, pyroligneous liquor 41.9, acetic acid 4.5%. The woody material offers greater resistance than esparto to hydrolysis by cold sodium hydroxide solution under 5 mm. pressure. The percentage of acetic acid, calculated on the weight of wood taken, reaches the value about 6.6 when the concentration of the alkali is raised to about 9% and then remains sensibly constant up to 12% alkali. The maximum proportions of acetic acid removed by the alkali solution for beech wood and esparto are very nearly in the ratio of the lignin contents of the two materials (cf. Pringsheim and Magnus, A., 1919, i, 473). Distillation of the dried residue from the alkali treatment yields about 1.6% of acetic acid (on the original wood), the total yield of acetic acid being thus 8.2%. Digestion of the wood with 2.5% sodium hydroxide solution under 9 atm. pressure (180°) yields 8% of acetic acid, this being increased to 9.5% by distillation of the dried residue. The so-called lignin appears to be composed of two groups of quite distinct components, some (including preformed acetyl groups) being removable by mild hydrolysis in the cold, whereas others are detachable from the cellulose only by more violent and disruptive processes. The amount of methoxyl groups in the alkali solution does not correspond with the difference between the amounts in the original wood and in the residue, but diminishes rapidly as the action proceeds, so that methoxyl groups are destroyed during the attack of the wood and the subsequent concentration of the liquors.

T. H. POPE.

Heat calculation for flash distillation [of petroleum hydrocarbons]. G. H. FANCHER (Chem. Met. Eng., 1930, 37, 307—308).—In calculating the heat added during vaporisation of hydrocarbons by means of Turner and Harrell's equation (B., 1930, 357), more reliable results can be obtained by taking into consideration the average sp. gr. and the temperature rather than the mol. wt. of the vapour formed. The value of the latent heat used in this calculation should be obtained from the relation $l = 1/d \cdot (110.9 - 0.09 t)$, where l is the latent of vaporisation in B.Th.U. per lb., d the sp. gr. of the liquid vaporised at 60°/60° F., and t

is the temperature in ° F.; this value is more accurate than that obtained from Hildebrand's or Trouton's rules.

D. K. MOORE.

Stabilisation of the knock value, gum content, and colour of gasoline by chemical inhibitors. G. EGLOFF, W. F. FARAGHER, and J. C. MORRELL (Petroleum, 1930, 26, 243—249).—The oxidation products, e.g., peroxides, acids, aldehydes, and ketones, which are formed in benzine on being kept lower the knock value. In a series of tests on Pennsylvanian cracked benzines, accelerated oxidation was found to decrease the benzol equivalent from 40% to 29%. The effect of chemical inhibitors on Mid-continent and Pennsylvanian cracked benzines shows a decrease of the knock value during accelerated oxidation. Such inhibitors are aniline, dimethylaniline, quinol, urea, anthracene, etc. The gum formation on keeping depends on the quantity of inhibitor added, the presence of water, the type of container, the oxidation medium, and on the effects of temperature and light. Oxidation by air or oxygen causes an increase in gum formation. Results are tabulated showing the effect of accelerated oxidation tests on gasolines with or without added inhibitors. The latter prevent gum formation. The colour is influenced by elementary sulphur and no colour stabiliser is known for benzine which has been kept. The anti-knock properties of cracked gasolines can be restored by treating them with fuller's earth, caustic soda, borax, etc.

W. S. E. CLARKE.

Motor oils from redistillation of mazout distillates from naphthenic-base crude oils. I. KLIGERMAN (Azerbaid. Neft. Choz., 1930, No. 2, 77—83).—High-grade lubricating oils are obtainable.

CHEMICAL ABSTRACTS.

Sludge of transformer oils. III. Catalytic effects of several metals on the formation of sludge and acids. T. YAMADA (J. Soc. Chem. Ind., Japan, 1930, 33, 113—114 B; cf. B., 1930, 542).—The relative catalytic influence of a number of metals on the formation of sludge and of acid in transformer oils has been measured by heating the oil at 140° for 100 hrs. in contact with air and with the metal. With refined oil many metals, and notably lead, zinc, iron and magnesium, tend to reduce the acidity of the oil; this, however, is due in part to the formation of metallic salts. With less highly refined oil copper, brass, silver, and lead increase considerably the quantity of sludge formed, whereas magnesium, tin, aluminium, and zinc have the reverse influence. Metals which tend to accelerate sludge formation increase, in general, the formation of acid.

H. F. GILLBE.

[Refining of petroleum oil crudes by] the Edelmann process. J. ROSENBERG (Petroleum, 1930, 26, 137—141).

Heat transmission. FISHENDEN. **Autoclaves.** HARRISON. **Fire extinguishers.** BIESALSKI.—See I.

PATENTS.

Apparatus for pulverising coal and like substances. ASHINGTON COAL CO., LTD., and W. J. DRUMMOND (B.P. 328,437, 24.4.29).—A pulverising mill in which the finely-divided particles are removed from the

grinding zone by means of a current of air is provided with a number of openings in the track where the heavier particles tend to accumulate; these lead to receptacles in which the heavy particles can collect and from which they are periodically and automatically discharged. A current of air is admitted into each receptacle in such a manner as to return any lighter or fully-ground particles back to the mill.

A. B. MANNING.

Rotary retort for low-temperature carbonisation. W. HAUSWALD and E. MILDNER (G.P. 461,703, 21.4.26).—The material to be carbonised is fed into tubes which consist of three sections of different diameter, fitting one into the other. The tubes are subdivided by partitions in the rotating drum in such a manner as to form a carbonising zone which is externally and internally heated. A method of constructing the retort is described.

A. B. MANNING.

Internally heated low-temperature carbonisation plant. METALLBANK U. METALLURG. GES. (Aust. P. 109,716, 4.10.26. Ger., 24.10.25).—The gaseous heating medium leaving the carbonising zone is freed from tar and passed through a multiple-stage countercurrent cooler in such a manner that while the whole of the gas is passed through the first stages of the cooler, only the excess gas which is being withdrawn from the circulating system is passed through the other stages.

A. B. MANNING.

Continuous gasification of granular or pulverulent material, such as pit coal, brown coal, coke, etc. KOHLENVEREDLUNG A.-G. (B.P. 311,775, 15.5.29. Ger., 16.5.28).—The finely-granular material together with steam, air, and/or oxygen is passed through parallel juxtaposed passages formed by dividing up a rotary drum by means of chequer bricks, the material being simultaneously heated to gasification temperatures. The heat required for gasification may be supplied from an external source, *e.g.*, by passing a heating gas along passages formed between the reaction passages by the chequer bricks, or from the combustion of part of the raw material.

A. B. MANNING.

Utilisation of the quenching vapours periodically resulting from the quenching of glowing coke in a pressure vessel. BAMAG-MEQUIN A.-G., and O. HELLER (B.P. 328,852, 25.6.29).—The mixture of steam and water-gas produced when the glowing coke is quenched in a pressure vessel is passed through the water contained in a steam accumulator. By means of an automatic pressure valve the pressure in the steam space of the accumulator is maintained at a value higher than that corresponding with the temperature of the water, and in consequence the entering steam is almost completely condensed, while the water-gas passes through the valve to the points of consumption. During the succeeding intermediate period the steam which has accumulated in the water of the accumulator is drawn off through a separate pipe. Several alternate arrangements of the apparatus for carrying out the process are described.

A. B. MANNING.

Purification of graphites. E. RABETRANO (B.P. 328,697, 25.6.29. Fr., 10.5.29).—The impure graphite is treated with hot, dilute sodium hydroxide under

pressure, and then, after washing, with boiling dilute acid, preferably sulphuric acid.

A. B. MANNING.

Gas generators or producers. HUMPHREYS & GLASGOW, LTD., and A. G. GLASGOW (B.P. 329,146, 4.5.29).—A mechanically clinkered gas generator is provided with one or more pockets for receiving the clinker and ash. The inlet to and outlet from each pocket are interlocked in such a manner that either can be opened only when the other is completely closed. The outlet closure may also be interlocked with the operating gear of the plant so that it cannot be opened while gas is being made in a downward direction through the generator.

A. B. MANNING.

Water-gas generators. D. TYRER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 328,642—3, 25.1.29).—(A) A deep coke bed is provided in the generator and the air is supplied thereto through several tuyères at different depths, the blow gases being withdrawn at the base. Steam is then supplied to traverse the whole depth of the bed in an upward direction. The blow gases are passed through a combustion chamber, wherein any carbon monoxide is burned with secondary air, and then through a regenerator which is used for preheating the steam. A second regenerator, through which the water-gas is passed during the "make," serves to pre-heat the air during the "blow." (B) The combustion chamber consists of an annular jacket surrounding the lower portion of the generator. It is separated from the coke bed by a thin wall and communicates with the generator through ports near its bottom end.

A. B. MANNING.

Control of gas-making plants. W. T. GLOVER and H. J. TOOGOOD (B.P. 329,167, 16.5.29).—The gas main is provided with a compartment in which the liquor can collect and which communicates with a vessel having an adjustable overflow. The cross-sectional area through which the gas can flow in the compartment is determined by the height of the liquor therein, *i.e.*, by the height of the overflow; this is controlled by means of a movable bell which is exposed on one side to the atmospheric pressure and on the other to the gas pressure in the main.

A. B. MANNING.

Production of mixed oil gas and water-gas. HUMPHREYS & GLASGOW, LTD., Assees. of (A) H. G. TERZIAN, and (C) E. L. HALL (B.P. 329,245—6 and 329,598, 11.9.29. U.S., [A] 8.11.28, [B] 3.1.29, [C] 29.12.28).—(A) The fuel bed in the generator is intermittently air-blasted and the blast gases are used for heating the carburettor and superheater. Between air-blasting operations the heat stored therein is utilised to vaporise, or vaporise and lightly crack, liquid hydrocarbons. The resultant oil vapours and oil gas are then passed through the fuel bed in the generator to complete their cracking, the carbon thereby formed being deposited in the fuel bed. At suitable intervals the fuel bed is blasted with steam, the blue water-gas produced being carburetted if desired. (B) Means are provided for extracting tar from the oil gas, produced as above described, both before and after its passage through the fuel bed. (C) The generator fuel bed is blasted with air, the resultant gases being burned in the carburettor and superheater. The step is followed either by a water-gas

production step or by a re-formed oil-gas production step. The latter consists in supplying oil and previously made gas together to the superheater, the gas being used to carry the resulting oil vapours and oil gas through the carburettor and then through the fuel bed. The water-gas produced may be carburetted in the usual manner, if desired. A. B. MANNING.

Continuous production of water-gas, or of producer gas rich in hydrogen or carbon monoxide, from powdered fuel. K. FISCHER (G.P. 461,481, 27.11.24).—The fuel is gasified while suspended in the gasifying agent, the necessary heat being supplied by radiation from a flame maintained in the producer. The sensible heat of the waste gases from the radiating flame may be utilised for heating a boiler or industrial furnace. A. B. MANNING.

Purification of gases. W. D'LENY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 329,135, 26.4.29).—Hydrogen sulphide and cyanogen are removed from gas by passing it in contact with an alkaline suspension of ferric hydroxide, the suspension being in the form of a continuous, freely falling, liquid film. The suspension may be recirculated, a part being withdrawn continuously for regeneration, which is effected by exposing it, again in the form of a freely falling film, to the action of air. A. B. MANNING.

Removal of ammonia and hydrogen sulphide from gases. C. J. HANSEN, Assec. of H. KOPPERS A.-G. (B.P. 316,278, 30.11.28. Ger., 28.7.28. Addn. to B.P. 309,116; B., 1930, 546).—The process described in the prior patent is modified by using the metal thionate liquor to scrub gas which contains ammonia and the metal hydroxide liquor to scrub ammonia-free gas. The two spent liquors are then mixed and re-divided into two parts for regeneration with sulphurous acid and oxidising gas, respectively. Accumulation of sulphur in the hydroxide liquor is thereby avoided, whilst the advantages of separate scrubbing operations are obtained. A. B. MANNING.

Separation of acetylene from mixtures of gases. SOC. CHEM. IND. IN BASLE, Assecs. of H. ANDRIESENS (B.P. 316,888, 2.8.29. Ger., 4.8.28).—Acetylene is separated from gas mixtures containing methane, hydrogen, and, perhaps, nitrogen by adsorption on activated carbon or similar adsorbent. Ethylene, if present, may be adsorbed at the same time. The adsorbent, when saturated with acetylene, is first evacuated in the cold whereby most of the other gases are removed without loss of acetylene. The latter is then recovered in a high degree of concentration by heating and evacuating the adsorbent. It is advantageous to carry out the adsorption at a low temperature, *e.g.*, -40° . A. B. MANNING.

Washing of gas liquors. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 328,388, 15.3.29).—Phenols and other organic substances are extracted from gas liquors by bringing the latter into intimate contact with tri-aryl or -alkyl phosphates, *e.g.*, tri-*o*-tolyl phosphate. The phenols are recovered by extraction of the phosphoric acid ester with dilute sodium hydroxide, and the bases and neutral oils are removed from the ester by subsequent steam-distillation. A. B. MANNING.

Distillation of tar at a gas-retort plant. BARRETT Co., Assecs. of S. P. MILLER (B.P. 309,946, 16.2.29. U.S., 18.4.28. Cf. B.P. 303,166; B., 1930, 545).—The gases from a number of coal-distillation retorts are passed directly into a single vertical stand-pipe, which is heat-insulated. Tar or pitch is brought into intimate contact with the hot gases in the stand-pipe and is thereby distilled. The residual pitch is withdrawn from the bottom of the stand-pipe, while the gases and vapours, after a cleaning treatment to remove suspended pitch if necessary, are passed to the condensing system. A. B. MANNING.

Production of higher tar yields in carbonisation processes. P. JUNG (G.P. 461,425, 4.6.24).—The fuels are mixed with chlorides of the earth or alkaline-earth metals, ammonium chloride, ferric chloride, etc., either before or during carbonisation. Metals also may be added to the mixture, which, if desired, may be briquetted before carbonisation. A. B. MANNING.

Preparation of [rubberised]-fabric coating composition. E. I. DU PONT DE NEMOURS & Co. (B.P. 303,368, 29.12.28. U.S., 31.12.27).—Asphalt, petroleum residue, stearine pitch, or other bituminous material is cracked, and the distillate is fractionated or "bodied," *i.e.*, heated in an open container, to obtain a product of suitable viscosity. A coating composition for rubberised fabrics is prepared by mixing this product with a pigment, a drying oil, and a thinner. Part or all of the drying oil in the mixture may be replaced by asphalt, petroleum flux, or other bituminous material. A. B. MANNING.

Feeding pastes of coal and oil or the like into hydrogenation converters. W. R. TATE, H. P. STEPHENSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 329,044, 21.2.29).—The paste or slurry of coal and oil is continuously circulated through the storage vessel in order to prevent settling, and is withdrawn as required from the circulating system for injection into the hydrogenation converter. A single pump may serve the dual purpose of feeding the injector and circulating the slurry. A. B. MANNING.

Destructive hydrogenation of coal, tars, mineral oils, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,992, 5.1.29. Cf. B.P. 326,184; B., 1930, 498).—In order to maintain the finely-dispersed catalysts continuously in uniform suspension in the liquids or pastes to be hydrogenated, the difference in sp. gr. between the material to be treated and the catalysts is reduced either by making additions of suitable sp. gr. to the former or by incorporating a suitable carrier, *e.g.*, active carbon, powdered pumice, with the latter. A. B. MANNING.

Recovery of refined products from carbonaceous materials such as coal, tars, mineral oils, and the like. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,997, 12.11.28).—The reaction products from the hydrogenation under pressure of coal, oil, tar, etc. are fractionated by passing through them, in a suitable column, gases, vapours, or liquids having lower temperatures than the reaction products. A. B. MANNING.

Depolymerisation of hydrocarbons. J. MERCIER (B.P. 302,899, 7.12.28. Fr., 22.12.27).—The hydro-

carbon is vaporised and the vapours are passed up through a zig-zag retort containing a depolymerising catalyst. The successive segments of the retort extend across a vertical shaft, which is traversed by a heating medium; a series of baffles within the shaft direct the flow of heating medium along the segments of the retort. The upper section of the retort is heat-insulated. The vapours issuing from the retort pass into a cooling and expanding chamber, which may also contain a catalyst. The heavy oils condensing therein may be returned for re-treatment; the uncondensed vapours may proceed to a second expansion and cooling unit, or to the bottom of a fractionating tower. A. B. MANNING.

Catalytic synthesis of liquid and gaseous hydrocarbons. A. FOLLIET (F.P. 637,891, 19.7.27).—Mixtures of hydrocarbons, oxides of carbon, and, if desired, hydrogen, are heated at 500–1000° under 5–100 atm. pressure in a metal apparatus encased in a jacket of refractory material, and, after being cooled if necessary, are allowed to expand suddenly into the catalyst chamber. A. B. MANNING.

Ketone fuels. SOC. DES BREVETS ÉTRANGERS LEFRANC ET CIE. (F.P. 637,993, 30.11.26).—The fuels consist of complex ketones produced by the dry distillation of calcium salts, *e.g.*, calcium butyrate, and liquid fuels obtained synthetically (methyl alcohol), by fermentation, or by the distillation of carbonaceous material (benzol). A. B. MANNING.

Purification of [used mineral] oils. L. H. CLARK, ASSR. to SHARPLES SPECIALTY CO. (U.S.P. 1,747,161, 18.2.30. Appl., 11.7.25).—Mineral oils containing decomposition products, *e.g.*, used transformer or switch oils, are emulsified with about 1% of an activated carbon and an aqueous alkaline reagent, *e.g.*, a solution of caustic soda or sodium silicate; the renovated oil is separated by centrifuging. E. LEWKOWITSCH.

Apparatus for gasifying and distilling solid fuel. E. ROSER (U.S.P. 1,765,702, 24.6.30. Appl., 31.12.25. Ger., 6.7.25).—See B.P. 257,479; B., 1926, 972.

Coke oven. E. LECOCQ, ASSR. to SOC. GÉN. DE FOURS À COKE SYSTÈME LECOCQ SOC. ANON. (U.S.P. 1,761,835, 3.6.30. Appl., 23.3.25. Belg., 29.3.24).—See B.P. 231,483; B., 1926, 4.

Carbonisation of coal. W. E. TRENT, ASSR. to TRENT PROCESS CORP. (U.S.P. 1,767,778, 24.6.30. Appl., 27.2.25).—See B.P. 261,954; B., 1927, 99.

Distillation of carbonaceous substances. E. R. SUTCLIFFE (U.S.P. 1,767,231, 24.6.30. Appl., 16.7.24. U.K., 25.7.23).—See B.P. 227,879; B., 1925, 235.

Bleaching of montan wax. T. HELLTHALER, ASSR. to RIEBECK'SCHE MONTANWERKE A.-G. (U.S.P. 1,767,886, 24.6.30. Appl., 18.7.27. Ger., 26.7.26).—See B.P. 303,036; B., 1929, 198.

Emulsions from partially oxidised petroleum wax and their manufacture. A. W. BURWELL, ASSR. to ALOX CHEM. CORP. (U.S.P. 1,768,523, 24.6.30. Appl., 23.3.27).—See B.P. 287,514; B., 1929, 348.

Screens employed for the screening of coal. W. H. BERRISFORD (B.P. 330,554, 9.3.29).

Crushing machine (U.S.P. 1,750,573). **Separation of materials** (U.S.P. 1,750,090).—See I. **Tar-stained wool** (B.P. 309,871).—See VI. **Oxidation of ammonia** (B.P. 309,583).—See VII. **Making roads etc.** (B.P. 330,072 and 330,440). **Covering roads etc.** (B.P. 310,922).—See IX. **Insulating oil** (B.P. 301,876).—See XI. **Emulsions containing rubber** (B.P. 329,965).—See XIV. **Sugar for fermentation** (B.P. 330,275).—See XVII.

III.—ORGANIC INTERMEDIATES.

Absorption of organic solvents by washing with liquids. E. BERL and L. RANIS (Z. angew. Chem., 1930, 43, 600–603).—The relations between vapour pressure and concentration of various organic liquids absorbed in cresol, sulphuric acid, and paraffin oil were determined by means of the Haber-Löwe gas interferometer. The heats of mixing of the different pairs of liquids were also determined, and the theoretical vapour pressures according to Planck's formula, which assumes a process of dissolution without chemical action, were calculated. A large number of liquid pairs are thus classified into three groups: (1) those in which the partial pressures correspond to the formula; (2) those in which they are greater than those indicated by theory, owing to the splitting of associated molecules; and (3) those in which they are less, owing to the formation of a compound. The form of the partial pressure-concentration curve, whether linear, convex, or concave, and the heat evolved on mixing in some cases afford supporting evidence. In some cases, *e.g.*, cresol-tetrachloroethane, contradictory results were obtained by the different methods, and here it is suggested that both disassociation and combination may occur. Absorbents which lower the vapour pressure of the more volatile liquid are naturally the most efficient.

C. IRWIN.

Detection of ethyl *o*-phthalate. J. SUCHODOLSKI (Przemysl Chem., 1930, 14, 265–267).—The presence of ethyl *o*-phthalate in alcoholic solutions can be detected by the following method: the solution is evaporated to dryness with 1 c.c. of 1% sodium hydroxide, 5 c.c. of concentrated sulphuric acid are added to the residue, and, after heating for a further 5–10 min. at 100°, 25–30 mg. of resorcinol are added and the whole is heated for 5 min. at 160–170°. The liquid is then dissolved in 100 c.c. of water, and 50 c.c. of sodium hydroxide solution (*d* 1.4) are added. Under the above conditions permanent fluorescence appears only if ethyl *o*-phthalate was originally present; essential oils, and other substances ordinarily present in perfumes, give either a transient fluorescence or none at all.

R. TRUSZKOWSKI.

Utilisation of soya beans. III. Y. TAKAYAMA (J. Soc. Chem. Ind., Japan, 1930, 33, 91–92B).—Results are summarised of the experiments previously described (*cf.* B., 1928, 529) on the separation of glutamic acid (as the dibasic calcium salt) from the products of hydrolysis (with 25–50% sulphuric acid) of crude soya-bean protein. Hydrolysis with more concentrated acid gave greater yields. The solubility of dibasic calcium glutamate in water was found to be 1.322, 1.928, 1.979, 3.943, and 5.698 g. of anhydrous salt per 100 g.

of solution at 0°, 19°, 21°, 61°, and 100° respectively. Incipient decomposition, with liberation of ammonia (as sulphate), was observed on prolonged heating of pure glutamic acid with 10*N*-sulphuric acid at 100° and 135–145°. E. LEWKOWITSCH.

Fractionating column. McCULLOUGH and GITTINGS.—See I. **Acetic acid from pyroligneous liquor.** PADOVANI and MARIOTTI.—See II. **Glycerin by fermentation.** TOMODA.—See XVIII.

PATENTS.

Manufacture of acetaldehyde. C. N. HAND, T. W. BARTRAM, and A. H. MAUDE, Assrs. to RUBBER SERVICE LABS. CO. (U.S.P. 1,738,649, 10.12.29. Appl., 12.4.26).—Acetylene at 25 lb./in.² is passed into 25–30% sulphuric acid at 68°, sufficient mercurous sulphate being added to maintain the temperature at 68–80°. Entrained water and acid are removed from the exit gas, and the acetaldehyde is separated from the excess acetylene. C. HOLLINS.

Production of (A) formic acid (B) formaldehyde [from carbon monoxide and water]. W. C. ARSEM, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,740,140—1, 17.12.29. Appl., [A] 12.9.25, [B] 3.2.26).—(A) The carbon dioxide (55%) and hydrogen (45%) evolved from butyl-acetonic fermentation is led under pressure over a mixed catalyst (reduced copper with alumina, thoria, etc.) and the resulting mixture of carbon monoxide and water vapour is converted into formic acid by passage over a cuprous chloride catalyst at 50–150° and 20 atm. or more. (B) Carbon monoxide and water vapour are passed over zinc oxide at 150–350° and 10–100 atm. Other weak basic oxides may be used. About 10% conversion is obtained. C. HOLLINS.

Manufacture of alkyl halides. R. H. MCKEE and S. P. BURKE (U.S.P. 1,738,193, 3.12.29. Appl., 8.8.27).—An alcohol vapour is passed with hydrogen halide over chloride of magnesium, calcium, or barium at about 300°. C. HOLLINS.

Manufacture of furyl alcohol and methylfuran. E. RICARD and H. M. GUINOT, Assrs. to SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (U.S.P. 1,739,919, 17.12.29. Appl., 29.12.27. Fr., 31.1.27).—Hydrogenation of furfuraldehyde in presence of reduced copper at 140° gives mainly furyl alcohol, with 10–20% of methylfuran which is easily removed by distillation; excess furfuraldehyde distils as azeotropic mixture with water and may be returned to the process. The furyl alcohol can be reduced to methylfuran by repeating the hydrogenation. C. HOLLINS.

[Croton]aldehyde manufacture. A. H. MAUDE, Assr. to RUBBER SERVICE LABS. CO. (U.S.P. 1,738,659, 10.12.29. Appl., 29.6.27).—Aldol is heated at 160° with 3% of 85% phosphoric acid and 1% of aluminium hydroxide, *i.e.*, a mixture of phosphoric acid and aluminium phosphate formed *in situ*. High yields of crotonaldehyde are claimed. C. HOLLINS.

Manufacture of phenols. W. J. HALE and E. C. BRITTON, Assrs. to DOW CHEM. CO. (U.S.P. 1,737,841—2, 3.12.29. Appl., [A] 21.2.23, [B] 23.9.27).—(A) Diphenyl

ether and aqueous sodium phenoxide solution are heated above 350° in an autoclave for 15 min. The layer of free phenol is removed from the reaction product after cooling, more diphenyl ether (or chlorobenzene, which produces the ether during the heating up) is added, and the process repeated. (B) Diphenyl ether is hydrolysed with aqueous solutions of sodium salts of weak acids, *e.g.*, sodium carbonate, borax, di- or tri-sodium phosphate, having alkaline reaction, or with a suspension of lime. The presence of chlorobenzene in the ether is advantageous. C. HOLLINS.

Manufacture of phosphoric acid esters. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,963, 31.1.29. Addn. to B.P. 300,044; B., 1929, 10).—The process of the prior patent is applied to the production of mixed alkyl or mixed aryl alkyl phosphates, *e.g.*, *n*-butyl diisoamyl phosphate, b.p. 121–122°/1.5 mm., diethyl isoamyl phosphate, b.p. 84–85°/2 mm., and phenyl dibutyl phosphate, b.p. 161°/4 mm. C. HOLLINS.

Manufacture of carbazole derivatives. I. G. FARBENIND. A.-G. (B.P. 328,933, 4.1.29. Ger., 5.1.28).—2-Chlorocarbazole, m.p. 244°, carbazolecarboxylic acid, m.p. 271–272°, and carbazole-2-sulphinic acid are obtained by the usual methods from diazotised 2-amino-carbazole. (Cf. B.P. 303,520; B., 1930, 603.) C. HOLLINS.

Preparation of disubstituted tetrazoles. A. BOEHRINGER (B.P. 309,949, 5.4.29. Ger., 18.4.28).—Imino-esters, R·C(OAc):NR', in which Ac represents an organic or inorganic acid residue, are treated with hydrazine, and the resulting hydrazidines are converted by nitrous acid into 1:5-disubstituted tetrazoles. The imino-esters are preferably made *in situ*, *e.g.*, from oxime esters or *N*-substituted acid amides; the hydrazidines also are not isolated. *cyclo*Hexanoneoxime *p*-toluenesulphonate in benzene is warmed at 30–35° with alcoholic hydrazine acetate, and the hydrazidine solution so obtained yields with nitrous acid 1:5-pentamethylene-1:2:3:4-tetrazole, m.p. 59°, b.p. 192°/10 mm., similarly obtainable from the *o*-benzenesulphonate of β -leucine lactam. C. HOLLINS.

Manufacture of adhesive and binding agents. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,908, 1.11.28 and 22.3.29).—Butadiene with or without an organic diluent (aniline, dimethylaniline, tetrahydrostyrene), or isoprene with a diluent, is heated at 100–200°, the reaction being stopped before solid is formed; the oily product may be hardened below 100°, if desired. Application to paint and glue is indicated. [Stat. ref.] C. HOLLINS.

Production of gases for the manufacture of oxygenated organic compounds [methyl alcohol]. M. PIER, M. MÜLLER-CUNRADI, G. WIETZEL, and K. WINKLER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,763, 24.6.30. Appl., 15.9.23).—See B.P. 228,959; B., 1925, 299.

Concentration of acetic acid. G. SCHWAEBEL and F. SCHLEICHER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,404, 24.6.30. Appl., 10.5.27. Ger., 12.5.26).—See B.P. 295,238; B., 1928, 746.

Production of lactic acid and its derivatives.

M. LUTHER and H. BELLER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,715, 24.6.30. Appl., 4.2.28. Ger., 12.2.27).—See B.P. 290,464; B., 1928, 543.

Manufacture of [crystalline] urea.

W. MEISER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,761,893, 3.6.30. Appl., 17.2.27. Ger., 19.2.26).—See B.P. 266,378; B., 1928, 255.

Production of phosphoric esters of aliphatic

alcohols. F. NICOLAI, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,720—1, 24.6.30. Appl., [A] 4.9.28, [B] 4.9.29. Ger., [A, B] 12.10.27).—See B.P. 300,044; B., 1929, 10.

Production of thymol.

H. JORDAN, Assr. to CHEM. FABR. AUF ACTIEN VORM. E. SCHERING (U.S.P. 1,768,257, 24.6.30. Appl., 15.8.27. Ger., 16.8.26).—See B.P. 276,010; B., 1929, 237.

[Manufacture of] formaldehyde-bisulphite [derivatives of] aminoaryl antimony compounds [stibinic acids].

H. SCHMIDT, Assr. to WINTHROP CHEM. CO., INC. (U.S.P. 1,766,361, 24.6.30. Appl., 28.9.28. Ger., 6.10.27).—See B.P. 298,234; B., 1930, 394.

Manufacture of *vic.* - trihalogenobenzenes.

R. HERZ and E. ALBRECHT, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,766,747, 24.6.30. Appl., 29.8.28. Ger., 5.10.27).—See B.P. 319,149; B., 1930, 95.

Process for introducing sulphocyanic groups

into organic compounds. H. P. KAUFMANN and M. SCHUBERT, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,765,678, 24.6.30. Appl., 6.3.28. Ger., 11.3.27).—See B.P. 303,813; B., 1929, 236.

Intermediates derived from aminosulphones.

K. H. SAUNDERS, Assr. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,766,951—2, 24.6.30. Appl., [A] 31.8.28, [B] 10.10.29. U.K., 21.10.24).—See B.P. 245,865; B., 1926, 233.

Preparation of sulphur-containing hydroxy-

quinones. F. MAYER and K. ZAHN, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,765,687, 24.6.30. Appl., 11.8.28. Ger., 7.9.27).—See B.P. 296,761; B., 1929, 807.

Manufacture of condensation products of the

benzanthrone series. G. KALISCHER, H. SCHEYER, P. NAWIASKY, and E. KRAUCH, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,768,071, 24.6.30. Appl., 17.6.27. Ger., 28.6.26).—See B.P. 296,490; B., 1928, 847.

Manufacture of intermediate compounds and of

azo dyes therefrom. M. MENDOZA, Assr. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,766,949, 24.6.30. Appl., 25.5.28. U.K., 27.5.27).—See B.P. 297,855; B., 1928, 887.

Hexamethylenetetramine-triphenol for light

filters (U.S.P. 1,751,220).—See I. **Depolymerisation of hydrocarbons** (B.P. 302,899). **Ketone fuels** (F.P. 637,993).—See II. **Vat dye intermediates** (B.P. 305,489).—See IV. **Oxidisable compounds** (B.P. 316,251).—See XIV. **Polyhydric alcohols** (B.P. 327,193).—See XVII.

IV.—DYESTUFFS.**Colour lakes.** CLAYTON.—See XIII.**PATENTS.****Manufacture and use of dyes [derived from naphthazarin].**

BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLFIN, and E. W. KIRK (B.P. 327,860, 1.8.28).—Naphthazarin or a derivative, *e.g.*, methylnaphthazarin (obtained from toluquinol and succinic anhydride), is condensed with aqueous ammonia, preferably in presence of a reducing agent (or the leuco-naphthazarin may be used) and a copper salt. Grey to violet-grey dyes for acetate silk are obtained. C. HOLLINS.

Manufacture of vat dyes of the anthraquinone

series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 328,248, and 328,793, [A], 12.1.29, [B] 1.5.29).—(A) A 1 : 1'-dianthraquinonylamine, containing a 4 : 3-thioxanthone ring and in the other anthraquinone nucleus either a 3' : 4'-acridone ring or an acylamino-group, is treated at 20° with sulphuric acid to give a carbazole derivative. 4-Aminoanthraquinone-2 : 1-thioxanthone is condensed with 5-chloro-1-benzamidoanthraquinone or 4-chloro-5-methoxy-1-benzamidoanthraquinone and the product is carbazolised to give a black-brown vat dye. A grey-green vat dye is obtained by carbazolisation of the product from 4-aminoanthraquinone-2 : 1-thioxanthone and 4 : 3' : 5'-trichloroanthraquinoneacridone. (B) 1-Chloroanthraquinone-2-carboxylic acid is condensed with 2-amino- or 2 : 6-diaminoanthracene and the product is cyclised to an anthraquinoneacridone or anthraquinonediacridone, both of which are red-brown vat dyes. C. HOLLINS.

Manufacture of vat dyes of the anthraquinone

series. I. G. FARBENIND. A.-G. (B.P. 305,488, 4.2.29. Ger., 3.2.28).—Phenanthridone is condensed with phthalic anhydride in presence of aluminium chloride at 180—230° to give two isomeric phthaloylphenanthridones, separable by crystallisation from nitrobenzene or trichlorobenzene. The main product is a yellow vat dye; the less soluble product gives paler shades. C. HOLLINS.

Manufacture of vat dyes of the benzanthrone

series. I. G. FARBENIND. A.-G. (B.P. 305,679, 9.2.29. Ger., 10.2.28, and Addn. B.P. 328,993, 9.2.29).—(A) Benzanthrone-1-aminoanthraquinones, having a free 2-position and an aroylamino-substituent in the anthraquinone residue, are treated with acid condensing agents (sulphuric or chlorosulphonic acid) to give brown vat dyes. Examples of starting materials are the products from 1-amino-5-benzamidoanthraquinone with 3-bromobenzanthrone, and 1-amino-4-benzamidoanthraquinone with 9-chlorobenzanthrone. (B) Aluminium chloride is used as condensing agent. C. HOLLINS.

Manufacture of vat dyes [of the dibenzanthrone

series]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,334, 27.11.28. Addn. to B.P. 307,328; B., 1929, 388).—Halogenated (*etc.*) dibenzanthrones and isodibenzanthrones are condensed with other amino-compounds than aminoanthraquinones, preferably those capable of being vatted; *e.g.*, bromodibenzanthrone with aminoanthanthrone (dark reddish-blue), bromoisodibenzanthrone with aminopyranthone (dark blue),

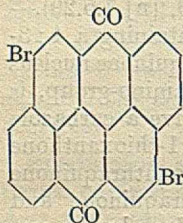
dibromodibenzanthrone with aminoanthanthrone (dark blue), chlorotetrabromoisodibenzanthrone with aminoanthanthrone (dark blue), dibromoisodibenzanthrone with the carbazole produced by heating 4-benzamido-1:1'-dianthraquinonylamine with sulphuric acid (violet).

C. HOLLINS.

Manufacture of halogen derivatives [vat dyes] of the dibenzanthrone series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,263, 4.6.29. Cf. B.P. 325,525; B., 1930, 453).—Dibenzanthrone or a bromo-derivative is brominated by grinding with bromine together with a carrier (iodine and iron). The product is a reddish-navy-blue vat dye.

C. HOLLINS.

Manufacture of vat dyes and intermediates of the anthanthrone series. I. G. FARBENIND. A.-G. (B.P. 305,489, 4.2.29. Ger., 3.2.28).—A halogenated anthanthrone is heated with sulphur or an agent yielding sulphur. 4:10-Dibromoanthanthrone (annexed formula) heated in nitrobenzene at 130–145° with potassium xanthate yields the 10:10'-dibromosulphide, which is a reddish-violet vat dye. C. HOLLINS.



Manufacture of vat dyes of the 3:4:8:9 [1:2:6:7]-dibenzpyrene -[3:8]-quinone series. I. G. FARBENIND A.-G. (B.P. 310,891, 2.5.29. Ger., 2.5.28).—Dihalogenated (particularly dibrominated) 1:2:6:7-dibenzpyrene-3:8-quinones are improved in shade and in fastness to washing by treatment with an alkaline oxidant, e.g., alkaline hypochlorite. C. HOLLINS.

Manufacture of vat dyes containing sulphur. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,705, 13.2.29).—Anthraquinonylthiol-benzanthrones or -dibenzanthronyls, other than those derived from 2- and 3-thiolbenzanthrones, are fused with alkali to give vat dyes. Dyes are thus obtained from 9- α -anthraquinonylthiolbenzanthrone (navy-blue), 9:9'-di- α -anthraquinonylthiol-2:2'-dibenzanthronyl (navy-blue), 1:1'- α -anthraquinonylthiolbenzanthrone (greenish-blue).

C. HOLLINS.

Manufacture of monoazo dyes [for wool]. I. G. FARBENIND. A.-G. (B.P. 305,174, 31.1.29. Ger., 1.2.28). An amine of the benzene series is diazotised and coupled with a sulphonic acid of 2-phenylindole. Examples are: *p*-aminoacetethylanilide \rightarrow 2-phenylindole-7-sulphonic acid (yellow); 2:4:6-trichloroaniline \rightarrow 2-*m*-sulphophenylindole (orange). The dyes are fast to fulling.

C. HOLLINS.

Manufacture of azo dyes [for wool, viscose silk, or acetate silk]. IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 329,049, 21.2.29).—A mono-oxamic acid of 4:4'-diaminodiphenyl sulphide, m.p. 108°, or trisulphide, m.p. 122°, is diazotised and coupled with a coupling component. The azo dye may be hydrolysed, diazotised, and coupled further, or in suitable cases may be coupled with diazo compounds. Examples are: the mono-oxamic acid, m.p. above 265°, of the monosulphide [4-*p*-aminophenylthiolphenyloxamic acid] \rightarrow phenyl-J-acid (bluish-red on viscose silk), 2S-acid (violet on viscose silk), phenyl- γ -

acid (brown on wool), phenylmethylpyrazole (yellow on acetate silk), Brönnner acid (orange on wool), γ -acid (red on wool or viscose silk), or *m*-tolylenediamine \leftarrow 2:4-dinitroaniline (red-brown on acetate silk); the trisulphide-oxamic acid, m.p. above 290°, \rightarrow salicylic acid (yellow on acetate silk or wool), J-acid (yellowish-red on viscose silk), or *m*-phenylenediamine \rightarrow *p*-chloroaniline (orange-brown on acetate silk). C. HOLLINS.

Manufacture of monoazo dyes [ice colours and pigments]. I. G. FARBENIND. A.-G. (B.P. 303,838, 10.1.29. Ger., 10.1.28).—A monoaroyl-*p*-phenylenediamine is diazotised and coupled in substance or on the fibre with a 2:3-hydroxynaphthoic arylamide. E.g., 6- α -naphthamidocresidine, m.p. 183–184°, obtained from 6-nitrocresidine, is coupled with the 5-chloro-*o*-toluidide for a bluish-violet. C. HOLLINS.

Manufacture of azo dyes [for lakes]. MAJOR & CO., LTD., H. H. HINCHLIFFE, and W. J. DARBY (B.P. 328,292, 19.1.29).—The monoazo dyes from diazotised toluidinesulphonic acids (e.g., *p*-toluidine-3-sulphonic and 5-chloro-*o*-toluidine-4-sulphonic acids) coupled with 2:3-hydroxynaphthoic acid are condensed with 1 or 2 mols. of an arylsulphonyl chloride (e.g., toluene-*p*-sulphonyl chloride) in presence of caustic alkali.

C. HOLLINS.

Manufacture of disazo dyes. E. F. HITCH, H. JORDAN, and A. O. BRADLEY, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,739,031, 10.12.29. Appl., 7.8.26).—A non-phenolic arylamine is diazotised and coupled with a non-phenolic middle component; the resulting aminoazo compound is nitrobenzoylated, reduced, diazotised, and coupled with a coupling component. Examples are: β -naphthylamine-6:8-disulphonic acid \rightarrow *m*-toluidine, *m*- or *p*-nitrobenzoylated, reduced, \rightarrow *p*-aminobenzoyl-J-acid (orange on cotton, redder by development with β -naphthol); Brönnner acid \rightarrow α -naphthylamine, *p*-nitrobenzoylated, reduced, \rightarrow S-acid (bluish-brown on cotton, full brown with β -naphthol, yellow-brown with phenylmethylpyrazolone); β -naphthylamine-6:8-disulphonic acid \rightarrow *o*-anisidine, *p*-nitrobenzoylated, reduced, \rightarrow 1-*m*-aminophenyl-3-methyl-5-pyrazolone (yellow on cotton); etc.

C. HOLLINS.

Manufacture of [dis]azo dyes and their application to the dyeing of regenerated cellulose materials. IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 329,056, 25.2.29).—A diaminodiphenyl, in which not more than one amino-group is in the 4-position, is tetrazotised and coupled with coupling components of which at least one is an aminonaphtholsulphonic acid or an *N*-derivative thereof; the diphenyl compounds must carry no phenolic, nitro-, carboxyl, or sulphonic groups. Examples are: 3:3'-diaminodiphenyl \rightarrow 2 mols. of γ -acid (brown); diphenylene \rightarrow 2 mols. of phenyl- γ -acid (brown); 2:2'-diaminodiphenyl with salicylic acid and phenyl- γ -acid (brown) or with *o*-cresotic acid and 1:5:7-aminonaphtholsulphonic acid (bluish-red); etc. The dyes give level shades on viscose silk.

C. HOLLINS.

Manufacture of azine dyes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 328,291, 18.1.29).—An *o*-phenylenediamine is condensed with an 8-acylamino-

1:2-naphthaquinone-4- or -5-sulphonic acid. The acyl groups may be removed by dilute sulphuric acid, the sulphonic group by concentrated acid; the amino-group may be aroylated. From *o*-phenylenediamine and 8-acetamido-1:2-naphthaquinone-5-sulphonic acid are obtained 1-acetamido- α -naphthaphenazine-4-sulphonic acid (greenish-yellow on wool), the 1-amino- (red-orange), 1-benzamido- (reddish-yellow), 1-*o*-chlorobenzamido- (orange), and 1-*m*- or -*p*-nitrobenzamido- (greenish-yellow) compounds, 1-amino- α -naphthaphenazine and its benzoyl (m.p. 218–219°), acetyl (m.p. 233–234°), and *p*-nitrobenzoyl (m.p. 305–307°) derivatives; *o*-phenylenediamine-4-sulphonic acid gives a mixture of 4:8- and 4:9-disulphonic acids (pure yellow on wool), from which the 1-amino- and 1-*p*-nitrobenzamido-disulphonic acids are obtained. 8-Acetamido-1:2-naphthaquinone-4-sulphonic acid yields similar products. The wool dyes are very fast to light; the desulphonated aminonaphthaphenazine may be used for dyeing acetate silk.

C. HOLLINS.

Yellow vat dyes. G. KRÄNZLEIN, H. GREUNE, and H. VOLLMANN, Asss. to GEN. ANILINE WORKS, INC. (U.S.P. 1,767,377, 24.6.30. Appl., 27.11.25. Ger., 16.2.24).—See F.P. 593,117; B., 1926, 266.

Preparation of [vat-dye] derivatives of 1:4:5:8-naphthalenetetracarboxylic acids. W. ECKERT and H. GREUNE, Asss. to GEN. ANILINE WORKS, INC. (U.S.P. 1,765,661, 24.6.30. Appl., 27.1.27. Ger., 9.2.26).—See B.P. 265,964; B., 1928, 225.

Alkaline condensation products [vat dyes] of Bz-1-ethers of benzanthrone. K. THIESS, W. GMELIN, and K. ZAHN, Asss. to GEN. ANILINE WORKS, INC. (U.S.P. 1,766,413, 24.6.30. Appl., 19.7.26. Ger., 26.8.25).—See B.P. 257,618; B., 1928, 83.

Anthraquinone dyes and dyeing therewith. A. SHEPHERDSON and W. W. TATUM, Asss. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,761,369 and 1,761,370, 3.6.30. Appl., [A] 9.2.27, [B] 27.1.28. U.K., [A, B] 19.8.26. Renewed [B] 21.10.29).—See B.P. 275,421; B., 1927, 771.

Azo dyes. H. WAGNER, O. SOHST, and J. RACHOR, Asss. to GEN. ANILINE WORKS, INC. (U.S.P. 1,765,640, 24.6.30. Appl., 27.8.28. Ger., 19.9.27).—See B.P. 297,362; B., 1930, 365.

Manufacture of fast azo dyes. L. LASKA and A. ZITSCHER, Asss. to GEN. ANILINE WORKS, INC. (U.S.P. 1,765,681, 24.6.30. Appl., 27.8.28. Ger., 5.9.27).—See B.P. 309,216; B., 1929, 552.

Manufacture of [chromable] azo dyes. K. H. SAUNDERS, Assr. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,766,947, 24.6.30. Appl., 10.6.26. Renewed 10.9.29. U.K., 4.8.25).—See B.P. 256,775; B., 1926, 866.

Azo dyes derived from aminosulphones. K. H. SAUNDERS, Assr. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,766,946, 24.6.30. Appl., 29.8.25. U.K., 21.10.24).—See B.P. 245,865; B., 1926, 233.

(A) **Mordant azo dyes.** (B) **[Dis]azo dyes.** M. MENDOZA and K. H. SAUNDERS, Asss. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,766,948 and 1,766,950, 24.6.30. Appl., [A] 29.9.26, [B] 31.8.28. U.K., [A, B] 13.11.25).—See B.P. 262,243; B., 1927, 101.

[Production of] naphthacarbazolehydroxyazo dyes. H. GRIMMEL and H. CLINGESTEIN, Asss. to GEN. ANILINE WORKS, INC. (U.S.P. 1,766,434, 24.6.30. Appl., 28.6.26. Ger., 26.6.25).—See F.P. 617,211; B., 1927, 902.

Production of complex metal compounds of α -hydroxyazo dyes. H. KRZIKALLA, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,765,680, 24.6.30. Appl., 27.8.27. Ger., 28.8.26).—See B.P. 296,819; B., 1928, 849.

Treatment of complex metallic compounds of α -hydroxyazo dyes. H. KRZIKALLA and H. KÄMMERER, Asss. to GEN. ANILINE WORKS, INC. (U.S.P. 1,767,379, 24.6.30. Appl., 24.3.27. Ger., 31.3.26).—See B.P. 268,754; B., 1928, 667.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Bark of the young woods of spruce, pine, and red beech. C. G. SCHWALBE and K. E. NEUMANN (Cellulosechem., 1930, 11, 113–128).—The properties of the cambial layers of the three woods have been investigated with a view of formulating a simple process for the removal of bark prior to pulping. Woods felled in spring and in autumn have been examined, and their chemical characteristics are differentiated. In the spring woods the cambial cells are soft and the bark is easily removed, the contrary being the case in the autumn woods, which are of the greatest practical interest. Extraction of all three woods with water gave negligible amounts of free sugars. By means of acid hydrolysis higher yields of sugars are obtained from the cambial layers of the coniferous woods than from the heart-wood, indicating higher concentration of hemicelluloses. Tests for pentoses among the sugars produced by hydrolysis gave negative results, hexoses alone being found. Similar treatment of the bark of the deciduous wood (beech) shows that the concentration of easily hydrolysed hemicelluloses is lower than in the body of the tree, as is also the pentosan content. The hexosan content being even lower, both in the bark and in the wood, it is held that the pentosans are very difficultly hydrolysed, concentrated acids or alkalis being necessary. Treatment with lime- and baryta-water failed to affect the pentosan content of beech bark, but in the case of the wood the content was lowered, due to the presence in the pentosan complex of acid groupings of low mol. wt. Extraction with ether shows the bark of both classes to yield larger extracts than do the corresponding woods, the ratio being higher in beech, although the absolute quantity is lower. In order to attempt to trace the differences in character of the barks of the two types of wood, the saps flowing through the trees in spring were investigated, without positive result.

T. T. POTTS.

Extraction of pure cellulose from plant raw materials (Reports 2–4). Pretreatment prior to chlorination. I.—III. K. KANAMARU (J. Soc. Chem. Ind., Japan, 1930, 33, 78–80 B, 80 B, 81–82 B).—The effect produced by various treatments prior to chlorination on the extraction of cellulose by Cross and Bevan's method has been investigated. I. The best pretreatment, in order to remove non-cellulosic material and to cause swelling of the cell-walls without loss or

degradation of cellulose, in the case of wood sawdust (previously extracted with ether and alcohol) is to immerse the material in the concentrated caustic soda solution at room temperature for 24 hrs. (Applied in the determination of cellulose, this method gives the purest product in maximum yield by the subsequent chlorination.) Purification with hot dilute caustic solution lowers the yield of cellulose and α -cellulose. Treatment with 20–60% calcium chloride solution at 60° is also effective. Pentosans can be removed completely by digesting a 1.2–1.3% suspension of α -cellulose in water for 70 hrs. at 35° with the powder (to the amount of 0.07–0.08% of malt extract) obtained by pouring a water extract of dry malt meal into alcohol (p_H adjusted to 4–5).

II. The best conditions (concentration, temperature, etc.) for the pretreatment are discussed theoretically; the experimental results are interpreted on the basis of the theories of von Weimarn on the dispersion of cellulose in salt solutions and of Katz on swelling.

III. Rice straw has been investigated as in I. The fibrous tissue of the straw is porous and contains a large amount of non-cellulosic material, so that pretreatment is itself a process of cellulose extraction: treatment with hot dilute caustic soda is the most effective, but partial loss of cellulose is unavoidable. Pretreatment of the straw with malt extract (which can be used to remove all pentosans) causes a loss of 6–8% in weight (mostly pentosans) and increases the yield of cellulose, but greatly diminishes the content of α -cellulose. Treatment with malt extract converts the cellulose of straw, wood, and cotton into a form soluble in alkali.

E. LEWKOWITSCH.

Wood-pulp testing: a plea for standardisation.

G. H. GEMMELL (Proc. Tech. Sect. Papermakers' Assoc., 1930, 10, 381–386).—The necessity for the standardisation of pulp-testing methods is urged. The fat and rosin content of various woods has been investigated, and it is concluded that the fatty-alcoholic extract is responsible for most of the "pitch" troubles of the papermaker. The addition of paraffin of d 0.8–0.85 to the pulp during bleaching is recommended.

T. T. PORTS.

Microscopical examination of pulps for rosin.

T. T. PORTS (Proc. Tech. Sect. Papermakers' Assoc., 1930, 10, 339–340).—A modification of the Sudan III test is described. The pulp is stained in a 0.2% solution of Sudan III in 70% alcohol for 1 hr., washed rapidly with 50% alcohol, cleared in 50% glycerin, and mounted in glycerin jelly. Rosin and fats are stained red, the fibres remaining colourless. Attention is drawn to the distribution of rosin in pulps.

T. T. PORTS.

Viscose. III. Determination of free and thiocarbonate-carbon disulphide in viscose. Their change during ripening of viscose. IV. Volumetric analysis of viscose containing sodium sulphite. Change of each component during the ripening of viscose. K. TANEMURA (J. Soc. Chem. Ind., Japan, 1930, 33, 50–51 B, 52–54 B; cf. B., 1930, 322).—III. With T. TAKAHASHI.] The free carbon disulphide content of viscose is determined by shaking an approx. 10% aqueous solution of viscose with ether for 15 min.,

treating the ethereal solution with ethyl-alcoholic potassium hydroxide, acidifying with acetic acid, neutralising with calcium carbonate, and titrating the aqueous layer with iodine. The total carbon disulphide, except cellulose xanthate, content is obtained by trituration of the viscose with solid sodium chloride, washing with excess of saturated sodium chloride solution, extraction of the filtrate with ether after previous acidification with acetic acid, and then proceeding as described above. The difference between these two values is due to sodium thiocarbonate. In the ripening of viscose containing sodium sulphite the free carbon disulphide decreases and the thiocarbonate-carbon disulphide increases.

IV. [With S. MIYOSHI.] A method is described for the volumetric determination of the components cellulose xanthate, sodium hydroxide, carbonate, sulphide, thiocarbonate, thiosulphate, and sulphite of viscose, and is applied to the determination of the variation of these components during the ripening process.

A. I. VOGEL.

Viscose. V. Sodium sulphate in viscose containing sodium sulphite. VI. Influence of sodium sulphite on viscose. K. TANEMURA (J. Soc. Chem. Ind., Japan, 1930, 33, 144–145 B, 146–147 B).—V. [With T. TAKAHASHI.] A method for the determination of sodium sulphate in viscose containing sodium sulphite is described; the content of sodium sulphate remains constant during ripening. In the case of cellulose xanthate, sodium sulphite corresponding to 0.14% of the viscose was oxidised to sulphate.

VI. [With S. MIYOSHI.] The changes undergone by viscose containing added sodium sulphite, with regard to content of sodium hydroxide, carbonate, sulphide, thiocarbonate, thiosulphate, sulphite, and cellulose xanthate alkali, during ripening have been studied, and compared with those occurring under the same conditions in viscose without added sodium sulphite. A part of the sodium sulphite is converted into sulphate, but the remainder does not change during ripening; the sulphite thus eliminates dissolved oxygen and retards the decomposition of cellulose xanthate. The formation of thiocarbonate from the carbon disulphide set free in the decomposition proceeds slowly. Both processes retard the coagulation of the viscose. The requisite quantity of commercial sodium sulphite ($\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$) is 3 kg. per 100 kg. of pulp.

C. W. SHOPPEE.

Viscose. XXIX. Properties of viscose prepared from alkali-cellulose immersed for a long time in alkali, and of the resultant spun fibres. T. NAKASHIMA, J. MURAKAMI, and S. OHORA (J. Soc. Chem. Ind., Japan, 1930, 33, 44 B).—Reduction in viscosity of viscose leads generally to weak fibres, but under certain conditions strong fibres can be spun from cellulose, the viscosity of which has been reduced by prolonged immersion in alkali.

A. I. VOGEL.

Viscose. XXXI. Effect of caustic alkali on cellulose (Part 2). XXXII. Effect of chlorination on cellulose. G. KITA (J. Soc. Chem. Ind., Japan, 1930, 33, 142–143 B, 143–144 B).—XXXI. [With T. NAKASHIMA, S. OHORA, and J. MURAKAMI.] The viscosity of commercial cellulose in cuprammonium solution is unexpectedly decreased by steeping in caustic alkali in an

atmosphere of hydrogen, and the copper number of the product obtained is distinctly diminished (cf. Nakashima, B., 1929, 917). To find whether cellulose by removal of oxidised products after milling would exhibit the original viscosity (cf. Waentig, B., 1927, 327), the products obtained from cotton paper by thorough milling in a hollander or by dissolution in concentrated zinc chloride solution and reprecipitation, and subsequent steeping for a long period, have been examined. In both cases the viscosity in cuprammonium solution is lower than that of the original cellulose, even if this has been treated by steeping. It is concluded that the decrease in viscosity produced by milling is due to other causes than oxidation.

XXXII. [With S. MAZUDA and T. SUZUKA.] The copper number of cellulose diminishes with repeated chlorination; the viscosity of the chlorinated product is slightly less than that of the original material, but is unaltered by repetition of the chlorination. The viscose threads spun from the chlorinated products possess the same properties as the original material, and are superior from many points of view. C. W. SHOPPEE.

Elimination of "dirt" from paper stock. R. H. CLAPPERTON (Proc. Tech. Sect. Papermakers' Assoc., 1930, 10, 341—354).—A summary of the methods employed for the removal of dirt from raw materials, half-stuffs, and pulps is given. Centrifugal separation of beaten pulps is advocated, and the operation of this method by means of the "Erkensator" is described at length. T. T. POTTS.

Rosin sizing [of paper]. S. R. H. EDGE (Proc. Tech. Sect. Papermakers' Assoc., 1930, 10, 289—294).—A continuation of previously published work (cf. B., 1928, 564). Further experimental evidence is given in support of the contention that aluminium sulphate (papermakers' "alum") is essential to sizing. Unbeaten pulps can be sized with rosin, "self-sizing" increasing with degree of beating. Acid aluminium resins have been prepared and found to have considerable sizing action when added to pulp; 2% added to a moderately beaten pulp of p_H 5.0 has, in the presence of a trace of free alum, the effect of hard sizing. T. T. POTTS.

Eliminating personal factors from the measurement of colour and gloss [of paper]. B. DESBLEDS (Proc. Tech. Sect. Papermakers' Assoc., 1930, 10, 235—282).—The Toussaint "photoelectric colorimeter" is described, and the application of the instrument to the matching of coloured papers is illustrated by means of "colour curves." Light is passed successively through 6 light-filters, covering the visible spectrum, on to a sample of the paper under test. The light reflected from the paper is received by a potassium photoelectric cell, and the intensity measured by means of a galvanometer. The galvanometer deflections plotted against the optimum wave-lengths of the light-filters furnish a "colour curve." Gloss is measured by comparing the galvanometer deflections obtained by means of reflected light from the test sample with that from a standard. T. T. POTTS.

Homogenisers. MÖLLERING. **Medium for flotation cells.** DYER and McCLELLAND.—See I. **Colour lakes.** CLAYTON. **Aircraft finishes.** GARDNER.—

See XIII. **Nitrocellulose.** DEMOUGIN, also BRESSER. —See XXII. **Water purification in the paper mill.** BERRIMAN.—See XXIII.

PATENTS.

Degreasing and opening wool and other fibrous materials. AKTIS PATENT-VERWERTUNGSGES.M.B.H., Assees. of A. UHLMANN (B.P. 304,295, 18.1.29. Ger., 18.1.28).—The material is immersed in water or dilute alkali or acid in a glass or porcelain vessel and exposed to the magnetic field produced by a high-frequency current of, e.g., 3×10^6 cycles per sec. [Stat. ref.]

D. J. NORMAN.

Treatment [oiling] of wool. C. A. BROWN and G. J. ESSELEN, JUN., Assrs. to MANUF. IMPROVEMENT CORP. (U.S.P. 1,749,410, 4.3.30. Appl., 23.7.27).—The oiling of wool is more effectively accomplished by the use of a chemically and mechanically stabilised emulsion of oil in water.

D. J. NORMAN.

Degumming of fibres. E. C. DUHAMEL (U.S.P. 1,748,832, 25.2.30. Appl., 15.3.29. Fr., 5.3.28).—Ramie and like fibres are immersed in a hot suint bath (d 1.056 or above) and then rinsed in warm or cold water.

H. ROYAL-DAWSON.

Retting of textile fibres. M. MARCUS, Assr. to (Mrs.) K. H. CHRISTENSEN (U.S.P. 1,746,316, 11.2.30. Appl., 28.5.28).—Retting is effected by nitrogen-fixing bacteria at 38°.

D. J. NORMAN.

Manufacture of waterproof fabrics, papers, and the like. S. KINUGASA and S. HASHIMOTO (B.P. 314,015, 6.6.29. Jap., 21.6.28).—After impregnation with a solution of soap, the material is dipped into the clear upper liquor obtained by mixing calcium carbonate with an aqueous solution of alum and acetic acid.

F. R. ENNOS.

[Manufacture of] vulcanised fibre. A. F. JACOBS (B.P. 330,394, 30.5.29).—Sheets of cotton cellulose in the form of absorbent paper are treated with a solution of zinc chloride and pressed together between heated rollers; after washing out the zinc chloride, the wet material is immersed in a bath of soluble oil ("hydraulic oil"—an emulsifiable product containing sulphonated oil) and water, and is subsequently dried and pressed between heated platens and rollers. F. R. ENNOS.

Pulp-treating process. G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,742,219, 7.1.30. Appl., 1.12.25).—High- α -cellulose pulp is obtained by digesting wood under conditions of high temperature and pressure with a sulphite liquor containing approx. 3—4% each of free and combined sulphur dioxide and agitating the resulting pulp for 0.5—2 hrs. at about ordinary temperature with alkaline solutions of sufficient concentration to remove a substantial proportion of the non- α -cellulose constituents of the pulp without causing mercerisation, e.g., 5—6% caustic soda solution. The action of the alkali is facilitated if the raw sulphite pulp is first treated with 3—4% of bleaching powder. The purified pulp is finally bleached at not above 32°.

D. J. NORMAN.

Production of high- α -cellulose fibre. G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,742,218, 7.1.30. Appl., 28.4.25).—Recovery of the heat and sodium compounds from the alkaline digestion liquor used in the

production of α -pulp from sulphite pulp is effected by recirculating the spent liquor and washings with addition of sufficient fresh alkali (to compensate for losses) over further quantities of pulp until the liquor has d 1.074—1.1154. The liquor is maintained at this concentration by diverting some of the liquor to a soda-recovery plant and proportionally increasing the flow of water through the countercurrent washer to maintain equilibrium.

D. J. NORMAN.

Manufacture of cellulose esters. G. W. MILES and C. DREYFUS, Assrs. to CELANESE CORP. OF AMERICA (U.S.P. 1,742,611, 7.1.30. Appl., 14.4.26).—Cellulose or its conversion products may be esterified by organic acids alone without the addition of acid anhydrides if the reaction is carried out in the presence of ortho- and/or meta-phosphoric acid. Catalysts, *e.g.*, sulphuric acid, may be used and the cellulose pretreated with glacial acetic acid or mixtures thereof with phosphoric acid. Fourteen examples are given.

D. J. NORMAN.

Cellulose esters or ethers and their uses. G. P. DAVIES, W. J. JENKINS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 328,934, 25.10.28).—Aqueous emulsions of cellulose ester or ether solutions, applicable as spraying enamels, are prepared by dispersing in water in a colloid mill, with the aid of a suitable emulsifying agent, a solution of a cellulose ester or ether in a solvent which (a) is immiscible or but slightly miscible with water and is not freely soluble in water, (b) gives a cellulose ester or ether solution of low interfacial tension against the water-phase, and (c) is sufficiently non-volatile to prevent precipitation of the ester or ether during the drying of the film even though the ratio of solvent to water is low and yet is sufficiently volatile to avoid unduly increasing the time of drying. Suitable solvents include methyl cyclohexanone, cyclohexyl acetate, and butyl propionate, and as emulsifying agents there may be used gelatin or casein for pigmented emulsions, or soap, preferably prepared from oleic acid in the non-aqueous phase and caustic soda in the aqueous phase, for unpigmented emulsions.

D. J. NORMAN.

Manufacture of cellulose butyric esters. G. B. ELLIS, From SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 328,259, 22.1.29).—Cellulose tributyrate is dissolved in aqueous butyric (or other aliphatic) acid or a mixture of such acids and hydrolysed for 16—18 hrs. at 45—50° until after precipitation by water the butyrate is soluble in methyl alcohol. Alcohol may be added to homogenise the aqueous acid solution.

C. HOLLINS.

Construction of spinning pots or boxes utilised in the manufacture of artificial silk. J. BRUHL (B.P. 312,041, 6.3.29. Fr., 19.5.28).—The walls of the pots or boxes are made by superimposing layers of fibrous material, such as cellulose or asbestos paper or fabric impregnated with synthetic resin, the resin also serving as an adhesive; the asbestos is arranged at the points at which there is liable to be attack by the material under treatment.

F. R. ENNOS.

Device for use in spinning of artificial silk. LUSTRAFIL, LTD., and G. DOUGILL (B.P. 330,130, 29.5.29).—The spinning funnel is provided with a number of asymmetric constrictions, the inner surfaces of which act as thread guides.

F. R. ENNOS.

Nozzles for spinning artificial silk. E. BRILL (B.P. 314,381, 18.6.29. Ger., 26.6.28).—The nozzle sleeve and base, the latter being provided with spraying apertures, are moulded in one piece from a basic porcelain paste to which is added a quantity of rare earths, *e.g.*, beryl or zircon, and are afterwards baked, whereby the entire surface, including the inner walls of the apertures, are coated with a smooth, hard glaze.

F. R. ENNOS.

Apparatus for the manufacture of artificial threads and the like. COURTAULDS, LTD., F. T. WOOD, and E. H. WELLS (B.P. 330,266, 4.3.29).—The spinning machine is so constructed that the current of air which is drawn in near the rotating guide-wheels is kept separate from that drawn in near the centrifugal boxes, until both currents are about to leave or have actually left the machine.

F. R. ENNOS.

Manufacture of artificial threads, bands, films, etc. I. G. FARBENIND. A.-G. (B.P. 307,758, 12.3.29. Ger., 12.3.28).—In order to avoid aeration of the spinning solution while it passes from the place of manufacture to that of coagulation, the use of gaseous pressure is avoided; the warm solution is transported by means of a mechanical pump or by gravity while subjected to a partial vacuum.

F. R. ENNOS.

Spinning of viscose. I. G. FARBENIND. A.-G. (B.P. 313,503, 12.6.29. Ger., 12.6.28).—The spinning bath contains a sulphonic acid of a lower homologue of the aliphatic series, *e.g.*, methionic acid, acetaldehyde-disulphonic acid, with or without a mineral acid, a salt, or an organic compound other than the sulphonic acid.

F. R. ENNOS.

Manufacture of artificial fibres. I. G. FARBENIND. A.-G. (B.P. 307,829, 1.3.29. Ger., 14.3.28).—Artificial fibres of denier even less than unity and of high tenacity are produced by spinning non-matured viscose, made from non-matured alkali-cellulose, into a bath containing one or more acid esters formed from a polybasic inorganic acid (sulphuric or phosphoric) and a mono- or poly-hydric aliphatic or aromatic alcohol.

F. R. ENNOS.

Precipitating baths for making threads or the like from viscose. I. G. FARBENIND. A.-G. (B.P. 303,514, 3.1.29. Ger., 5.1.28).—The improved results obtained, particularly in the spinning of unmatured viscose solution, by adding to the coagulating bath organic compounds of high mol. wt. having tanning properties (*e.g.*, sodium carbazolesulphonate) are most marked when the bath is approximately saturated with the organic compound. To effect this with a relatively small quantity of the compound the acid content of the bath is adjusted to 25—42% (calc. as sulphuric acid).

D. J. NORMAN.

Manufacture of artificial silk and like products. W. P. DREAPER (B.P. 328,627, 25.1.29).—Regenerated cellulose filaments or films containing vulcanised rubber are obtained by adding to the spinning solution rubber latex together with reagents which in contact with the coagulating solution will yield sufficient free sulphur to vulcanise the rubber present. Alternatively, the sulphur may be introduced by direct precipitation subsequent to the spinning operation. Vulcanisation is

effected at not above 90° by using an accelerator, such as diphenylguanidine, or a super-accelerator, optionally in the presence of anti-agers. Suitable proportions are 10% of rubber and 1% of free sulphur on the weight of cellulose.

D. J. NORMAN.

Manufacture of filaments, threads, films, etc. from cellulose derivatives. BRIT. CELANESE, LTD., W. A. DICKIE, and P. F. C. SOWTER (B.P. 328,911, 5.11.28).—Cellulose ester or other products may be washed free from soluble salts without becoming delustred if the washing is effected with aqueous solutions containing protective agents, *e.g.*, a 10% solution of sodium chloride or sodium acetate. (Cf. B.P. 246,879, 259,265—6; B., 1926, 317, 1008, 976.)

D. J. NORMAN.

Gas container for aircraft. K. HUERTTLE, Assr. to GOODYEAR-ZEPPELIN CORP. (U.S.P. 1,763,586, 10.6.30. Appl., 27.1.28).—See B.P. 304,775; B., 1930, 610.

Conversion of difficultly soluble or insoluble carbohydrate ethers into a soluble state. O. LEUCHS, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,767,382, 24.6.30. Appl., 24.6.26. Ger., 27.6.25).—See B.P. 277,111; B., 1927, 860.

Preparation of bodies from acylcelluloses. E. HUBERT, L. LOCK, and O. LEUCHS, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,822, 24.6.30. Appl., 17.4.25. Ger., 29.4.24).—See Swiss P. 115,676; B., 1927, 296.

Apparatus for spinning solutions of cellulose esters or ethers. M. KLEIN, Assr. to RUTH-ALDO CO., INC. (U.S.P. 1,767,216, 24.6.30. Appl., 27.3.28. Fr., 22.6.27).—See B.P. 292,561; B., 1929, 595.

Production of artificial threads by the cuprammonium stretch-spinning process. H. WINKELMANN, Assr. to AMER. BEMBERG CORP. (U.S.P. 1,761,860, 3.6.30. Appl., 19.1.28. Ger., 20.1.27).—See B.P. 283,923; B., 1929, 390.

Substitute for hard paper, ebonite, fibre, and the like, and its preparation. H. FRIEDLANDER and A. JANSER (U.S.P. 1,766,817, 24.6.30. Appl., 24.6.25. Austr., 26.6.24).—See B.P. 236,224; B., 1926, 986.

Manufacture of [non-curling] adhesive paper. J. MUIR, Assr. to G. H. WILKINSON (U.S.P. 1,766,302, 24.6.30. Appl., 15.6.28. U.K., 2.3.28).—See B.P. 308,444; B., 1929, 469.

Washing machine for raw materials for shoddy. H. WAFFENSCHMIDT (B.P. 331,158, 14.10.29).

Drying of sheets, plates, hanks, etc. SOC. EN NOM COLLECTIF ELECTRICITÉ GÉN. R. PONTIÈRE, and J. LE CLECH (B.P. 312,999, 4.6.29. Fr., 4.6.28).

Processes and devices for drying tubular bodies [composed of regenerated cellulose]. S. SOKAL. From KALLE & Co. A.-G. (B.P. 330,542, 8.3.29).

Scraper blade for detaching threads of cellulosic material from the winding-up drum of a spinning machine. M. DASSONVILLE (B.P. 330,753, 29.5.29. Fr., 30.10.28).

Blotting paper [having one face polished]. G. H. WILKINSON (B.P. 330,756, 31.5.29).

Machines for coating paper, fabric, etc. T. H. L. DIXON (B.P. 330,638, 15.3.29).

Rubberised fabric (B.P. 303,368).—See II. **Treatment of water-absorbent materials** (B.P. 326,940).—See VI. **Hypochlorites from waste lyes** (B.P. 305,931).—See VII. **Compound sheets of glass** (B.P. 330,265).—See VIII. **Plastics containing cellulose derivatives** (B.P. 312,604). **Moulded articles** (B.P. 329,273).—See XIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Wool dyeing, with special reference to the hosiery and tweed trades. J. G. GRUNDY (J. Soc. Dyers and Col., 1930, 46, 157—162).—The principal dye groups which can be applied are the acid, Cloth Fast, Neolan, Chrome Fast, and indigoid vat colours. For dyeing knitted fabrics in fast-to-light fawn, grey, and similar shades it is recommended to use Kiton Fast Yellow 2G, a colour of the Alizarine Sapphire Blue series, and a 1:1 mixture of Kiton Red G and Kiton Fast Red GL or BL, since these dyes fade at equal rates. Cloth Fast dyes are not so level-dyeing as the acid dyes, but yield shades much faster to washing and perspiration; they are much used in the tweed trade. Neolan dyes, which contain chemically combined chromium residues, are applied similarly to acid dyes, but have the fastness properties of Chrome Fast dyes. Since Neolan dyes require a large proportion (5—8%) of sulphuric acid in the dye bath the dyed wool must be thoroughly washed (preferably with dilute ammonia); difficulties in the subsequent processes of milling and back-washing have been traced to residual acid retained by the wool. The fastness properties of a very large number of dyes are tabulated. Wool treated for $\frac{1}{2}$ hr. at the boil with 80% of tannic acid and 6% of acetic acid (40%), and then fixed in a bath containing 5% of stannous chloride and 5% of chromium acetate (*d* 1.16) for $\frac{3}{4}$ hr. at 60° loses its affinity for Acid, Cloth Fast, Neolan, and Chrome Fast dyes; wool thus treated can be used in materials required in two colours. A list of dyes unaffected by this reserving process is given.

A. J. HALL.

Standardisation of tests for fastness of dyed fabrics. S. G. BARKER (J. Soc. Dyers and Col., 1930, 46, 162—164).—Results of investigations previously carried out (cf. Hedges, B., 1928, 260; King, B., 1928, 2639, 707; Cunliffe, B., 1929, 280) are summarised.

A. J. HALL.

Dyeing of furs. DOHOGNE.—See XV.

PATENTS.

Bleaching sensitive vegetable and animal fibres and materials, more particularly feathers, skins, pelts, and hairs (bristles) by treatment with oxidising bleaching agents. V., J., and R. BÖHM (BRÜDER BÖHM), Assees. of H. GOLDARBEITER (B.P. 310,030, 11.4.29. Austr., 21.4.28).—Bleaching is carried out by means of alkaline hydrogen peroxide in the presence of an oil mordant (a sulphonated oil such as Turkey-red oil) and a decomposition catalyst for hydrogen peroxide (salts of copper, nickel, cobalt, or silver).

F. R. ENNOS.

Dyeing cotton and artificial silk with indigosols. L. LIBMANN (F.P. 637,833, 18.7.27).—Fabric is mordanted with a dioxide (*e.g.*, of manganese or lead), washed, and dyed in a solution of an indigosol also containing sulphuric, hydrochloric, or acetic acid. Pattern effects are obtained by discharging the mordant with reducing agents before dyeing. A. J. HALL.

Manufacture of coloured fabrics. [Application of vat dye resists to yarn before weaving.] J. MORTON, E. G. HARRIS, J. I. M. JONES, and MORTON SUNDOR FABRICS, LTD. (B.P. 328,996, 6.11.28).—Sodium *m*-nitrobenzenesulphonate is applied in hot 25% aqueous solution, with or without size, to white or coloured yarn, which after weaving with untreated fibres is dyed or printed with vat dyes. C. HOLLINS.

Dyeing of piece goods and yarns with vat dyes. I. G. FARBENIND. A.-G. (B.P. 305,230, 1.2.29. Ger., 2.2.28).—Well penetrated dyeings are obtained by impregnating tightly beaten fabrics, or fabrics containing sized or tightly twisted yarns, with a liquor containing an unvat dye and an agent for promoting "bleeding," and then treating with an alkaline reducing liquor containing sodium hyposulphite. Suitable "bleeding" agents include an emulsion of an oil, or fat, or a free acid or water-soluble derivative thereof, or a cellulose derivative capable of forming in water a neutral or alkaline colloidal solution such as an alkali salt of carb-oxymethylcellulose (*cf.* B.P. 317,117; B., 1929, 894). A. J. HALL.

Dyeing of viscose. I. G. FARBENIND. A.-G. (B.P. 306,153, 15.2.29. Ger., 17.2.28).—Uniform shades on viscose silk are obtained by using couplings of tetra-azotised *m*- or *p*-azoxyanilines, which may also be produced by alkaline reduction of couplings of diazotised *m*- or *p*-nitroanilines. Examples are: *p*-nitroaniline-*o*-sulphonic acid \rightarrow β -naphthylamine-7-sulphonic acid, reduced to azoxy-compound (reddish-blue); *m*-nitroaniline \rightarrow β -naphthol-8-sulphonic acid, reduced to azoxy-compound (orange); *p*-nitroaniline-*o*-sulphonic acid \rightarrow phenol, methylated, reduced to azoxy-compound (golden-orange), etc. C. HOLLINS.

Dyeing of regenerated cellulose materials. IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 328,988 and 329,014, [A] 8.2.29, [B] 7.2.29).—Viscose silk is dyed in level shades (A) with monoazo couplings of an oxamic acid of the benzidine series with a naphthol- or naphthylamine-sulphonic acid, a 1:8-aminonaphthol- or dihydroxynaphthalene-sulphonic acid, or a *m*-diamine, or (B) with disazo dyes of the type: *p*-nitroaniline etc. (excluding nitroanisidines) \rightarrow a naphthol, a naphtholsulphonic acid, or an *N*-substituted 2:8-aminonaphtholsulphonic acid, reduced, \rightarrow 1:8- or 2:8-aminonaphthol-mono- or -di-sulphonic acid. Examples are: (A) benzidineoxamic acid \rightarrow N.W.-acid (bluish-scarlet), S-acid (brownish-violet), H-acid (violet), *m*-phenylenediamine (brown); dianisidineoxamic acid \rightarrow 2S-acid (bluish-violet), H-acid (bluish-violet), naphthionic acid (red-orange); (B) *p*-nitroaniline \rightarrow 8-hydroxy-2:2'-dinaphthylamine-3:6-disulphonic acid, re-reduced, \rightarrow H-acid (blue); *p*-nitroaniline \rightarrow N.W.-acid, reduced, \rightarrow 2S-acid (blue); 5-nitro-*o*-toluidine \rightarrow 2:7-naphtholsulphonic acid, reduced, \rightarrow H-acid (greenish-

blue); *p*-aminoacetanilide \rightarrow Schäffer acid, hydrolysed, \rightarrow S-acid (Prussian-blue). C. HOLLINS.

Production of coloured articles made from cellulose esters. I. G. FARBENIND. A.-G. (B.P. 308,780, 14.2.29. Ger., 29.3.28).—Threads and films manufactured from chlorinated organic acid esters of cellulose (*cf.* B.P. 306,132; B., 1930, 504), or mixtures of such derivatives with similar non-chlorinated esters (*e.g.*, cellulose acetate), are dyed with basic dyes; the chlorinated derivatives are more easily dyed than the non-chlorinated. A. J. HALL.

Coloration of fabrics [containing cellulose esters or ethers]. BRIT. CELANESE, LTD. (B.P. 306,534, 4.2.29. U.S., 23.2.28).—In printing fabrics or fibres containing cellulose derivatives with vat dyes which produce delustering of the silk, an addition of one or more of the following agents is made to the printing paste to prevent or inhibit the loss of lustre: methyl and ethyl ethers of ethylene glycol, diacetone alcohol, ethylene glycol with or without alcohol, benzyl alcohol, triacetin, cyclohexanone, potassium or ammonium thiocyanate, inorganic salts (*cf.* B.P. 246,879; B., 1926, 317), sugar (*cf.* B.P. 259,266; B., 1926, 977), phthalimide, and xylol monomethylsulphonamide. [Stat. ref.] A. J. HALL.

Printing of materials made from or containing cellulose esters or ethers. IMPERIAL CHEM. INDUSTRIES, LTD., A. SHEPHERDSON, and L. SMITH (B.P. 330,652, 19.12.28).—The printing strength of aminoanthraquinone (*cf.* B.P. 211,720; B., 1924, 375) is much improved by the addition of up to 5% of a hydroxyethylamine, particularly triethanolamine, to the printing paste. A. J. HALL.

Production of prints on cloth or like material. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 329,207, 22.6.29).—Cloth printed with vat or sulphide dyes to which has been added aqueous polyvinyl alcohol (with or without other thickening agent), but no alkali, may be subsequently slop-padded in an alkaline (reducing) bath and steamed without danger of the colour running. A mixture of 1 pt. of aqueous polyvinyl alcohol and 3 pts. of 10% starch paste is effective. C. HOLLINS.

Discharge effects on cellulose acetate silk. I. G. FARBENIND. A.-G., Assees. of A. FISCHESSE (G.P. 461,753, 18.6.26).—Dyed cellulose acetate silk fabric is printed with zinc salts of formaldehyde-sulphoxylic acid, then steamed, and treated in the usual manner. Non-dischargeable basic dyes may be added to the discharge paste for the production of multi-coloured effects. A. J. HALL.

Treatment of wool, fur, or like materials with organic liquids. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 330,598, 9.3.29).—The usual objectionable grey or white appearance produced by treating wool materials with an organic solvent and then evaporating this (as in moth-proofing) may be avoided by adding 1–2% of a second solvent or mixture of solvents having a high b.p., *e.g.*, butyl and benzyl alcohols, cyclohexanol, cyclohexanone, methyl phthalate. A. J. HALL.

Decolorising [cellulosic] film. E. B. MIDDLETON, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P.

1,743,155, 14.1.30. Appl., 31.12.25).—Film is treated with a chlorine bleaching liquor or gas and then with a liquor containing activated charcoal and an organic solvent (ethyl alcohol) capable of dissolving the products of decomposition of the dyes originally present without attacking the film. A. J. HALL.

Obtaining effects on textile materials of vegetable, animal, or other origin. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 328,978, 5.11.28).—Preparations containing "mono- or di-methylolurea" are applied locally to textiles, or are applied generally and removed from portions by solvent. Such preparations in printing pastes give clean prints, or without dyes they lead to lustre and damask effects. On mercerisation of the printed goods the methylolureas protect the textile, and crêpe effects are obtained. C. HOLLINS.

Delustring of artificial filaments, yarns, or threads. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 328,247, 8.1.29).—All types of artificial silk threads are delustred to any desired degree by subjection to a process of abrasion in which the threads are led over abrasive surfaces, or over loose abrasive material, or in which a stream of the abrasive material dispersed in a liquid medium is projected on the moving threads. Suitable abrasives include kieselguhr (to pass 120-mesh for cellulose acetate silk), pumice powder, powdered glass, carborundum, and emery. Other abrasives which are soluble in solvents and can thus be completely removed from the delustred threads are sodium sulphate, alum, and sodium chloride. A. J. HALL.

Weighting of silk. A. FEUBEL (U.S.P. 1,749,699, 4.3.30. Appl., 17.11.26. Ger., 28.11.24).—Natural or artificial silk is impregnated with basic aluminium nitrate solution obtained by dissolving freshly precipitated aluminium hydroxide in about half the equivalent quantity of nitric acid or by dissolving highly basic chloride or sulphate of aluminium in the least possible quantity of nitric acid. The aluminium is finally fixed on the fabric as its silicate or other insoluble compound. D. J. NORMAN.

Sizing of fibrous materials. E. PINEL (F.P. 637,654, 18.11.26).—The fibres are treated with a size containing carob-bean gum (tragasol), glycerin, and water, and then with another prepared with flour, dextrin, and wax. A. J. HALL.

Treatment of fibrous materials. I. TUDOR and O. TUDOR-HART (B.P. 328,638, 2.11.28).—A resilient character resembling that of horse hair may be imparted to vegetable fibres such as coir by first boiling the fibre for about 1 hr. with 3–5% caustic soda solution, rinsing, drying, preferably at 80° with continuous teasing, and finally polishing the fibre. D. J. NORMAN.

Treatment of fibres and fabrics. H. LIVSEY, G. E. HOLDEN, and J. & J. M. WORRALL, LTD. (B.P. 328,831, 30.5.29. Addn. to B.P. 313,980; B., 1929, 679).—In the process of the prior patent, animal glues and sizes are employed instead of the gelatin. H. ROYAL-DAWSON.

Treatment of water-absorbing materials to render them non-absorbent, acid-resisting, and alkali-resisting. H. N. MORRIS (B.P. 326,940, 2.2.29).—Paper, textiles, building materials, etc. are

impregnated with a common solution of rubber and one or more salts of cellulose prepared as described in B.P. 274,968 (B., 1927, 789), and are then dried. A. J. HALL.

Imparting bacteria- [mildew]-resistant properties to textile materials. C. B. WHITE and E. SCHAEFER, ASSTS. to VIVATEX PROCESSES, INC. (U.S.P. 1,745,134, 28.1.30. Appl., 23.10.26).—Sized cellulosic yarns and fabrics are protected from attack by mildew by the previous addition to the size of soluble compounds of rare-earth elements such as cerium, lanthanum, didymium, uranium, zirconium, and particularly thorium; 0.5–2.0% (calc. on the weight of the size) of thorium fluoride gives practically complete resistance to mildew attack. A. J. HALL.

Cleaning of wool material stained with tar- and pitch-tips. I. G. FARBENIND. A.-G. (B.P. 309,871, 16.4.29. Ger., 16.4.28).—The wool is treated in two successive baths, the first being a clear concentrated emulsion of heavy benzol (etc.) and ammonia, the second an emulsion containing less benzol or merely a solution of an alkali salt of the emulsifying agent, which in each case is a mixed fatty aromatic sulphonic acid, such as dibutyl-naphthalenesulphonic acid. C. HOLLINS.

Carrying out washing operations [on textiles] with hard water. G. ULLMANN and W. SECK (U.S.P. 1,746,170, 4.2.30. Appl., 9.11.28. Austr., 5.8.27).—In addition to ordinary soap, certain sulphonic compounds having colloidal properties and forming water-soluble calcium soaps are added to the water in amount less than that equivalent to the substances causing the hardness; suitable compounds are the sulphonated (polynuclear) aromatic or hydroaromatic hydrocarbons with an alkyl side-chain, or condensation products of these with aliphatic sulphonic acids or with a fatty oil. E. LEWKOWITSCH.

Dyeing and printing cellulose esters and ethers. F. FISCHER and C. E. MÜLLER, ASSTS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,765,142, 17.6.30. Appl., 20.4.27. Ger., 23.4.26).—See B.P. 269,934; B., 1928, 121.

Production of fast-coloured resists under aniline black. W. COTTON, ASST. to GEN. ANILINE WORKS, INC. (U.S.P. 1,765,658, 24.6.30. Appl., 28.1.26. Ger., 5.2.25).—See B.P. 247,211; B., 1927, 329.

Process in which cellulose acetate artificial silk and like products are treated with hot liquors. A. J. HALL, ASST. to CELANESE CORP. OF AMERICA (U.S.P. 1,765,581, 24.6.30. Appl., 22.7.25. U.K., 31.7.24).—See B.P. 246,879; B., 1926, 317.

Treatment [weighting] of artificial silks. R. CLAVEL (U.S.P. 1,761,707, 3.6.30. Appl., 27.5.27. Ger., 17.9.26).—See B.P. 277,602; B., 1928, 154.

Protection of wool, rugs, and the like against the attack of moths. E. HARTMANN, M. HARDTMANN, and P. KUMMEL, ASSTS. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,819, 24.6.30. Appl., 26.11.26. Ger., 27.11.25).—See B.P. 303,092; B., 1929, 241.

Method and apparatus for applying colours to fibrous textile fabrics. P. RAUER (B.P. 330,935, 18.3.29).

Machines for washing and similarly treating fabrics with liquids. H. WHITEHEAD (B.P. 330,915, 15.2.29).

Dyes from naphthazarin (B.P. 327,860). Application of disazo dyes (B.P. 329,056).—See IV. Waterproof fabrics (B.P. 314,015).—See V. Materials for marking animals (B.P. 329,408).—See XIII.

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

Automatically controlling acid effluents. H. K. RICHARDSON (Chem. Met. Eng., 1930, 37, 293—295).—To neutralise effluents containing free sulphuric acid with limestone it is necessary to keep the acid concentration less than 0.3%, otherwise a coating of calcium sulphate formed on the stone prevents further action. An electrolytic conductivity recorder records the strength of the effluent and rings a warning bell if the concentration exceeds 0.8%. A second conductivity recorder operates a valve through which water is added to dilute the acidity of the effluent to 0.3%. This recorder graphs the acid concentration of the diluted effluent. A third recorder records the p_H of the diluted effluent after neutralisation by limestone. It also opens a valve admitting a solution of soda ash, should the desired neutrality not have been effected, as sometimes happens owing to abnormal conditions, *e.g.*, when an excessively large volume of effluent has flowed through the limestone so rapidly that the reduced time of contact prevents the neutralisation from being completed.

D. K. MOORE.

Magnesium in commercial calcium citrate. F. PERCIABOSCO (Annali Chim. Appl., 1930, 20, 211—217).—All the samples of commercial calcium citrate examined contained appreciable proportions of magnesium, which occurs in the crude juice as the double salt, $MgCa_2(C_6H_5O_7)_2 \cdot 5H_2O$, and may amount to 0.9% (MgO) in the recovery liquid. To prevent this contamination of the recovered citrate with magnesium, treatment with either calcium chloride or hydrochloric acid is recommended.

T. H. POPE.

Nitrophosphates. G. CALCAGNI (Annali Chim. Appl., 1930, 20, 218—222).—The action of nitrogen dioxide on various phosphates moistened with water and kept shaken during the action yields fertilisers containing proportions of soluble phosphoric acid and nitrogen varying with the conditions employed. When treated in the cold (hot) with excess of the nitrogen dioxide, (1) tricalcium phosphate (41.89% P_2O_5), (2) apatite (40.22), (3) pebble phosphate (34.24), and (4) Kosseir phosphate gave fertilisers containing the following percentages of soluble P_2O_5 and N: (1) 41.69, 29.27 (31.82, 14.2); (2) 40.08, 33.37 (40.30, 27.04); (3) 33.96, 42.15 (33.87, 29.63); (4) 29.92, 46.84 (30.16, 32.65). These nitrophosphates, which are much less heavy than the corresponding amounts of superphosphate and nitrates with the same fertilising constituents, may be obtained with any required ratio between the proportions of soluble phosphoric acid and nitrogen, by using the calculated quantities of crude phosphate and nitrogen dioxide.

T. H. POPE.

Modernised silica grinding. R. L. CAWOOD (Chem. Met. Eng., 1930, 37, 304—305).—Sand obtained from sandstone by crushing, washing, and grinding is pulverised in a ball-mill lined with silex. This sand is unloaded from wagons and by means of an elevator and enclosed belt conveyor is discharged into a steel bin. From the bottom of this bin the sand is fed by a motor-operated feed device of variable capacity into a pit. A scoop on the mill lifts it from the pit and feeds it into the mill. A current of air carries the fines to an elevator, which lifts them to a centrifugal air separator, which removes the fines while the oversizes are returned to the mill. A screen fastened to the discharge end of the mill prevents balls and pebbles from passing. The process is continuous and automatic. By slight adjustment a product of varying fineness from 97% through 100-mesh to 97% through 300-mesh can be obtained. The power requirements of the various items of plant are given.

D. K. MOORE.

Solubility of sulphur. D. HENVILLE (Analyst, 1930, 55, 385).—Precipitated sulphur dissolves readily in carbon disulphide and slowly but completely in methylated spirit and petroleum spirit, whereas sublimed sulphur (the sample used) was only soluble to about 83% in each of these solvents. The sulphur samples extracted contained only traces of mineral matter. Sulphur ointment containing 10.2% S yielded 3.75% of residual sulphur after extracting 2 g. for 2 hrs. with petroleum spirit in a continuous extractor.

D. G. HEWER.

Autoclaves. HARRISON.—See I. Triple fertiliser. PESTOV and KALABEKOVA. Apatite and nepheline rock as fertiliser. SOMOV. Ground sulphur for dusting etc. STREETER and RANKIN.—See XVI.

PATENTS.

Manufacture [synthesis] of hydrogen chloride or hydrogen bromide or their corresponding acid solutions. RÖHM & HAAS A.-G. (B.P. 312,908, 7.5.29. Ger., 2.6.28).—Hydrogen and the halogen are passed with a velocity sufficiently high to prevent reaction of the gases or products with the metal through a burner constructed (preferably) of copper, or of iron, lead, or an alloy, and thence into a combustion chamber constructed (preferably) of lead and provided with means for cooling the combustion gases to about 80°.

L. A. COLES.

Dehydration of materials [caustic alkali]. J. F. WAIT, Assr. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,749,455, 4.3.30. Appl., 31.12.24).—Caustic alkali which has had its moisture reduced to 10% by steam and direct fire heat, is, while molten, subjected to an electric current between electrodes. Either alternating or direct current may be used, *i.e.*, the final moisture may be removed by evaporation or electrolysis.

B. M. VENABLES.

Catalytic oxidation of [impure] ammonia. SELDEN Co., Assees. of A. O. JAEGER (B.P. 309,583, 26.3.29. U.S., 14.4.28).—Impure ammonia produced, *e.g.*, in the distillation of coal tar is oxidised in one or more stages with the use as catalyst, during at least one stage, of diluted multi-component zeolites produced

as described in B.P. 279,466 (B., 1928, 603); *e.g.*, the air-ammonia mixture is passed at 400–450° over a zeolite catalyst to burn away phenolic and other impurities and then, after washing with alkali solutions etc. if necessary, over platinum gauze or an iron-aluminium multi-component zeolite at 650–800° to effect oxidation of the ammonia. L. A. COLES.

Manufacture of alkali hypochlorites from [artificial-silk] waste alkali lyes. I. G. FARBENIND. A.-G. (B.P. 305,931, 9.2.29. Ger., 11.2.28).—Chlorine is passed at about 50° into waste lye containing organic impurities, whereby organic matter is oxidised to carbon dioxide, and alkali hypochlorite solution remains.

H. ROYAL-DAWSON.

Manufacture of alkaline-earth and alkali formates and chromium-green. I. G. FARBENIND. A.-G. (B.P. 305,588, 6.2.29. Ger., 7.2.28).—A suspension or solution of an alkaline-earth or alkali chromate is reduced at 150–350° under increased pressure by carbon monoxide.

H. ROYAL-DAWSON.

Manufacture of acid calcium phosphate [for use in baking powders, self-raising flour, etc.]. R. HADDAN. From FEDERAL PHOSPHORUS CO. (B.P. 330,777, 20.6.29).—Milk of lime, *d* about 1.2, is added slowly with stirring to 75% phosphoric acid until the suspension has *d* 1.4 and contains 1–3% excess of lime, and the suspension is dried by spraying in warm air.

L. A. COLES.

Production of barium sulphide. KALI-CHEMIE A.-G., Assees. of RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G. (B.P. 316,966, 26.6.29. Ger., 7.8.28).—The reduction of heavy spar, especially that with poor agglomerating properties, is facilitated by precalcination at, *e.g.*, 600–700°, or by disintegration to particles not exceeding 1 mm. in diameter, or by the addition of small quantities of mineral acids or alkali or alkaline-earth salts, or by a combination of two or more of these processes.

L. A. COLES.

Reduction of metallic compounds and production of arsenates. G. N. KIRSEBOM (B.P. 315,811, 18.7.29. U.S., 18.7.28).—Material containing reducible metal compounds, such as flue dust, antimony ore, cadmium dust, slime, caustic lead slag, etc., is heated with sodium hydroxide and arsenic trioxide; the metals or alloys collecting at the bottom of the melt are run off and the slag is treated with water to extract sodium arsenate and excess sodium hydroxide. The sodium arsenate is recovered by crystallisation and treated with milk of lime to precipitate calcium arsenate and to yield sodium hydroxide solution which, after filtration, is returned, together with the mother-liquor from the crystallisation, to the process. L. A. COLES.

Reduction of metal oxides, anhydrides, or metalloid oxides [to lower oxidation stages]. J. CHABANNAIS (F.P. 637,403, 11.7.27).—The pulverised oxide is treated at a suitable temperature in a container or autoclave under continuous agitation with a liquid or viscous reducing agent (*e.g.*, hydrocarbons or anhydrides of not too low b.p.). The oxide produced has an improved reactivity towards acidic or basic reagents.

S. K. TWEEDY.

Treatment of siliceous minerals. A. J. MOXHAM, Assr. to ELECTRO CO. (U.S.P. 1,748,989, 4.3.30. Appl., 25.11.24).—Siliceous material containing potassium, aluminium, and ferric iron, such as greensand, is digested with not too concentrated sulphuric acid, *e.g.*, in 100% excess. Part of the potassium and aluminium sulphates formed is allowed to crystallise out as potassium alum; the remainder of these sulphates, together with the ferric sulphate, is precipitated out in concentrated sulphuric acid. This precipitate may be utilised in iron or steel manufacture or as a pigment. The mother-liquor is used in subsequent digestions. S. K. TWEEDY.

Production of oxides of titanium. J. BLUMENFELD (B.P. 307,881, 11.3.29. Ger., 15.3.28).—A titanium halide (*e.g.*, tetrachloride) distributed upon a soluble salt, *e.g.*, an alkali chloride or sulphate, is treated with steam and the mass is calcined, cooled, and extracted with water to remove the salt; the product is obtained in a finely-divided form suitable for use as a pigment.

L. A. COLES.

Preparation of oxygen-evolving preparations suitable for use in respiratory apparatus and the like. DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 305,101, 30.1.29. Ger., 30.1.28. Addn. to B.P. 280,554; B., 1928, 447).—An alkali peroxide, together with catalysts and inert substances is hydrated in cooled rotating drums with 9–23% of water introduced in an atomised form, or as vapour or snow. The hydrated material is formed into shaped pieces and is heated, before or after shaping, to increase its stability.

W. G. CAREY.

Contact sulphuric acid process. SELDEN CO., Assees. of A. O. JAEGER (B.P. 314,858, 27.5.29. U.S., 3.7.28).—See U.S.P. 1,741,310; B., 1930, 660.

Production of hydrocyanic acid. P. T. DOLLEY, Assr. to CALIFORNIA CYANIDE CO., INC. (U.S.P. 1,761,433, 3.6.30. Appl., 19.10.27).—See B.P. 299,019; B., 1929, 850.

Preparation of vanadic acid. A. O. JAEGER, Assr. to SELDEN RES. & ENG. CORP., and J. A. BERTSCH (U.S.P. 1,765,870, 24.6.30. Appl., 15.5.26).—See B.P. 287,401; B., 1928, 366.

Manufacture of ammonia. C. URFER, Assr. to SOC. D'ETUDES MINIÈRES ET INDUSTRIELLES (U.S.P. 1,767,780, 24.6.30. Appl., 2.6.27. Fr., 8.1.25).—See B.P. 253,540; B., 1927, 218.

[Production of] stable ammonium bicarbonate. E. DEHNEL, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,705, 24.6.30. Appl., 8.6.25. Ger., 10.6.24).—See B.P. 244,645; B., 1926, 155.

Electrolytic manufacture of [per-]compounds containing active oxygen. O. NITZSCHKE and E. NOACK, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,722, 24.6.30. Appl., 4.6.27. Fr., 26.4.26).—See B.P. 290,750; B., 1928, 523.

Production of calcium nitrate, alumina, and phosphorus. R. GRIESSBACH, O. SCHLIEPHAKE, and O. HEUSLER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,766,785, 24.6.30. Appl., 17.1.28. Ger., 24.1.27).—See B.P. 296,832; B., 1928, 815.

Manufacture of calcium molybdate. A. KISSOCK (U.S.P. 1,763,712, 17.6.30. Appl., 6.11.26).—See B.P. 280,240; B., 1929, 393.

Dissociation of sulphur vapour. P. C. H. SIEDLER and E. SCHULTE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,767,319, 24.6.30. Appl., 1.10.28. Ger., 12.10.27).—See B.P. 298,599; B., 1929, 128.

Manufacture of sulphuryl chloride. R. H. McKEE and C. M. SALLS (U.S.P. 1,765,688, 24.6.30. Appl., 26.6.23).—See Can.P. 251,586; B., 1926, 406.

Carbon dioxide ice apparatus, process, and product. A. P. THURSTON. From DRYICE EQUIPMENT CORP. (B.P. 331,077, 13.6.29).

Ammonia and hydrogen sulphide from gases (B.P. 316,278).—See II. **Electric furnace** (B.P. 303,798).—See XI. **Satin-white** (B.P. 327,848).—See XIII. **Fertiliser** (B.P. 309,175).—See XVI.

VIII.—GLASS; CERAMICS.

Glass-house refractories. Study of corrosion-resisting properties. J. H. PARTRIDGE and H. C. BIGGS (J. Soc. Glass Tech., 1930, 14, 63—75 T).—The results of an examination of seven fireclay blocks, two samples of sillimanite blocks, and one of cast mullite by Bowmaker's method (B., 1929, 815) were correlated with those observed in practice. It is shown that under standard conditions the test gives results of value in conjunction with accurate porosity determinations in estimating probable behaviour in service. High alumina content (other things being equal), porosity of less than 20%, fine close uniform texture, and high firing temperature gave good resistance. The test extended to pot-clay mixtures indicated corrosion-resistances less than those of good tank blocks. Small crucibles, made from various mixtures of clay with ball clay and grog and with addition of alumina, were used for founding at 1270—1300° successive charges of a glass having the composition: SiO_2 57.8, PbO 26.55, Na_2O 5.3, K_2O 6.92%; the corrosion was determined by visual inspection. It is concluded that addition of alumina to a pot-clay mixture improves the corrosion-resistance, provided that the alumina combines with some of the free silica to form mullite. This did not always occur at temperatures as low as 1350°. M. PARKIN.

Chemical reactions in the melting of potassium borosilicate glass. M. A. BESBORODOV and L. M. SILBERFARB (J. Soc. Glass Tech., 1930, 14, 39—51 T).—Using quartz ground to pass 3000-mesh/cm.², potassium carbonate, and boric acid, all chemically pure, a glass having SiO_2 63, B_2O_3 25, K_2O 12% was investigated. The batch was ground in a ball-mill and heated in lots of 20—25 g. to temperatures ranging in 100° steps from 500° to 1200°. Reaction, as detected by loss in weight, water-soluble and residual silica, was considered to set in below 500°. The amount insoluble in water increased rapidly between 700° and 900°, and at lower temperatures potash appeared to form soluble borates or silicates, no insoluble potassium borosilicates being found below 700°. At 500° some insoluble borosilicates were formed, the amount being small below 700°, and became significant only when potash also entered into

the composition of the "insoluble," which was found to range from SiO_2 99, B_2O_3 1.15%, K_2O nil at 500° to SiO_2 68.58, B_2O_3 21.07, K_2O 10.25% at 1200°. At 900° the K_2O rose to a maximum of 15.3% with SiO_2 73.3 and B_2O_3 11.4%. M. PARKIN.

Choice of annealing schedule for commercial glassware. J. B. MURGATROYD (J. Soc. Glass Tech., 1930, 14, 3—12 T).—The final strain in the article was determined by the temperature gradient across the strain-free glass when it "set," and the greatest value, θ , of the temperature difference between inner and outer walls was given by $\theta = Bx/50NaY$, where x is the percentage of the breaking stress which could safely be permitted in the annealed article at room temperature, B the breaking stress in kg./mm.², Y the Young's modulus, a the coefficient of expansion for the glass at room temperature, and N the ratio of expansion coefficient above the critical temperature to a . The rate of loss of temperature allowable to yield ware possessing $x\%$ of breaking stress at room temperature is $BKx/50NaYdSL^2$ degrees per sec., where d is the density in the annealing range, S the sp. heat, K the coefficient of thermal conductivity, and L the thickness of the glass wall in cm. Taking average values for a soda-lime-silica glass, the author arrived at a cooling rate of $1.3^\circ/\text{L}^2$ per min. to leave 5% of the breaking stress. For ware 1 cm. in thickness the annealing schedule was then 15 min. for ware to become uniform and strain-free at the annealing temperature, 11 min. to cool at the rate of $5^\circ/\text{min.}$ through the annealing range (given as 56°), and 35 min. cooling at $10\text{--}15^\circ/\text{min.}$, i.e., 61 min. in all. Thicker ware would take longer. Ware with up to 20% of the breaking stress present did not show any lessening of resistance to mechanical shock, whilst it withstood thermal shock better. If this figure were substituted for x in the expressions given, the rate of annealing would become $5.2^\circ/\text{L}^2$ per min. At 500° the loss of heat by radiation, ignoring the departure from black-body conditions, from glassware in a lehr was 0.013 g.-cal./sec., whilst that by conduction from one face to the other was 0.01 g.-cal./sec.

M. PARKIN.

Nature of minerals in artificial melts of $2\text{CaO}, \text{SiO}_2$, of $3\text{CaO}, \text{SiO}_2$, and of $8\text{CaO}, 2\text{SiO}_2, \text{Al}_2\text{O}_3$. K. KOYANAGI (J. Soc. Chem. Ind., Japan, 1930, 33, 161 B).—The solids deposited from melts of the above composition have been examined microscopically. In all three mixtures strongly birefringent, crystalline needles were obtained, most abundantly from the melt $2\text{CaO}, \text{SiO}_2$, which probably have this composition. Crystals corresponding with alite from Portland cement clinker were obtained only from the melt $3\text{CaO}, \text{SiO}_2$. Crystals of lime have also been observed in some of the mixtures. O. J. WALKER.

Velocity of crystallisation of soda-lime-silica glasses. K. TABATA (J. Soc. Chem. Ind., Japan, 1930, 33, 95—96 B).—The work of Zschimmer and Dietzel (B., 1926, 877; 1929, 472) is criticised. The crystallisation of silica may be observed during surface devitrification accompanied by surface contraction, and the importance of the latter phenomenon is emphasised.

H. F. GILLBE.

Beryllium glass. II. Potassium-beryllium series. C. F. LAI and A. SILVERMAN (J. Amer. Ceram. Soc., 1930, 13, 393—398; cf. B., 1928, 894).—A preliminary investigation of the properties of beryllium glasses is described, and results are given in the form of triaxial diagrams for K_2O - BeO - SiO_2 and Na_2O - BeO - SiO_2 . The region of high beryllia and low silica indicates better glasses than that of low beryllia and high silica. Density determinations result in lower values for potash than for soda glasses. Refractive index, composition, and properties of these glasses are also tabulated.

R. J. CARTLIDGE.

Determination of boric oxide in glass. V. DIMBLEBY (J. Soc. Glass Tech., 1930, 14, 51—60 T).—In tests on the methods of Wherry and Chapin (B., 1908, 1179) and of Sullivan and Taylor (B., 1914, 1154), the author found that good-quality resistance-glass flasks were superior, in general, to those of other types, even though containing boric oxide. After ageing, the amount of attack was practically constant. The amount of calcium carbonate in excess and the time of boiling caused variations in blank titration of the order of 0.65 c.c. of the sulphuric acid used. Serious loss of boric oxide could occur if the precipitate obtained with calcium carbonate were not dissolved in dilute hydrochloric acid and reprecipitated by the reagent, and use of more than 0.3 c.c. of phenolphthalein as indicator caused low values. Careful control of these points enabled good agreement to be obtained by independent analysts. Detailed analytical procedures embodying these findings are given (a) for simple soda-lime-boric oxide-silica glasses and (b) for those rich in boric oxide or containing lead or zinc.

M. PARKIN.

Thermal expansion of glass. I. General form of the expansion curve. II. Glasses of the series sodium metasilicate-silica. W. E. S. TURNER and F. WINKS (J. Soc. Glass Tech., 1930, 14, 84—109 T, 110—126 T).—I. Contrary to expansion curves described by other workers up to 1928, this curve was not substantially linear up to the critical or transformation temperature, C_t , but, as shown also by Klemm and Berger, possessed inflexion points between 0° and C_t . These in both silicate and borosilicate glasses occurred at temperatures closely alike, in spite of considerable differences in composition, viz., at 120 — 150° , at 250° , and a third, where it existed, at 350° . Certain glasses did not show these points, and two were encountered that gave practically linear curves right up to the upper annealing temperature A_t . Annealing influenced the form of the curve between A_t and C_t , the deviation from the straight portion occurring at a lower temperature on strained specimens. The increase in expansion in the range C_t - A_t was most marked in the case of glasses rich in boric and iron oxides.

II. Fifteen glasses of the system sodium metasilicate-silica, ranging from 51.15 to 91.6% SiO_2 , were prepared by melting in a platinum crucible in an electric furnace, precautions being taken to obtain homogeneous glass from which rods were drawn to give test pieces 10 cm. \times 0.5 cm. The glass with 51.15% SiO_2 gave an expansion curve in which the normal critical range was entirely absent, and was not induced

by re-annealing. Glasses with 51.15—85.04% SiO_2 behaved normally, whilst those richest in silica gave indications of the C_t point, but no A_t point, as shown by the bending over of the curve even up to 650° , the optically determined annealing point being much below this. The annealing temperatures rose from 440° for the glass having 51.15% SiO_2 to 530° for that with 85.04% SiO_2 , whilst the temperatures of the change points t_1 , t_2 , and t_3 (cf. preceding abstract) were very constant at 120 — 140° , 240 — 250° , and 350° , respectively. The C_t value rose with silica content up to 455° for 72.8% SiO_2 and then remained practically constant; A_t rose similarly, but irregularly, till 85% SiO_2 was reached, after which the rise was more rapid. The new measurements over the range 0 — 130° substantially confirmed those of English and Turner, and differed notably from those of Samsen. The relationship between chemical composition and thermal expansion was strictly linear for all ranges of temperature below C_t for glasses with 60—85% SiO_2 and probably linear with a different slope for those with 85—100% SiO_2 .

M. PARKIN.

Sp. heat of pyrex glass from 25° to 175° . T. DE VRIES (Ind. Eng. Chem., 1930, 22, 617—618).—A thermocouple and a nichrome heating element were fused into a piece of pyrex glass silvered over and suspended in a highly evacuated container surrounded by an oil-bath. The energy input and rise of temperature for 3-min. periods of heating and for various initial temperatures were determined. The values found were in close agreement with the equation: sp. heat = $0.1839 + 0.0003134(t - 20^\circ)$.

C. IRWIN.

[Strength of glass containing cracks.] F. W. PRESTON (J. Soc. Glass Tech., 1930, 14, 37—38 T).—In a communicated discussion of Milligan's observations (B., 1930, 557) the present author suggests that Milligan's polariscope figures reveal "bearing stresses," and that hydrofluoric acid would probably notably increase the strength.

M. PARKIN.

Development of acid-resisting, white, sheet-steel enamels. A. I. ANDREWS (J. Amer. Ceram. Soc., 1930, 13, 411—426).—The acid-resistance of enamels has been studied with a view to control such variables as composition, smelting, milling, and firing. Simple acid-resisting enamels were made from the series Na_2O - B_2O_3 - SiO_2 and Na_2O - PbO - SiO_2 . The results of acid-resistance as shown by the spot test check well with those of the grain test. The data given show that silica and titania both improve the acid-resistance, whilst fluxes, unless properly proportioned, tend to decrease this property. Fineness of grinding and smelting conditions make no difference to acid-resistance, but increase of firing temperature increases the acid-resistance of most enamels. Addition of titania to an enamel does not increase the refractoriness as much as does silica.

R. J. CARTLIDGE.

Clays from Shdanka (Sysran-Viasma Rly.). V. PERMYAKOV (Trans. Ceram. Res. Inst., Russia, 1929, No. 17, 4—31).—The composition and ceramic properties of the grey or brown coarse-grained clays are recorded.

CHEMICAL ABSTRACTS.

Examination of china clay [for grit]. R. G. THIN (Proc. Tech. Sect. Papermakers' Assoc., 1930, 10,

388—393).—An elutriation method for the determination of grit in china clay is preferred to sedimentation methods. A modification of Schöne's apparatus is described. Higher percentages of grit are obtained by this method than by Strachan's method (unpublished), but it is pointed out that appreciable variation in results obtain if standard experimental conditions are not adhered to. A brief note on the moisture content of clays is given. T. T. POTTS.

[Examination of china clay for grit.] J. STRACHAN (Proc. Tech. Sect. Papermakers' Assoc., 1930, 10, 395—397; cf. preceding abstract).—Thin's method is criticised, and it is held that a sedimentation method alone can correctly determine grit in a clay, elutriation serving only for grading the true clay according to particle size. The Schöne method is held to take too small a sample, to require too long a time, to be subject to disturbing convection currents in the elutriating tube, and to be difficult to wash out. The volume, temperature, and p_H of the water require careful control. By a sedimentation method it is possible to remove quantitatively all grit over 0.05 mm. in 1—1½ hrs. Mineralogical analysis of the grit should supplement the quantitative determination. T. T. POTTS.

Technical analysis of clays with the aid of the microscope. H. MÖHL (Sprechsaal, 1929, 62, 731—734; Chem. Zentr., 1930, i, 885).—The methods employed are described, together with the microscopical characteristics of various clays. A. A. ELDRIDGE.

Solubility of feldspars in water. C. W. PARMELEE and A. J. MONACK (J. Amer. Ceram. Soc., 1930, 13, 386—392).—Data are presented showing the relation between the rate of solubility and the time, using different feldspars. The alkalinity or solubility of the feldspar was measured by colorimetric determinations of the p_H values. Equations are given merely as indications of the true relations. The results obtained show that no definite relation exists between the ratio K_2O/Na_2O and the solubility of feldspar in water. High-soda and high-potash feldspars are stated to have equal solubilities, whilst intermediate feldspars possibly have the highest solubilities. The data also indicate that there may be sufficient increase in alkalinity to affect the viscosity of casting slips. R. J. CARTLIDGE.

PATENTS.

Manufacture of coloured glassware. T. DAVIDSON (B.P. 329,022, 11.2.29).—Variegated, cloud, or streak effects are obtained in glass by the appropriate inter-fusion of two or more glasses of different shades or colours; the finished articles are then given a backing or surface coating of coloured enamel or paint.

H. ROYAL-DAWSON.

Manufacture of compound sheets of glass and cellulose derivative composition. ACETEX SAFETY GLASS, LTD., and W. JOHNSTON (B.P. 330,265, 4.3.29).—Two sheets of glass with an interposed sheet of, e.g., cellulose acetate, are cemented together by treating the sheet of cellulose acetate with a solvent (triacetin) of high b.p., pressing the sheets together by means of compressed air, and finally subjecting them to steam

under pressure and at a temperature below the b.p. of the solvent. C. A. KING.

Enamelling of glass articles. M. JEANDIDIER (F.P. 633,277, 25.4.27).—Directly after or during moulding the articles are dusted over with an enamel which has a coefficient of expansion differing from that of the glass constituting the articles. The enamel fuses and on cooling adheres to the glass, to give a crazed effect. S. K. TWEEDY.

Drying of ceramic products. J. KOTTERBA (G.P. 461,077, 1.8.25).—The articles are placed in chambers with perforated bottoms through which the hot drying air is introduced under pressure and the wet air is withdrawn. The hot air rising in the chambers is thus moistened by direct contact with the gravitating moist air leaving the same, so that the articles are not brought into contact with hot dry air, whereby drying of merely the outer layer, with consequent non-removal of the interior moisture, is prevented. The dried articles are free from cracks. S. K. TWEEDY.

Drying of refractory materials. W. J. GARDNER (B.P. 330,447, 29.7.29).—A longitudinal drying chamber, through which articles of refractory material are conveyed on trucks, is divided into several zones of increasing temperature by means of baffle walls. Fans are provided in each zone to cause a turbulent motion of the atmosphere and ports are provided for renewing the air supply. C. A. KING.

Terra-cotta firing system. A. H. SHEFFIELD (U.S.P. 1,749,988, 11.3.30. Appl., 15.4.27).—The interior of the kiln is provided with an inner wall which extends upwards for about a third of the height of the kiln and is spaced from the wall of the latter. The flames from gas jets projecting through apertures in the outer wall play upon the inner wall and pass up the space between the walls. The inner wall may also be mounted upon a separate base which is spaced from the true bottom of the furnace. The gas flames then pass under the base as well and up a flue provided in the middle of the latter. S. K. TWEEDY.

Tunnel kiln. P. A. MEEHAN, ASST. to AMER. DRESSLER TUNNEL KILNS, INC. (U.S.P. 1,764,460, 17.6.30. Appl., 3.4.28).—See B.P. 312,786; B., 1929, 645.

Forming abrasive [surfaces on] articles [by spraying]. E. GARNER, and CARBORUNDUM CO., LTD. (B.P. 330,322, 16.3.29).

[Segmental] grinding or abrading wheels. NORTON CO. (B.P. 308,991, 2.4.29. U.S., 2.4.28).

Light filter (U.S.P. 1,751,220).—See I. Nozzles for artificial silk (B.P. 314,381).—See V. Electric furnace for glass (B.P. 303,798). Tunnel kiln (U.S.P. 1,749,762). Insulating material (B.P. 330,268).—See XI.

IX.—BUILDING MATERIALS.

Mixed Portland cements. VI. S. NAGAI (J. Soc. Chem., Ind., Japan, 1930, 33, 82—84 B; cf. B., 1930, 284).—The strength of Portland cement mixtures containing various proportions of ground clinker and spent shale is found to be considerably greater than that of ordinary Portland cement. H. F. GILLBE.

Draught, temperature, and combustion conditions in commercial kilns burning building brick. H. P. KIMBELL (Univ. Toronto Fac. Appl. Sci. Eng., Eng. Res., Bull. No. 8, 1928, 1—34).—Temperatures, draughts, and gas analyses of the burning of four different charges of bricks in four kilns of two different types are given, and calculations are made both of the efficiency of burning and also of the principal loss, viz., the heat in the products of combustion. Of the useful heat, the sensible heat in the hot bricks is by far the largest item, being usually more than ten times that required to drive off moisture. The efficiency of burning appears to be 20—25%, though since the sp. heat of the material is not known with any accuracy this figure is in doubt. There are too many variables for any conclusions to be drawn. B. M. VENABLES.

High iron oxide Portland cement. II. S. NAGAI and K. AKIYAMA (J. Soc. Chem. Ind., Japan, 1930, 33, 85—87 B; cf. B., 1930, 462).—Further data are given relative to the chemical composition, density, and compressive strengths of Portland cements rich in iron.

H. F. GILLBE.

High iron oxide Portland cements. III. S. NAGAI and K. AKIYAMA (J. Soc. Chem. Ind., Japan, 1930, 33, 137—140 B; cf. preceding abstract).—The compressive strengths and other physical properties of some 1:3 high iron oxide Portland cements (made from "Colloidal Jodo," a Japanese natural product containing soluble silica and alumina) were compared with the properties of similar cements prepared from pure chemicals.

S. K. TWEEDY.

Calcium ferrites and iron cements. I. S. NAGAI and K. ASAOKA (J. Soc. Chem. Ind., Japan, 1930, 33, 130—133 B).—The ferrite of highest lime percentage in clinker of high lime content is $2\text{CaO} \cdot \text{Fe}_2\text{O}_3$ (cf. Sosman and Mervin, B., 1916, 1108). This ferrite is readily obtained by heating a mixture of 5 pts. of quicklime and 1 pt. of ferric oxide above 1200° ; it is soluble in 0.5*N*-hydrochloric acid, whereas monocalcium ferrite is insoluble.

S. K. TWEEDY.

Calcium ferrites and iron cements. II. S. NAGAI and K. ASAOKA (J. Soc. Chem. Ind., Japan, 1930, 33, 161—164 B; cf. preceding abstract).—Dicalcium ferrite, $2\text{CaO} \cdot \text{Fe}_2\text{O}_3$, has been obtained by extracting the free lime from heated mixtures of lime and ferric oxide by means of a glycerin-alcoholic solution of ammonium acetate. By heating an equimolecular mixture of the two oxides between 1000° and 1200° , monocalcium ferrite, $\text{CaO} \cdot \text{Fe}_2\text{O}_3$, was obtained. The two ferrites can be separated from lime-ferric oxide mixtures by treating firstly with 0.4*N*-hydrochloric acid and then with 0.8—2.0*N*-acid, which dissolves the di- and mono-ferrite, respectively.

O. J. WALKER.

Hydration of Portland cement. Influence of monocalcium phosphate [? gypsum]. I—V. K. KOYANAGI (J. Soc. Chem. Ind., Japan, 1930, 33, 147—153 B).—The hydration of efficiently burnt Portland cement always commences with the formation of needles and plates of calcium aluminate, which brings about the binding of the cement; in the course of time the needles are completely converted into plates. When gypsum is

added to the cement calcium sulphoaluminate, $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 2.5\text{CaSO}_4 \cdot 30\text{H}_2\text{O}$ is formed instead; this compound prolongs the binding time. The hardening process depends on the formation of a gel mass of calcium hydrosilicate which occurs much later. Finally, large hexagonal crystals of hydrated lime are deposited. The hydration of inferiorly burned cement commences with calcium hydrosilicate gel formation; both the binding and hardening depend on the formation of this compound. Only when the water addition exceeds a certain limit, which is higher the greater is the proportion of free lime, are the short, thick needles of calcium aluminate produced. The large hexagonal plates of hydrated lime separate out earlier than in the case of properly burned cement; the separation is earlier the greater is the quantity of free lime present. Very much later (*e.g.*, after several weeks) there is a slight formation of well-defined hexagonal plates of calcium aluminate, which promotes the hardening process. No evidence was found for the formation of needles of calcium silicate during the hydration of cement. The optical properties of the calcium sulphoaluminate crystals are briefly mentioned; in order to obtain these crystals, properly burned clinker, containing not more than 0.4% of free lime and as free as possible from sulphuric acid and sulphides, must be treated for 8—14 hrs. with 30 times its weight of water. After filtration and addition of saturated gypsum solution to the filtrate, the mixture is allowed to crystallise.

S. K. TWEEDY.

Lime-alumina cement. I. S. NAGAI (J. Soc. Chem. Ind., Japan, 1930, 33, 167—169 B).—The compositions and compressive strengths of lime-alumina cements containing about 30% Al_2O_3 and 50% CaO have been determined.

O. J. WALKER.

Small-piece testing on strength of cement mortars. IV. S. NAGAI (J. Soc. Chem. Ind., Japan, 1930, 33, 87—89 B; cf. B., 1930, 462).—Further data are given of variations of the compressive strength produced by varying the method of curing. For mixed Portland cements, *e.g.*, cements containing blast-furnace slag, the strength resulting from curing for 1 day in moist air, 6 days in water, and 21 days in air is lower than that attained by curing for 1 day in moist air and 27 days in water. Cements rich in alumina, *e.g.*, "ciment fondu" and lumnite cement, diminish in strength if cured in water for long periods, on account of the dissolution of $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot n\text{H}_2\text{O}$.

H. F. GILLBE.

Hydration of calcined gypsum. W. C. HANSEN (Ind. Eng. Chem., 1930, 22, 611—613).—Earlier theories of the acceleration or retarding of the settling of calcined gypsum by salts in solution are criticised. The progress of the reaction was recorded by means of time-temperature curves. It was found that neither the solubility of gypsum in the salt solution, nor the action of the positive ion in forming an adsorption complex has anything to do with the matter. Results also show that normally the gypsum does not precipitate so fast as the hemihydrate dissolves. By seeding the mixture with newly formed gypsum nuclei the setting rate was increased. It was found that ammonium acetate and citrate, which retard the setting, also

retard the precipitation of gypsum from supersaturated solutions. Gypsum pastes examined microscopically showed the formation of crystals from the solution and not around the particles of hemihydrate. There is no evidence of gel foundation or colloidal action. It is simply a case of dissolution and precipitation, which latter may be accelerated or retarded. C. IRWIN.

Synthesis of calcium aluminates and their hydration. V. S. NAGAI and R. NAITO (J. Soc. Chem. Ind., Japan, 1930, 33, 133—137 B).—The aluminates $3\text{CaO}, 5\text{Al}_2\text{O}_3$ and $3\text{CaO}, \text{Al}_2\text{O}_3$ were prepared from the oxides; the latter aluminate always contains free lime. Comparative strength tests (cf. B., 1930, 241) showed that $\text{CaO}, \text{Al}_2\text{O}_3$ has the highest initial strength, but $3\text{CaO}, 5\text{Al}_2\text{O}_3$, having a lower initial strength, overtakes it after seven days. The other aluminates, $5\text{CaO}, 3\text{Al}_2\text{O}_3$ and $3\text{CaO}, \text{Al}_2\text{O}_3$, have lower initial strengths, which increase slightly with time. Solubility tests on the four aluminates showed that $3\text{CaO}, \text{Al}_2\text{O}_3$ and $5\text{CaO}, 3\text{Al}_2\text{O}_3$ form the hydrate $3\text{CaO}, \text{Al}_2\text{O}_3, n\text{H}_2\text{O}$. S. K. TWEEDY.

Synthesis of calcium aluminates and their hydration. VI. S. NAGAI and R. NAITO (J. Soc. Chem. Ind., Japan, 1930, 33, 164—166 B).—The effect of heating eutectic mixtures of lime and alumina has been studied. The supposed aluminate $2\text{CaO}, \text{Al}_2\text{O}_3$ is now believed to be a mixture of $3\text{CaO}, \text{Al}_2\text{O}_3$ and $5\text{CaO}, 3\text{Al}_2\text{O}_3$. The compressive strengths of the products obtained by heating the eutectic mixtures have been measured. O. J. WALKER.

Preparation of a new road binder. C. F. BROADHEAD (Gas World, 1930, 92, 621—627).—Tar obtained by carbonisation of Australian coal in vertical retorts was found unsuitable for roadmaking, even after distillation or blending. Polymerisation of unsaturated components of the tar could, however, be achieved by heating it with small amounts of formalin and aqueous ammonia in the presence of air. The properties of the substance produced are similar to those of bitumen, and it has been successfully used for road dressing. A plant for the preparation of one million gallons per year is to be constructed, and particulars are given of its design and operation.

R. H. GRIFFITH.

Colour lakes. CLAYTON.—See XIII.

PATENTS.

Hydraulic binding agent. S. L. A. ODÉN, D. R. E. WERNER, and S. GIERTZ-HEDSTRÖM (F.P. 633,197, 29.3.27).—For the purpose of regulating the binding time, cements, gypsum, hydraulic lines, etc., preferably after winnowing into portions of various granule sizes, are treated with gaseous media (sulphur dioxide or trioxide, hydrogen chloride or fluoride, aluminium chloride, carbon dioxide, etc.) which form difficultly soluble, but hydrolysable, compounds on the surface of the granules of the binding agent. The gaseous media may be used for effecting the winnowing or may be admixed with the winnowing gas. Cement may be produced from the products by mixing them with alloys of aluminium (with iron, zinc, magnesium, etc.) which evolve heat, and sometimes gases, when

treated with water. Calcium carbide, which may be impregnated with organic or inorganic substances, may also be added with the alloys. S. K. TWEEDY.

Production of aluminiferous cement. E. MOYAT (G.P. 457,463, 2.2.27).—The raw materials (essentially bauxite and lime) are heated separately to any desired temperature and then passed to a large common chamber where they fuse in consequence of mutual interaction. Slag formation is avoided.

S. K. TWEEDY.

Cement mixture of high mechanical strength. SOC. COMMERCIALE DES CEMENTS DE LA CROISSETTE SOC. ANON. (Swiss P. 124,715, 18.12.26).—The finely-ground mixture contains 30—70% of one or more cements (Portland or aluminiferous cement), 20—30% of a natural silicate which crystallises well (e.g., felspar), together with at least one mineral phosphate (apatite), and, if desired, 10—40% of a filler such as sand or marble waste.

S. K. TWEEDY.

Manufacture of porous [cementitious] products. J. A. RICE, Assr. to BUBBLESTONE CO. (U.S.P. 1,749,508, 4.3.30. Appl., 29.5.24).—A honeycombed mass of hard cementitious material is produced by mixing granules of a jelly-like material, preferably containing a large percentage of readily volatile liquid such as water, with any suitable mortar or plastic mixture capable of setting either during or subsequent to the preparation of the latter, and allowing the mixture to harden in the usual manner. A suitable jelly material is obtained by hardening glue with formalin or chromium compounds, but agar-agar, cellulose derivatives, etc. may also be employed.

S. K. TWEEDY.

Production of cementitious material. G. O. CASE, E. M. ELLIS, and L. H. MONTIGUE (B.P. 330,278, 1.3.29).—For use as a plaster or mortar a mixture of 10—25% of calcium (or magnesium) oxide and 90—75% of raw calcium (or magnesium) carbonate is ground together to the degree of fineness of Portland cement. The mixture may be used neat or mixed with sand.

C. A. KING.

Artificial stone compositions, cements, and the like. J. H. THOMPSON (Austral.P. 6171, 28.2.27).—Magnesium hydroxide or oxide is mixed with a solution of chloride of magnesium, iron (ferrous or ferric), zinc, or ammonium (which may contain magnesium nitrate or sulphate or other metal salts), or else is mixed with dehydrated magnesium chloride and then moistened with water, and then mineral or inorganic material, which is inert towards the magnesium or other salts and chosen according to the kind of stone it is desired to imitate, is incorporated with the mixture (e.g., sand, pumice, asbestos, kaolin). Colouring matter may be added, as well as metal oxides or filings. When the material is to be moulded to solid articles, solid vegetable material (timber or cane) may be added for reinforcing purposes.

S. K. TWEEDY.

Production of well-adhering coatings on asbestos-cement-slate plates. HAWENTA-PLATTEN-GES.M.B.H., and A. FRICKE (B.P. 327,871, 24.4.29).—The plates are superficially ground, heated if desired, and then wire-brushed, before being coated with paint etc.

S. S. WOOLF.

Making of roads, pavings, foundations, etc. BERRY, WIGGINS & Co., LTD., and H. H. HOLMES (B.P. 330,072, 22.4.29).—Stone or road metal which is damp or dirty is rendered suitable for coating with bitumen by an initial treatment in a mixer with a small quantity of cold creosote before adding heated bitumen thereto.

W. G. CAREY.

Materials for use in construction of roads, roofs, etc. SOUTH METROPOLITAN GAS CO., H. PICKARD, and C. M. C. HUGHES (B.P. 330,440, 23.7.29).—A dispersion of fine coal in tar is prepared at 250–300°, the proportions being such that the mixture has m.p. 97–103° (ring-and-ball) and a penetration of 12–20 at 25°. A heated mixture of 60% of stone ($\frac{1}{4}$ in. to dust), 10% of peat moss, coconut fibre, or similar fibrous material, and 30% of the bituminous binder may be used for roadmaking or may be pressed into blocks.

C. A. KING.

Covering roads and tiles and manufacturing tiles, plates, blocks, and the like. N.V. MIJNBOWEN CULTUURMAATS. "BOETON" (B.P. 310,922, 8.3.29. Holl., 3.5.28).—A mixture prepared at the ordinary temperature of a filler, solid bitumen, a bitumen solvent (e.g., carbon disulphide), and water is applied, after heating, if desired, at not above 100°, to road surfaces, concrete tiles, etc.; the water is then expelled and the mass bound by rolling or compression. (Cf. B.P. 310,923; B., 1930, 666.)

L. A. COLES.

Coating wood and other materials with a hard covering. J. A. GUSTAFSSON (B.P. 330,736, 17.5.29. Swed., 8.3.29).—The wood etc. is successively provided with a priming layer of thin and a grounding layer of thicker cement insoluble in water, preferably casein-lime cement, strewn with fairly fine sand, emery, or glass powder, and coated with an emulsion comprising a drying oil, an aqueous paste or glue solution, and finely-pulverised quartz, emery, or glass and, if desired, pigments; further coatings of nitrocellulose lacquers and decorative material may also be applied.

L. A. COLES.

Manufacture of a cement or plaster from gypsum. F. MULLIGAN (U.S.P. 1,766,448, 24.6.30. Appl., 20.2.25. U.K., 28.5.24).—See B.P. 238,949; B., 1925, 851.

Manufacture of cellular building materials. L. DESMARQUEST (U.S.P. 1,761,108, 3.6.30. Appl., 16.4.29. U.K., 18.4.28).—See B.P. 312,764; B., 1929, 599.

Pavement. L. KIRSCHBRAUN, Assr. to FLINTKOTE Co. (U.S.P. 1,767,533, 24.6.30. Appl., 8.10.28).—See B.P. 322,835; B., 1930, 192.

Gypsum fireproofing blocks. F. S. BRIDGES (B.P. 331,001, 3.4.29).

Construction of roads or pavements. UNAPHALT (ROADS), LTD., and H. E. G. RICHARDS (B.P. 330,271, 4.3.29. Addn. to B.P. 316,108).

[Photogravure process for] reproduction of the natural appearance of articles [e.g., wood] on other surfaces. OXFORD VARNISH CORP., Assees. of L. V. CASTO (B.P. 308,303, 27.2.29. U.S., 21.3.28).

[Linoleum] floor coverings. ARMSTRONG CORK Co., Assees. of S. H. HARTMAN (B.P. 317,004, 15.7.29. U.S., 8.8.28).

Surfacing for concrete floors [with linoleum etc.]. F. B. DEHN. From PARAFFINE COMPANIES, INC. (B.P. 330,951, 14.2.29).

Determining moisture in aggregate (U.S.P. 1,749,606).—See I. **Treatment of water-absorbent materials** (B.P. 326,940).—See VI. **Electric furnace** (B.P. 303,798).—See XI. **Freezing of soils** (B.P. 308,310).—See XVI.

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

Substitution of limestone by sodium chloride in fluxes of blast-furnace smelting. E. V. BRITZKE, A. N. KRESTOVNIKOV, and I. B. CHMANENKOV (Min. Suir. Tsvet. Met., 1929, 359–375).—In experiments in a laboratory electric furnace the most favourable composition of the slag was: SiO_2 42.72, FeO 2.25, Al_2O_3 15.46, CaO 33.76, MgO 2.56, Na_2O 50.4. Substitution of 10.67% CaO by 5.04% Na_2O lowers the m.p. of the slag by 300°. Sulphur combines with the slag according to the equation: $\text{Na}_2\text{CO}_3 + \text{FeS} = \text{Na}_2\text{S} + \text{FeO} + \text{CO}_2$; $\text{Na}_2\text{S} + 2\text{CaO} \cdot \text{SiO}_2 = \text{CaS} + \text{Na}_2\text{O} \cdot \text{SiO}_2 + \text{CaO} \cdot \text{SiO}_2$.
CHEMICAL ABSTRACTS.

Preservation of highly-polished surfaces [of steel]. A. C. HANSON (Ind. Eng. Chem., 1930, 22, 603–604).—Experiments in which samples of steel plate were stored for a period in atmospheres of varying humidity showed that rusting commences when the temperature falls below the dew point. However high the humidity may be, there is no rusting unless condensation occurs.

C. IRWIN.

Protective coatings for iron and steel. I. Metallic coatings. H. N. BASSETT (Ind. Chemist, 1930, 6, 241–243).—The processes of galvanising, metal spraying, and tin plating are shortly described. Calorisation is the formation of a surface alloy, Al_3Fe , with a layer of solid solution of aluminium in iron below. The Meker process, which produces solid solution with practically no Al_3Fe , gives more satisfactory results. The articles to be coated are heat-treated with powdered Al_3Fe with about 0.7% of aluminium chloride. The life of steel so treated is increased tenfold at 1100°. Other processes for the protection of iron and steel at high temperatures produce an artificial coating of triferrous tetroxide, of ferric phosphate, or of nitride. The Coslett (phosphate) method is largely used in the cycle industry.

C. IRWIN.

Determination of iron oxide in liquid steel. C. H. HERTY, JUN., J. M. GAINES, JUN., H. FREEMAN, and M. W. LIGHTNER (Amer. Inst. Min. Met. Eng. Tech. Pub., 1930, No. 311, 3–13).—Aluminium is added to the molten steel, in which alumina is then determined. The alumina tends to segregate in the small ingots.

CHEMICAL ABSTRACTS.

Electrolytic determination of non-metallic inclusions in steel. C. H. HERTY, JUN., G. R. FRITTERER, and W. E. MARSHALL, JUN. (Min. Met. Invest., U.S.A.,

Co-op. Bull., 1929, No. 44, 25 pp.).—None of the aqueous extraction methods hitherto proposed completely removes inclusions containing manganous oxide. Inclusions extracted by electrolytic means are likely to be contaminated by atmospheric oxidation, liberation of hydrogen, metallic iron, and porous cup material.

CHEMICAL ABSTRACTS.

Loss of silica in the determination of silicon in technical iron. V. LINDT (Chem.-Ztg., 1930, 54, 327—328).—Six types of iron of high, medium, and low silicon content, principally such materials as are used for transformer stampings, have been analysed by four different methods. The results showed that the loss of silica was proportional to the amount and the concentration of the hydrochloric acid used for the dissolution of the iron and for washing the precipitates. The best results were obtained by dissolving the iron in nitric acid, evaporating down with sufficient concentrated sulphuric acid to leave an excess after $\frac{1}{2}$ hr., cooling, taking up with water, boiling, filtering, and washing the residue with water very slightly acidified with hydrochloric acid. Repeated evaporation of the silica with concentrated hydrochloric acid to render the precipitate more easily filterable was found undesirable and unnecessary. The brisk heating with sulphuric acid decomposes the resistant carbides, leaving pure silica.

H. J. DOWDEN.

Physical properties of the iron-nickel-chromium system. W. A. DEAN (Rensselaer Poly. Inst., Eng. & Sci. Series, 1930, No. 26, 29—55).—The resistance to oxidation at 1000°, sp. resistance, crystal structure, hardness, and magnetostriction have been investigated. Iron-chromium alloys containing 20—40% Cr with varying amounts (0—55%) of nickel resist corrosion as well as the more expensive commercial alloys. A 50:50 nickel-chromium alloy had the maximum resistivity, and a 20:60:20 iron-nickel-chromium alloy the minimum temperature coefficient ($0.65 \times 10^{-1}/^{\circ}\text{C}.$). The predominant lattice is face-centred, but in some regions a body-centred lattice is found, and in others the two co-exist. Hardness increases as chromium is substituted for iron and nickel, reaching a maximum in alloys containing both lattices. The 80:5:15 iron-nickel-chromium alloy has a low coefficient of expansion, as have also the invar alloys. Magnetostriction is confined to iron-chromium and iron-nickel alloys, and to adjacent ternary alloys containing not more than 10% of the third component.

C. W. GIBBY.

Determination of the hardness-resistance to bending diagram of grey cast iron. J. NAVARRO (Anal. Fis. Quím., 1930, 28, 501—506).—The relation between the Brinell hardness and the resistance to bending (as measured on a Frémont machine) of grey cast iron can be expressed by the straight line $y = 3.23x + 42$, where y is the hardness and x the coefficient of bending resistance, i.e., the transverse breaking stress in kg./mm.² The irons used in the tests contained 1.88—4.35% Si, 0.03—0.17% S, 0.05—0.47% P, and 2.92—3.58% C, and their microstructure resembled that of a mild steel with graphite inclusions. The hardness of the cast irons in all cases was about 30 Brinell numbers lower than that of the

steel having a similar structure to that of the ground mass of the cast iron.

A. R. POWELL.

Method and apparatus giving the extension coefficient and breaking load of metallurgical products in thin sheets. C. JOVIGNOT (Compt. rend., 1930, 190, 1299—1302).—The test-piece (thickness e) is pressed tightly between two circular jaws, and a measured hydraulic pressure is applied through a reservoir below it till rupture of a leather sphere (radius r) occurs. Both the pressure required (P) and the deformation (f) of the metal are recorded, and $x = f^2 \div \text{constant}$ gives the coefficient of deformation and $Pr/2e$ the breaking load. The nature of the break furnishes an indication of the homogeneity of the test-piece.

J. GRANT.

Electrolytic refining of copper, using complex salts of cuprous chloride. VI. N. KAMEYAMA and K. ONODA (J. Soc. Chem. Ind., Japan, 1930, 33, 101—102 B; cf. B., 1930, 331, 195).—The presence in the electrolyte of nickel, zinc, iron, or lead in proportions up to 10% of the copper present yields on electrolysis at 50° for about 50 hrs. with a current density of 2 amp./dm.² and a copper concentration of 42—52 g./kg. a cathode deposit containing not more than 0.007% of the foreign metal.

H. F. GILLBE.

Rapid analysis of manganese-brasses. HABERLAND (Chem.-Ztg., 1930, 54, 346).—The method consists in separating the manganese as manganese dioxide by treating the evaporated nitric acid solution with solid potassium chlorate, followed by separation of the copper and lead by electrolysis, removal of the iron and aluminium by treatment with ammonia, separation of nickel as "nickel oxime," and finally calculation of zinc by difference.

H. J. DOWDEN.

System nickel-iron-copper. P. R. KOSTING (Rensselaer Poly. Inst., Eng. & Sci. Series, 1930, No. 26, 1—27).—The following properties of iron-nickel-copper alloys have been investigated: temperature coefficients of electrical resistance between 20° and 100°, sp. resistances at 20°, thermoelectric forces against copper, and coefficients of thermal expansion. The boundary found for the immiscible area agrees with previous determinations. Additions to constantan of iron up to 15%, the limit of solubility, decrease the sp. resistance and thermoelectric force and increase the temperature coefficient of resistance. Addition of copper to invar up to the solubility limit of 22% decreases the resistance and increases the temperature coefficient of resistance, the thermoelectric force, the coefficient of expansion, and the temperature at which the expansion suddenly increases. Small additions of iron to non-magnetic copper-nickel alloys make them magnetic.

C. W. GIBBY.

Corrosion of monel metal and acid-resisting bronze. M. G. BINEK (Korrosion u. Met., 1929, 5, 247—248; Chem. Zentr., 1930, i, 1045).—The monel metal (Ni 62.2, Cu 34.7, Al 3.0%, lead, and iron) and the bronze (Cu 81.6, Pb 10.0, Sn 7.9%, iron, zinc, nickel, and phosphorus) were attacked in diminishing degree (the monel metal less than the bronze) by 10% hydrochloric acid, chamber sulphuric acid (19.5%)

containing nitric acid, contact sulphuric acid, and sulphuric acid (19.5%).

A. A. ELDRIDGE.

Rapid determination of bismuth and copper in lead bullion by internal electrolysis. E. M. COLLIN (Analyst, 1930, 55, 312—318).—The method as carried out by Sand's internal-electrolysis apparatus (cf. A., 1930, 880) was devised for the determination of amounts of bismuth less than 0.01 g. in the presence of up to 10 g. of lead. To 5 g. of sample dissolved in 50 c.c. of 20% nitric acid containing 1 g. of added tartaric acid are added 2 c.c. of 2% hydrochloric acid, the silver chloride and any insoluble residue from the lead are filtered off, the precipitate is washed with hot water, the filtrate diluted to 100 c.c. and cooled, and a 2% potassium permanganate solution added to convert any tervalent into quinquevalent antimony. After addition of 5 c.c. of a 5% solution of hydroxylamine hydrochloride and dilution to 200 c.c., the liquid is electrolysed at 85—90° for 15 min. The bismuth and copper are separated from the combined deposit by means of ammonia solution and ammonium carbonate. Amounts of copper and bismuth found agreed very closely with those added to a pure lead with unknown amounts of antimony and silver.

D. G. HEWER.

Influence of cyclic stress on corrosion. D. J. McADAM, JUN. (Amer. Inst. Min. Met. Eng. Tech. Pub., 1930, No. 329).—The results are expressed diagrammatically. Even low stresses accelerate the damage due to corrosion.

CHEMICAL ABSTRACTS.

Medium for flotation cells. DYER and McCLELLAND.—See I. **Functions of coke ovens.** STEWART.—See II. **Sheet-steel enamels.** ANDREWS.—See VIII. **Aircraft finishes.** GARDNER.—See XIII.

PATENTS.

Multiple-hearth furnace. E. J. FOWLER and D. BAIRD, ASSTS. to NICHOLS COPPER CO. (U.S.P. 1,751,142, 18.3.30. Appl., 8.3.28).—In a multiple-hearth roaster, the air which cools the rabbles is used also to withdraw additional heat from some of the intermediate hearths by means of pipe coils or other devices attached to the central shaft; the heat so withdrawn may be transferred to other hearths, preferably by blowing the air into contact with the ore.

B. M. VENABLES.

Hardening of copper. N. O. HEDMAN, N. R. NÄSLUND, and J. D. LARSSON (B.P. 330,408, 17.6.29).—The copper or an article made thereof is heated to about 600° and then cooled by dipping in a bath containing 14 vols. of sulphuric acid and 1 vol. of nitric acid, to which are added 5—10 g. of potash per litre.

H. ROYAL-DAWSON.

Separation of minerals by flotation. F. G. MOSES, R. W. HESS, and R. L. PERKINS, ASSTS. to BARRETT CO. (U.S.P. 1,739,369, 10.12.29. Appl., 20.5.27).—2-Thiopyridine is used as flotation agent.

C. HOLLINS.

Manufacture of fluxes [for low-temperature welding]. J. A. ROGNON (B.P. 316,196, 24.7.29. Fr., 24.7.28).—A flux suitable for welding aluminium and its alloys, without preparatory cleaning and without a metal solder, consists of a powdered mixture of borax

18%, zinc chloride 40%, sodium bromide 34%, and aluminium or ammonium bromide 8%.

W. G. CAREY.

Metallurgical furnace. D. CUSHING (U.S.P. 1,766,110, 24.6.30. Appl., 12.12.28. U.K., 26.9.27).—See B.P. 303,042; B., 1929, 175.

Apparatus for producing metals from their oxide ores. W. E. TRENT, ASSR. to TRENT PROCESS CORP. (U.S.P. 1,767,779, 24.6.30. Appl., 11.5.28).—See B.P. 316,986; B., 1930, 669.

Lead alloy. S. BECKINSALE and H. WATERHOUSE (U.S.P. 1,766,871, 24.6.30. Appl., 11.4.27. U.K., 16.4.26).—See B.P. 272,320; B., 1927, 606.

Composition for soldering metals. K. GEISEL, ASSR. to ALUMINUM SOLDER CORP. OF AMERICA (U.S.P. 1,761,116, 3.6.30. Appl., 28.5.28. Ger., 20.2.28).—See B.P. 315,010; B., 1929, 726.

Coating, impregnating, or alloying metals and other materials with aluminium and aluminium alloys. W. SMITH, ASSR. to EXPANDED METAL CO., LTD. (U.S.P. 1,761,850, 3.6.30. Appl., 2.3.27. U.K., 17.3.26).—See B.P. 279,273; B., 1927, 943.

Production of [metallic] acid-resisting wrapping material. K. HUMPERT, ASSR. to STANIOLFABR. BURG-DORF A.-G. (U.S.P. 1,765,920, 24.6.30. Appl., 16.8.27. Switz., 17.6.27).—See B.P. 292,162; B., 1929, 61.

[Jarring mould device for] production of sound ingots. A. G. EGLER (B.P. 303,157, 21.12.28. U.S., 30.12.27).

Apparatus for intercepting furnace dust in blast furnaces. J. STOECKER (B.P. 330,928, 19.3.29).

Method and apparatus for producing a metallic spray. E. BRIDGER (B.P. 308,355, 19.3.29. N. Zealand, 22.3.28).

Lead[—antimony] alloy tanks. W. T. BUTCHER, W. CARROTT, and LOCKE, LANCASTER and W. W. & R. JOHNSON & SONS, LTD. (B.P. 330,924, 18.3.29).

Utilising [by re-compression] the lost and residual gases produced when liquefied gases of low b.p. are drawn off or transferred from one vessel to another, particularly for welding and cutting purposes. GES. F. INDUSTRIEGASVERWERTUNG M.B.H. (B.P. 306,130, 31.1.29. Ger., 16.2.28).

Heating furnace (B.P. 302,652 and 330,160). **Heating of metal sheets** (U.S.P. 1,750,884).—See I. **Reduction of metallic compounds** (B.P. 315,811). **Siliceous minerals** (U.S.P. 1,748,989).—See VII. **Zinc-white pigments** (B.P. 312,648).—See XIII.

XI.—ELECTROTECHNICS.

Copper oxide rectifier. W. OGAWA, C. NEMOTO, and S. KANEKO (J. Soc. Chem. Ind., Japan, 1930, 33, 125 B).—The contact between the cuprous oxide and copper is imperfect; the oxide has a larger electron affinity than the copper, so that the number of electrons emitted from the copper by an alternating electric impulse exceeds the number emitted from the oxide, and consequently rectification results in the direction of oxide to copper. Microscopical examination of the

contact and investigation of the relation between the form of the copper and the rectifying action support this explanation. An efficient rectifying suboxide of copper (? cuprous oxide) is prepared by heating copper embedded in magnesium oxide. S. K. TWEEDY.

Influence of the shape of carbon electrodes on the properties of carbon-zinc elements. N. S. KRIVOLUTSKAYA and G. G. MOROZOV (J. Appl. Chem., Russia, 1929, 2, 703—718).—Better utilisation of manganese dioxide and improved depolarising properties of the elements are obtained by decreasing to an optimal value the current density through the carbon-manganese dioxide agglomerate. The use of flat carbon electrodes and square agglomerates increases the capacity of the element by 25%. The thickness of the layer of agglomerate is not important.

CHEMICAL ABSTRACTS.

Corrosion of the zinc electrode in a dry cell during open circuit. S. MAKINO (J. Soc. Chem. Ind., Japan, 1930, 33, 176 B).—The presence of positive manganese dioxide electrodes in dry cells increases the chemical corrosion of the negative electrodes during open circuit. O. J. WALKER.

Sludge of transformer oils. YAMADA.—See II. **Measurement of colour and gloss.** DESBLEDS.—See V. **Controlling acid effluents.** RICHARDSON.—See VII. **Coatings for iron and steel.** BASSETT. **Determination of inclusions in steel.** HERTY and others. **Copper refining.** KAMEYAMA and ONODA. **Analysis of manganese-brasses.** HABERLAND. **System nickel-iron-copper.** KOSTING. **Determination of bismuth and copper.** COLLIN.—See X. **Alkalinity of fruit produce.** HIRSCH and RICHTER.—See XIX.

PATENTS.

Electric [muffle] furnace. H. G. CAMPBELL (B.P. 329,830, 15.5.29).—Electric heating elements are arranged in series in the furnace casing and door, and current flows through both elements whether the door be open or shut. J. S. G. THOMAS.

Electric furnace. C. E. CORNELIUS (B.P. 303,798, 8.1.29. Swed., 9.1.28).—In furnaces suitable for the manufacture of glass, water-glass, aluminous cement, etc., the electrode bodies are fixed on the furnace bottom wholly within the furnace and are spaced from all the furnace walls. J. S. G. THOMAS.

Carbon electrodes for electric furnaces. GEBR. SIEMENS & Co. (G.P. 461,094, 6.5.25).—The electrode contains a metal insertion, *e.g.*, a network of expanded metal, which is connected, *e.g.*, by spot-welding, to a metal casing which embraces the exterior of the electrode. S. K. TWEEDY.

[Heating elements for crucible] electric furnaces. L. D. J. TISSEYRE (B.P. 305,631, 7.2.29. Fr., 8.2.28).—A continuous graphite resistance, formed of rigid elements placed end to end and preferably of rectilinear or curvilinear shape, surrounds the crucible, so that current flows through the resistance in a plane or planes at right angles to the longitudinal axis of the crucible. [Stat. ref.] J. S. G. THOMAS.

Electric furnace [for dental purposes]. R. SCHMITZ (B.P. 302,594, 17.12.28. Ger., 16.12.27).—An

auxiliary furnace with independent heating elements is inserted into a member, which is suspended in the shaft above the principal heating chamber, and can be easily replaced by a simple insulating member of the same external shape. Means are provided for connecting the muffles hermetically, for observing the melting, alloying, and casting processes, and for introducing compressed air. J. S. G. THOMAS.

[Electric] tunnel kiln and its operation. F. A. J. FITZGERALD and J. KELLEHER, Assrs. to HARPER ELECTRIC FURNACE CORP. (U.S.P. 1,749,762, 11.3.30. Appl., 10.6.25).—Material moved through the furnace is heated to an intermediate temperature by radiation from a metallic resistor exposed to the furnace atmosphere, and to its final temperature by radiation and conduction from a carbonaceous resistor surrounded by an inert atmosphere within a silicon carbide enclosure. J. S. G. THOMAS.

Flux-coated [iron] electrodes for electric welding. J. MEHL (B.P. 310,341, 23.4.29. Ger., 23.4.28).—The coating surrounding an iron core contains substances which, at the m.p. of the electrode, evolve oxygen which consumes a portion of the core. Thus, *e.g.*, the coating may consist of 5% of asbestos, 90% of manganese dioxide, 3% of chromium oxide, and 2% of vanadium oxide, worked up into a paste with sodium silicate. [Stat. ref.] J. S. G. THOMAS.

Incandescence-cathode device [rectifier]. H. C. RENTSCHLER, Asst. to WESTINGHOUSE LAMP Co. (U.S.P. 1,749,780, 11.3.30. Appl., 31.1.22).—A large iron anode, and a small thorium cathode in series with and heated by a tungsten heating coil, are arranged in a sealed envelope. J. S. G. THOMAS.

Thermionic cathodes. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of L. F. PERROTT and J. J. FITZPATRICK (B.P. 303,381, 31.12.28. U.S., 31.12.27).—A nickel core coated with the carbonate of an alkaline-earth metal and preferably with a binder, *e.g.*, a cellulose compound dissolved in amyl acetate, is heated in a protective environment, *e.g.*, *in vacuo*, to a temperature above 1050° until a material of higher electron-emissive power than that of the oxide of the alkaline-earth metal is produced. [Stat. ref.] J. S. G. THOMAS.

Manufacture of oxide, sulphide, and haloid cathodes for electric-discharge devices. VEREIN. GLÜHLAMPEN U. ELEKTRICITÄTS A.-G. (B.P. 313,151, 1.5.29. Ger., 8.6.28).—A cathode core, coated with the oxide, sulphide, or halide of a refractory metal, *e.g.*, tungsten or molybdenum, or, alternatively, with one or more compounds yielding such oxide, sulphide, or halide when heated, *e.g.*, more especially ammonium tungstate or sulphotungstate, is coated with a deposit of an alkaline-earth metal and then heated for a short time at 1500°. J. S. G. THOMAS.

Electron-discharge tubes or thermionic valves [with incandescence cathodes]. N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 329,854, 29.5.29. Holl., 15.12.28).—A conductor of high sp. electric resistance, *e.g.*, of zirconium, capable of absorbing residual gases or impurities at high temperatures, but which does not emit electrons, is connected in parallel with the cathode. J. S. G. THOMAS.

Incandescence bodies, e.g., filaments, for electric incandescence lamps, vacuum tubes, etc. SIEMENS & HALSKE A.-G. (B.P. 309,536, 2.3.29. Ger., 12.4.28).—A core of tungsten is coated with eka-manganese (atomic number 75) or with an alloy of these two metals.

J. S. G. THOMAS.

Electric filaments and resistances. S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 330,280, 4.3.29).—The core of an incandescence cathode consists of zirconium together with a small proportion, e.g., 1.5–2%, of aluminium. Such cathodes are characterised by comparatively small variations of voltage being produced by relatively large variations of current flowing in the cathode.

J. S. G. THOMAS.

Luminous electric-discharge tubes. GEN. ELECTRIC CO., LTD., ASSEES. OF PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 316,611, 5.7.29. Ger., 1.8.28).—Material for replenishing the gas filling of discharge tubes is provided in the form of a compact mass; e.g., magnesium carbonate, sodium nitride, or barium azide, mixed with powdered glass or enamel, is heated by an electric heater, which is normally out of circuit, but which is automatically connected in circuit when the current loading on the tube attains a predetermined value.

J. S. G. THOMAS.

Electric lamp. J. RISLER, ASST. TO RISLER CORP. OF AMERICA (U.S.P. 1,748,396, 25.2.30. Appl., 8.8.27. Fr., 2.11.26).—An electric discharge tube filled with a mixture containing 70% of neon and 30% of helium at a total pressure of between 10^{-4} and 5 mm. Hg is claimed.

H. ROYAL-DAWSON.

Photoelectric cells. N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 319,650, 26.2.29. Holl., 25.9.28).—A layer of, e.g., calcium fluoride or magnesia is sandwiched between a layer of the photoelectric material, preferably an alkali metal, e.g., caesium, and a substratum which is usually the support for the photoelectric material.

J. S. G. THOMAS.

Photoelectric cells. ASSOCIATED ELECTRICAL INDUSTRIES, LTD., ASSEES. OF V. K. ZWORYKIN (B.P. 307,082, 2.3.29. U.S., 3.3.28).—A very thin, approximately unimolecular, photosensitive film, e.g., of caesium, produced by heating caesium trinitride, is deposited upon the conducting surface, composed, e.g., of magnesium or an alkaline-earth metal, of the cell.

J. S. G. THOMAS.

Light-[photo]electric cells. F. ROTHER (B.P. 311,701, 10.4.29. Ger., 14.5.28).—The anode or cathode, or both, or an auxiliary electrode consist(s) of cold radioactive material (e.g., thorium or thorium alloy), the α -particles from which penetrate the photo-cathode, whereby the photoelectric current is increased.

J. S. G. THOMAS.

Selenium cell. "SELENOPHON" LICHT- U. TONBILDGES. M.B.H. (B.P. 330,258, 1.3.29. Austr., 9.2.29).—In a selenium cell of the condenser type, metal plates which cannot be wetted by molten selenium constitute the electrodes and are connected with a source of current, whilst plates capable of being wetted by molten selenium are arranged at the sides or between these plates and are not in direct connexion with a source of current.

J. S. G. THOMAS.

Dry metal rectifier cells. SIEMENS-SCHUCKERT-

WERKE A.-G. (B.P. 308,306, 8.3.29. Ger., 21.3.28).—A metal plate, e.g., of copper, coated on each side with a compound (cuprous oxide) and a superposed layer of sprayed metal (zinc), is provided with spring-metal strips, e.g., of copper or brass, bearing on the metal compound layers.

J. S. G. THOMAS.

Electrode for electrolytic cells. E. F. LUNDEEN, ASST. TO WILLARD STORAGE BATTERY CO. (U.S.P. 1,749,145, 4.3.30. Appl., 31.1.27).—An electrolytic cell comprises an electrolyte containing citric acid, a film-forming electrode, and an iron-chromium electrode containing more than 10% by wt., preferably 13%, of chromium.

J. S. G. THOMAS.

Electrolyte for rectifiers, condensers, and the like. W. D. DOOLEY (U.S.P. 1,748,011, 18.2.30. Appl., 18.7.27).—The solution consists of sodium silicate 30%, sodium hydroxide 5%, gelatin 5%, and water 60%.

H. ROYAL-DAWSON.

Conserving storage-battery solution. C. B. DILTZ (U.S.P. 1,749,665, 4.3.30. Appl., 4.2.27).—A storage-battery electrolyte consists of an aqueous solution of sulphuric acid (d 1.25) containing $\frac{1}{2}$ lb. of sodium carbonate, $1\frac{1}{2}$ oz. of boric acid, $1\frac{1}{2}$ oz. of tartaric acid, 1 oz. of carmine, and 2 oz. of sodium silicate per gal.

J. S. G. THOMAS.

Manufacture of electric insulating material [from glass]. R. HADDAN. From CORNING GLASS WORKS (B.P. 330,268, 4.3.29).—Insulating sheets of thickness less than 0.015 in. are made from glass having a softening point below 700°.

J. S. G. THOMAS.

Electric insulating material. INTERNAT. GEN. ELECTRIC CO., INC., ASSEES. OF ALLGEM. ELEKTRICITÄTS GES. (B.P. 310,894, 2.5.29. Ger., 2.5.28).—Artificial material made from a glass flux containing mica or asbestos is coated with mica or mica flakes.

J. S. G. THOMAS.

Insulating materials [for under-water cables etc.]. WESTERN ELECTRIC CO., LTD. From BELL TELEPHONE LABS., INC. (B.P. 329,683, 23.2.29).—Deproteinised rubber (cf. B.P. 307,966; B., 1929, 401) and deresinated gutta-percha are combined, preferably in about equal proportions, and, if desired, montan wax or pitch is added.

J. S. G. THOMAS.

Insulation of electric wires and cables. BRIT. INSULATED CABLES, LTD., and F. J. BRISLEE (B.P. 330,552, 9.2. and 9.3.29).—Insulation embodying an esterified cellulose composition, applied, under approximately atmospheric pressure, in layers round a conductor with a textile support and substantially freed from solvent, is consolidated by heat-treatment applied simultaneously or otherwise to all the layers, so that adjacent layers unite and the composition impregnates the textile support.

J. S. G. THOMAS.

Insulating oil for electrotechnical purposes. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 301,876, 6.12.28. Ger., 7.12.27).—Raw oil obtained by a process of non-cracking distillation, e.g., of Texas crude oil, is mixed with resin.

J. S. G. THOMAS.

Electrolytic apparatus. A. E. KNOWLES (U.S.P. 1,767,375, 24.6.30. Appl., 13.4.29. U.K., 14.4.28).—See B.P. 320,388; B., 1930, 21.

Gas washer for electrolytic apparatus. A. E. KNOWLES (U.S.P. 1,767,292, 24.6.30. Appl., 15.10.26. U.K., 19.10.25).—See B.P. 261,164; B., 1927, 81.

[Multi-cellular, high-voltage, dry] electric batteries. BURGESS BATTERY Co., Assees. of W. B. SCHULTE (B.P. 312,605, 5.4.29. U.S., 28.5.28).

[Sealing the covers of] electric accumulators. W. and W. HADDON (B.P. 330,698, 18.4.29).

[Pressure] electrolyser with collecting block. J. E. NOEGGERATH (B.P. 307,934, 15.3.29. Ger., 16.3.28).

[Mica-glass] gastight [anode] seals [for mercury-arc rectifiers] and their manufacture. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of G. BUECHNER (B.P. 318,555, 5.9.29. U.S., 5.9.28).

Thermionic cathodes. E. Y. ROBINSON, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 330,331, 26.3.29. Addn. to B.P. 278,787).

Electric-discharge tubes [emitting positive-column light]. N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 315,699, 17.6.29. Holl., 16.7.28).

[Cathodes for] thermionic valves. B. LOEWE (B.P. 307,028, 1.3.29. Ger., 1.3.28).

Metallic-vapour and gaseous arc-discharge devices. P. FREEDMAN (B.P. 329,627, 16.2. and 16.12.29).

Photoelectric cell. VEREIN. GLÜHLAMPEN U. ELEKTRICITÄTS A.-G. (B.P. 329,887, 5.7.29. Austr., 2.1.29).

Manufacture of light-sensitive cells. W. ENGELKE (B.P. 330,807, 18.7.29).

X-Ray tubes. S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 331,052, 17.5.29).

Electrolytic deposition of metal on metal tubes. J. STONE & Co., LTD., W. LAMBERT, and R. W. WILD (B.P. 330,639, 15.3.29).

Gas-analysis apparatus (U.S.P. 1,751,715). Light filter (U.S.P. 1,751,220).—See I. Purification of used mineral oils (U.S.P. 1,747,161).—See II. Degreasing wool etc. (B.P. 304,295).—See V. Dehydration (U.S.P. 1,749,455).—See VII. Coating composition (B.P. 303,169). Resinous products (U.S.P. 1,737,916—8).—See XIII. Sterilisation etc. of fruit juices (B.P. 330,368).—See XIX. Photographic etching (U.S.P. 1,751,909).—See XXI.

XII.—FATS; OILS; WAXES.

Antioxidants of fats and oils. I. Antioxygenic powers of α - and β -naphthols. Y. TANAKA and M. NAKAMURA (J. Soc. Chem. Ind., Japan, 1930, 33, 107—109 B).—At room temperature both naphthols have strong antioxidising activity (linseed oil containing 0.5—5% of α -naphthol and exposed to air retained its iodine value unchanged for 78 days), α -naphthol being rather the more active, and especially at higher temperatures; at 100—150° the antioxidising power is retained, but loss occurs through volatility. It is suggested that these substances may be added to semi-drying oils to be used as lubricants. E. LEWKOWITSCH.

Antioxidants of fats and oils. II. Action of phenols and some substitution products of mono-

hydric phenol. III. Combined effects of anti-oxidants and driers on the oxidation of linseed oil. IV. Action of antioxidants of rubber and some organic compounds on the oxidation of drying oils. Y. TANAKA and M. NAKAMURA (J. Soc. Chem. Ind., Japan, 1930, 33, 126—127B, 127—128B, 129—130B; cf. preceding abstract).—II. The action of various phenols (concentration 0.01 mol.-%) on the oxidation of linseed oil exposed to sunlight for about 39 hrs. has been examined. All phenols possess an antioxygenic effect toward linseed oil; the activity sequence is pyrogallol > quinol > pyrocatechol > phloroglucinol (anhydrous) > resorcinol > phloroglucinol (2H₂O) > phenol. It is stated that the antioxygenic activity varies, not with the number of hydroxyl groups, but according to their nuclear position. The cresols and mononitrophenols are more active than is phenol, whilst the monochlorophenols possess very weak antioxygenic activity. For the nitro- and chloro-phenols, the activity is not always proportional to the number of substituents, but depends on their nuclear positions. The antioxygenic power decreases in the order *o* > *p* > *m* for the cresols, but the activities of the three isomeric mononitrophenols are almost equal.

III. Experiments using lead and manganese salts of the fatty acids of linseed oil or lead and manganese resins as driers (0.2%, calc. as the metal content) and pyrocatechol, α -naphthol, and β -naphthylamine as antioxidants (0.5%) indicate that in the case of co-existence of antioxidant and drier, the former does not entirely destroy the activity of the latter, but prolongs the induction period of drying oil; the retarding action of the antioxidant on the activity of the drier is selective. The activity sequence for antioxidants in the presence of an equal amount of a certain drier corresponds with that for the antioxidants alone (cf. Long and Egge, B., 1928, 718).

IV. The antioxidant activity of 9 commercial preparations (used in the rubber industry) in 1% concentration on "insolated" linseed oil has been examined, together with 12 organic compounds in varying concentrations. Antioxidants of rubber all possess antioxidant activity toward linseed oil; the experiments were performed at the same time as those described in II (above), and the efficiencies of the various preparations are given in terms of the activity sequence (above). 1-Methylnaphthalene, anthracene, and alizarin (although possessing phenolic properties) accelerate oxidation of linseed oil, whilst *ac*-tetrahydro- β -naphthol (resembling an aliphatic alcohol) acts as an antioxidant. The greatest antioxidant activity was exhibited by β -naphthylamine, which was followed in order by pyrocatechol and α -naphthol. C. W. SHOPPEE.

Bromometry of fats and oils. S. YUSHKEVICH (Masloboino Zhir. Delo, 1929, 45, 39—44).—Rosenmund and Kuhnenn's method is recommended for technical purposes; the reaction time should be prolonged to 10—15 min. Results obtained by Winkler's method are higher and not concordant.

CHEMICAL ABSTRACTS.

Losses of nickel in hydrogenation plants. A. KALYUZHIN (Masloboino Zhir. Delo, 1929, 45, 10—13).—Loss of nickel in the filtrate after precipitation of the

sludge (treated with sulphuric acid) with sodium carbonate is avoided by precipitation at the b.p. Hydrolysis of fat, resulting in combination of nickel with fatty acids, is avoided by using dry hydrogen. The consumption of nickel sulphate was reduced from 2.17 to 1.81 kg. per ton of fat.

CHEMICAL ABSTRACTS.

Determination of acetyl value of oils and fats by thermometric titration. T. SOMIYA (J. Soc. Chem. Ind., Japan, 1930, 33, 140—142 B).—The dry oil is heated with an equal weight of acetic anhydride in a vapour-tight pressure bottle at 140° for 2 hrs., the excess of acetic anhydride in an aliquot part being determined in the manner already described (B., 1929, 274). The method gives accurate results. C. W. SHOPPEE.

Deodorisation of fish oil fatty acid or its soap by Varrentrapp's reaction. M. HIROSE (J. Soc. Chem. Ind., Japan, 1930, 33, 90—91 B).—Odourless soap can be obtained from fish oils by Varrentrapp's reaction in the presence of wax alcohols from sperm oil, e.g., by heating herring oil with an equal weight of the alcohols and 35—42% of caustic soda at 210—240°. As the wax alcohols themselves are oxidised to fatty acids the yield of insoluble fatty acid is over 100% (iodine value 40—65 compared with 105—127 for original fatty acids). The wax alcohols treated alone gave 20—30% of fatty acids and a loss of 50—30% by volatilisation (mostly cetyl alcohol). The soap obtained by this method possesses lathering and detergent properties comparable with those of commercial laundry soap and can be used in combination with other soaps. Soap free from wax alcohol is obtained by distilling under reduced pressure.

E. LEWKOWITSCH.

Physico-chemical principles of washing materials [soaps]. L. ZAKARIAS (Chem.-Ztg., 1930, 54, 328—329).—Methods and apparatus for the measurement of the protective colloid action and foam-producing capacity of soap solutions are briefly described. Measurement of p_H of a dilute solution of soap containing an excess (0.05—0.1%) of alkali showed that when the foam is gradually removed the solution has an acid reaction; part of the soap is therefore wasted unless additional alkali be added. Reference is made to the author's proposal to use alkaline polysaccharide solutions as detergents (cf. Leipziger Monatsschr. Textilind., March, 1930). The explanation of the washing action is that the alkali peptises the dirt, which then forms a mixed colloid with the colloidal component and is thus not easily reprecipitated on the fabric. The alkaline component also enlarges the pores of the fabric and facilitates rinsing.

H. J. DOWDEN.

Composition and polymerisation of Chinese wood (tung) oil. E. R. BOLTON and K. A. WILLIAMS (Analyst, 1930, 55, 360—367).—Figures obtained by the authors' method for polymerisable matter (B., 1926, 712) and for the glyceride of elæostearic acid in the same samples of tung oil showed such close agreement that it is assumed that the polymerisable matter consists entirely of the glyceride of elæostearic acid; hence it follows that the original method of determining the polymerisable matter determines also the glyceride. The separate determination of the glyceride of elæostearic acid in tung oil was made by dividing the differ-

ence between the bromine value and the true or instantaneous iodine value by 87.2 (the difference between the bromine value, in terms of iodine, corresponding to saturation of all three double linkings, and the true iodine value corresponding to absorption of four atoms of hydrogen per mol. of acid) and multiplying by 100, since the amount of iodine monochloride absorbed by all other constituents of the oil is exactly equivalent to that of the bromine. The true iodine value (*T.I.V.*; method described in detail) may be determined graphically from the iodine values (*I.V.*) obtained after absorption has proceeded for 30 min. and for 3 hrs., or from the expression: $T.I.V. = I.V._{30 \text{ min.}} - \frac{1}{5}(I.V._{3 \text{ hrs.}} - I.V._{30 \text{ min.}})$. If, however, the iodine value of elæostearic acid is defined as the percentage of halogen in terms of iodine absorbed by four of the unsaturated atoms, then this figure may be obtained with sufficient accuracy for practical purposes by an absorption limited to 20 min.

D. G. HEWER.

Apparent iodine value of tung oil. J. VAN LOON (Chem. Umschau, 1930, 37, 175—177).—It is emphasised that the "iodine value" of tung oil depends on the type of reagent used, the time of reaction, and the halogen excess, and therefore cannot be considered as a criterion of purity or quality of the oil. True iodine values can only be obtained after some 7 days' contact with considerable excess of iodine chloride or bromine solutions.

E. LEWKOWITSCH.

Oil of cucumber seeds. G. EINHORN, A. MILSKI, and E. KALASHNIKOV (Masloboino Zhir. Delo, 1929, 45, 44—48).—The ether-extracted oil had d_{4}^{25} 0.9251, n_D^{20} 1.4750, f.p. —3.5°, saponif. value 191.1, Hübl value 115.3, Reichert-Meißl value 1.05, Polenske value 0.87, acetyl value 16.6, free acids 1.76%, unsaponifiable matter 1.91%; the acids consisted of oleic (58.49), linoleic (22.29), palmitic (6.79), and stearic (3.72% of oil) acids. The oil was richer in vitamin-A than sunflower oil. The seed contained moisture 8.0, oil 31.47, protein 29.69, carbohydrates 1.88, cellulose 13.89, pentosans 4.67, pectins 0.59, ash 3.92, P_2O_5 (in ash) 2.25, phytin 1.1, lecithin 2.60%.

CHEMICAL ABSTRACTS.

Effect of dissociated water vapour on vegetable oils. G. I. LAVIN and E. E. REID (J. Amer. Chem. Soc., 1930, 52, 2454—2455).—When water vapour is passed through an electric-discharge tube and then through cottonseed, olive, linseed, or castor oil, a white fibrous substance, insoluble in organic solvents, is produced.

H. BURTON.

Causes of turbidity in processed drying oils. V. KISELEV and N. SUKHANOV (Masloboino Zhir. Delo, 1929, 46, 24—30).—Samples of raw, bleached, and cold-treated linseed oil were processed with cobalt, manganese, and lead driers. Since the cold-treated oil gave the best results, the mucilaginous substances cannot act as a protective colloid. Cobalt-manganese driers did not cause turbidity in 5 months, whilst the lead-treated oils became turbid during the first 2 weeks. Air-blown linseed oil keeps better than unblown oil when cobalt or manganese is used, whilst the reverse holds for lead. Light has only a slight effect. Moisture accelerates the appearance of turbidity.

CHEMICAL ABSTRACTS.

Homogenisers. MÖLLERING. Medium for flocculation cells. DYER and McCLELLAND.—See I. Utilising soya beans. TAKAYAMA.—See III. Colour lakes. CLAYTON.—See XIII. Fat in wheaten products. HERD and AMOS. Fatty acids of food fats. GROSSFELD and SIMMER.—See XIX.

PATENTS.

Extraction of oils from vegetable matter [cacao bean, ground-nut, etc.]. I. S. HOCKER, Assr. to HOCKER CORP. (U.S.P. 1,751,331, 18.3.30. Appl., 10.3.26).—The material is roasted, finely-divided, and mixed with water at a temperature above the m.p. of the fatty constituent; after heating the mixture to about the b.p. and cooling, a saccharifying enzyme and sugar are added (in order to solubilise undissolved starch) and the whole is then heated at about 65° for 5–10 min. before separating the oil by centrifuging.

E. LEWKOWITSCH.

Purification of vegetable oils. J. C. HEBDEN, Assr. to HEBDEN SUGAR PROCESS CORP. (U.S.P. 1,745,367, 4.2.30. Appl., 2.4.23).—The oil is mixed with about 10% of its volume of an aqueous suspension (0.01%) of the basic tannate of aluminium, iron, or titanium (cf. U.S.P. 1,545,318–9; B., 1925, 819) at ordinary temperatures. The non-oleaginous matter, foots, etc. are coagulated and removed with the water by centrifuging.

E. LEWKOWITSCH.

Treatment of cod-liver oil. J. C. HAMILTON, B. H. THURMAN, and L. G. COPES, Assrs. to BEST FOODS, INC. (U.S.P. 1,745,851, 4.2.30. Appl., 6.8.26).—The oil is drawn off from the fats after agitation with alkali and settling, and is allowed to percolate, at room temperature, through a mass of granular active carbon through which a countercurrent of inert non-toxic gas (e.g., carbon dioxide) passes. The oil may be heated with a clarifying agent and filtered before percolation, if desired.

E. LEWKOWITSCH.

Preservation of oils [e.g., cod-liver oil]. W. G. CHRISTIANSEN, F. R. CHAPPELL, and A. E. BRIOD, Assrs. to E. R. SQUIBB & SONS (U.S.P. 1,745,604, 4.2.30. Appl., 5.11.27).—The return of unpleasant flavour to a deodorised cod-liver oil is inhibited by the addition of small amounts (e.g., 0.1%) of certain compounds of the class of phenols, aromatic amines, and aminophenols, before storage.

E. LEWKOWITSCH.

Treatment of soap [during cooling]. A. JACOBI A.-G. (B.P. 308,985, 2.4.29. Ger., 30.3.28).—Soap is shaken during the whole or part of the cooling operation.

H. ROYAL-DAWSON.

Separation of substances of dissimilar volatilities [e.g., refining fats]. E. WECKER (U.S.P. 1,766,863, 24.6.30. Appl., 23.7.26. Ger., 17.3.26).—See B.P. 277,085; B., 1927, 883.

Production of soap threads. A. H. CHARLTON, Assr. to T. B. ROWE & CO., LTD. (U.S.P. 1,764,866, 17.6.30. Appl., 1.11.26. U.K., 24.11.25).—See B.P. 267,999; B., 1927, 417.

Washing of textiles (U.S.P. 1,746,170).—See VI. **Fatty products** (B.P. 324,276). **Artificial cream** (B.P. 330,307). **Bleaching foodstuffs** (B.P. 327,670). See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Mixing, grinding, and refining of paints and enamels. S. SMITH (J. Oil Col. Chem. Assoc., 1930, 13, 163–167).—A lecture. Progress in the industry during the last 30 years is reviewed. The method of mixing recommended is to place some dry colour in the machine, to add sufficient medium to form a paste, and then gradually to feed in sufficient colour and liquid to keep the material in the machine in the paste form. For "grinding," flat stone mills are considered best, though these have been largely replaced by roller mills. A two-roll mill with differential speeds not exceeding 4:1 will give a product superior to that obtained from a triple-roll mill. In the refining of paint the single-roll mill is very satisfactory. The recently introduced disc-bar mill not only refines but machines paste, paints, and enamels to any consistency, giving an output of ready-mixed paints and enamels of 60–80 gals./hr.

F. C. HARWOOD.

Influence of drying conditions on the resistance to weathering of paints. H. WOLFF (Farben-Ztg., 1930, 35, 1933–1934).—Three blue paints of different composition were allowed to dry under entirely different conditions of temperature, humidity, and light. After 1½ years' weathering under natural conditions the general conclusion is drawn that the conditions of drying of a paint film profoundly modify the film, and hence its resistance to weathering. Specifically, films dried under the influence of ultra-violet light showed the greatest durability.

J. O. CUTTER.

Colour lakes—their manufacture and uses. H. CLAYTON (J. Soc. Dyers and Col., 1930, 46, 154–157).—Colour lakes (largely red) usually consist of the calcium or barium salts of red dyes slightly soluble or insoluble in water. The barium lake of Lithol Red is brighter and yellower than the calcium lake, and neither is suitable as a pulp lake since each requires dry-grinding for full development of its tinctorial power. Lithol Red lakes require a certain minimum period to assume stable properties between drying and grinding with oil. The calcium lakes of Permanent Red 4B and 2B are important bright bluish-reds which give blue undertones and are thus popular with lithographic printers. Helio Fast Red RL, the Hansa Yellows, and Pigment Green B, being insoluble, are not precipitated as lakes, but are simply ground with an extender. Lakes from acid dyes are chiefly precipitated with barium chloride, and the higher the temperature of precipitation the brighter is the resulting lake. Lakes made from basic dyes by precipitation with tannic acid or Katanol are generally fugitive to light, spirit, and alkali, and after drying are difficult to grind. Tungstic acid lakes are slightly faster to light than those prepared with tannic acid, but much less fast than the Fanal and Brillfast colour lakes. Although direct-cotton dyes may be easily converted into lakes by precipitation with barium chloride or aluminium hydroxide, such lakes are generally dull and have low colouring power. With the exception of red alizarin lakes, which are among the fastest to light of lake reds, the mordant dyes are little used for the manufacture of lakes. Lakes made from vat dyes are very expensive and dull in shade; a vat colour lake

ground in oil with zinc oxide fades after a few days' exposure, whereas the same lake ground with white lead requires 3 months to fade to the same extent. The degree of grinding of a lake can largely affect the rate of drying of the paint made from it. Lime Green, made by grinding a solution of Brilliant Green Crystals or Malachite Green with dry green earth, whereby the dye is absorbed to the extent of 5%, is very fast to light, whereas Brilliant Green itself is very fugitive. A cellulose lacquer should contain not more than 10% of pigment, so that lakes for this purpose should be very concentrated and finely divided. The presence of a colour lake considerably prolongs the life of the cellulose lacquer film. The Fanal lakes are produced from basic dyes by the formation of a complex salt of phosphomolybdotungstic acid; the higher the mol. wt. of the lake the faster it is to light. Other applications of lakes in industry are: for paints and enamels, inks etc., paper coating or dyeing, distempers etc., leather, wallpaper, calico printing, rubber. A. J. HALL.

Effect of the addition of chalk on bright pigments. H. WAGNER and I. KESSELRING (Farben-Ztg., 1930, 35, 1982—1984).—Chalk when used as a substrate for pigments has the following advantages: small particle size, low sp. gr., soft texture, surface activity, and slight transparency to ultra-violet light; the pastes also have high plasticity. The external weathering of chalk paints is poor, but for internal use chalk gives durability and an excellent finish. J. O. CUTTER.

Polish turpentine. S. KRAUZE (Rocz. Chem., 1930, 10, 384—396).—The following substances have been found in Polish wood turpentine oil (b.p. 152—180°): α -pinene, isopinene, $\Delta^{1,2}$ - and $\Delta^{5,6}$ -carene, and sylvestrene. Satisfactory purification of the oil cannot be effected by the action of 3% sulphuric acid or of phosphoric acid; the use of the latter reagent, moreover, raises the b.p. and destroys the optical activity of the oil. Potassium hydroxide solutions destroy the α -pinene fractions and induce polymerisation. The addition of metallic sodium affords a convenient method of purifying the oil. The presence of degradation products in the oil is best detected by addition of calcium hypophosphite in hydrochloric acid solution, when a brown to black coloration is obtained. The iodine value of crude wood turpentine oil is 315.1; after purification with sodium 355, as compared with the value 349.4 for crude German oil of turpentine and 357.2 for commercial α -pinene. The α -pinene fraction, b.p. below 160°, yields 23% of crude borneol on treatment with tetrachlorophthalic acid, whilst the fraction of b.p. 161—163°, which does not contain α -pinene, yields under analogous conditions 7—14% of borneol, which is also obtained in still smaller yields from all fractions of b.p. 163—167°. The borneol obtained from fractions of b.p. above 160° originates probably from isopinene, as β -pinene was not detected in the oil.

R. TRUSZKOWSKI.

Aircraft finishes. H. A. GARDNER (Proc. Amer. Soc. Testing Materials, June, 1930, Preprint, 9 pp.).—Various schemes for doping and finishing fabric wings etc. and exposure tests thereon are detailed. In view of the relative expense of acetate dope and the satis-

factory results obtained with nitrate dope, the latter is considered likely to replace the former. The use of pigmented dopes and top varnishes and the protective coatings particularly suitable for steel and for duralumin parts (the latter having a high coefficient of thermal expansion as compared with the former) are also discussed. S. S. WOOLF.

Resins. VI. Gutta-percha resins. E. STOCK (Farben-Ztg., 1930, 35, 1981—1982; cf. B., 1930, 623).—The usual chemical and physical constants of two gutta-percha resins, a treated resin called "Astrocopal," and of a Borneo fossil copal are given. The gutta-percha resins may be suitable for nitrocellulose, and satisfactory varnishes were made from the fossil copal. J. O. CUTTER.

Practical brush-out test for hiding power of paints. H. A. GARDNER, G. G. SWARD, and S. A. LEVY (Proc. Amer. Soc. Testing Materials, 1930, 30, ii. Preprint, 4 pp.).—An extract from a paper already abstracted (cf. B., 1930, 569). S. S. WOOLF.

Homogenisers. MÖLLERING.—See I. **Determination of lead and copper in beverages.** RICHARDSON.—See XIX.

PATENTS.

[Titanium] paints. H. WADE. From TITANIUM PIGMENT Co., INC. (B.P. 329,333, 12.11.28).—Paints comprising titanium pigments, a vehicle consisting principally of a drying oil, and a small proportion (e.g., 5% on the wt. of the paint) of cellulose nitrate as hardening agent are claimed. S. S. WOOLF.

Making a washable paint [distemper]. N.V. INDUSTRIEEL MAATS. "AMSTELLIN" (B.P. 309,553, 11.4.29. Holl., 12.4.28).—Part of the water is boiled with a mixture of sugar and starch; a mixture of linseed (stand) oil and a drying-oil varnish, with or without thinners, is stirred into this dispersion; more linseed oil is added, and, after keeping, the rest of the water and finally the dry pigment are mixed in. E. LEWKOWITSCH.

Preparation of coating compositions. BRIT. CELANESE LTD. (B.P. 303,169, 28.12.28. U.S., 29.12.27).—Normally light-sensitive synthetic resins, or coatings containing them, are darkened by exposure to ultra-violet light before use; or the resin may be darkened during manufacture in the presence of benzoic or salicylic acid as catalyst. Coatings containing such darkened resins with cellulose derivatives, especially cellulose acetate, do not change colour on exposure to light. E. LEWKOWITSCH.

Materials for marking animals. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 329,408, 26.2.29).—Such materials comprise a suspension or solution of a colouring matter in an aqueous emulsion of a free resin acid, e.g., rosin, and, if desired, an ammonium salt of a water-insoluble fatty acid, e.g., oleic acid, with or without the addition of a water-soluble organic solvent, a plasticiser, e.g., castor oil, and a disinfectant or healing agent, e.g., cresol. These products dry readily and become resistant to weathering, but are readily removable from the wool by the use of sodium carbonate solution etc. S. S. WOOLF.

Production of zinc-white pigments. C. R. BERINGER (B.P. 312,648, 29.5.29. Hung., 29.5.28).—Metallic zinc, or a metal or alloy containing zinc, is heated, at the outset by external firing and subsequently by the heat of reaction alone, in a current of oxidising gas in a continuously or intermittently revolving, cylindrical, reverberatory furnace. Improved colour of the final zinc-white due to absence of violent bubbling in the zinc bath, and increased life of fireclay furnace linings are claimed.

S. S. WOOLF.

Manufacture of "satin white." RILEY'S CHEMICALS & COLOURS, LTD., and C. H. RILEY (B.P. 327,848, 28.3.29).—The violence of the reaction between lime and aluminium sulphate is retarded by addition of sodium sulphate to either ingredient before mixing or during the admixture, a flocculent precipitate of "satin white" being formed. In a preferred process, 1 pt. of anhydrous sodium sulphate in the form of salt cake is added to 3–25 pts. of molten aluminium sulphate containing 17–18% Al_2O_3 , and lime is subsequently added.

S. S. WOOLF.

Cellulose acetate [plastic] composition. W. GUMP, Assr. to J. C. DEHLS and L. STEIN (U.S.P. 1,740,854, 24.12.29. Appl., 30.9.27).—*p*-Hydroxy-*tert*-butylbenzene, m.p. 98°, b.p. 236–238°, its 3-bromo-derivative, m.p. 50°, and 3:5-dibromo-derivative, m.p. 78°, are used as plasticisers for cellulose acetate.

C. HOLLINS.

Plastic compositions containing cellulose derivatives. E. I. DU PONT DE NEMOURS & Co. (B.P. 312,604, 3.4.29. U.S., 28.5.28).—Such compositions containing unsymmetrical diarylethanes, *e.g.*, *as*-diphenylethane, as plasticisers are claimed.

S. S. WOOLF.

Manufacture of cellulose ether-oxy lacquers and application of the products obtained. I. G. FARBENIND. A.-G. (B.P. 307,361, 5.3.29. Ger., 5.3.28).—Lacquers produced by incorporating solutions of an oxy and a cellulose ether (plasticisers, resins, etc. being added as required) show a high stability to heat and low inflammability.

E. LEWKOWITSCH.

Synthetic resin varnishes and synthetic resins. IMPERIAL CHEM. INDUSTRIES, LTD., H. H. MORGAN, and A. A. DRUMMOND (B.P. 329,313, 9.11.28).—Phenolic compounds, formaldehyde, and a fatty acid ester, *e.g.*, raw or treated tung oil, linseed oil, fish oil, or olive oil, are caused to react under heat in the presence of a volatile organic solvent and a catalyst, driers being added at any stage. The solvent is then sufficiently removed to yield a substantially solid body, which may be dissolved in a suitable solvent to produce varnishes. The phenol may first be condensed with the formaldehyde in the presence of the solvent and an alkaline catalyst, and the product then acidified with a weak acid, *e.g.*, boric acid, and distilled until the b.p. indicates that all the water has been removed; the fatty acid ester is then added and distillation continued until a drop of the solution dries clear. Dyes or pigments may be incorporated.

S. S. WOOLF.

Manufacture of [resinous] condensation products (A, B) of phenols [and aldehydes], (C) of urea and formaldehyde. (A) O. A. CHERRY and F. KURATH,

(B, C) O. A. CHERRY, Assrs. to ECONOMY FUSE & MANUF. Co. (U.S.P. 1,737,916—8, 3.12.29. Appl., [A] 19.2.28, [B] 21.4.26, [C] 23.5.27).—(A) Furfuramide or hydrobenzamide is used as condensing agent for production of phenol-formaldehyde resins by a one- or two-stage process, intended for insulating compositions. (B) Phenol and formaldehyde are condensed in presence of 2–12% of formic acid. (C) "Mono- and di-methylolureas" [hydroxymethylcarbamides] are obtained from carbamide and formaldehyde by maintaining a p_{H} of about 5 by means of buffer salts.

C. HOLLINS.

Hardening of condensation products from phenols and aldehydes. F. SCHMIDT (B.P. 316,858, 4.12.28. Ger., 3.8.28. Addn. to B.P. 304,648; B., 1930, 69).—The products of the prior patent are improved by the addition of multivalent alcohols or their esters (especially those containing free hydroxyl groups), which retard hardening and remove the turbidity from phenol-formaldehyde resins.

E. LEWKOWITSCH.

Manufacture of synthetic resin. E. E. NOVOTNY and C. J. ROMIEUX, Assrs. to J. S. STOKES (U.S.P. 1,738,310, 3.12.29. Appl., 20.9.23).—Resin-forming mixtures containing phenol, *e.g.*, equimolecular proportions of phenol and benzaldehyde, are caused to react, if desired in presence of acid, basic, or neutral catalysts under a fractionating column so adjusted as to remove water of condensation as fast as it is formed. The reaction mixture is thereby maintained at a higher temperature than would be possible under an ordinary reflux condenser and the reaction proceeds further. The catalysts are removed from the fusible condensation product, which is then rendered infusible by addition of methylene-containing hardening agents, *e.g.*, hexamethylenetetramine.

S. S. WOOLF.

Resinous condensation product from lignin and furfuraldehyde. M. PHILLIPS (U.S.P. 1,750,903, 18.3.30. Appl., 6.4.29).—Lignin and furfuraldehyde heated at 100–150° in the presence of a small amount of hydrochloric acid condense to form a brittle, black, hard resin.

E. LEWKOWITSCH.

Manufacture of artificial masses. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 327,713, 29.9.28. Cf. B.P. 327,417; B., 1930, 625).—Bleached montan wax or oxidation products thereof consisting mainly of free fatty acids of high mol. wt., *e.g.*, montanic acids, are esterified alone or in admixture with water or high-boiling solvents, *e.g.*, naphthalene, which are subsequently removed, with resinous organic condensation products containing hydroxyl groups, but free from hydroaromatic or aromatic sulphur compounds. The esterification may take place simultaneously with the production of the resinous material. Accelerators, *e.g.*, sodium bisulphate, boric acid, or anhydrous zinc chloride may be used. Fillers, dyes, gums, plasticisers, etc. may be incorporated, and the product is pressed and heated, preferably after comminution. [Stat. ref.]

S. S. WOOLF.

Manufacture of moulded articles. BRIT. THOMSON-HOUSTON Co., LTD., H. W. H. WARREN, C. G. GARTON, and A. E. SMITH (B.P. 329,273, 11.2.29).—The felted, sheeted, fibrous mass which is fed to the final presses

or calendars in the manufacture of pressboard is used as basic material for the blank in the moulding of thin-walled articles. The wet mass is soaked in alcohol, removed, and immediately immersed in a bonding material, *e.g.*, a synthetic resin. The impregnated mass is allowed to dry and moulded under heat and pressure.

S. S. WOOLF.

Production of an ornamental metallic surface on a resinous moulded composition or fibre. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of J. EATON (B.P. 301,432, 29.11.28. U.S., 29.11.27).—The inner surface of the mould is sprinkled with powdered metal, *e.g.*, bronze powder, before the composition is filled in and hot-pressed.

E. LEWKOWITSCH.

Bituminous paint. R. MEZGER, Assr. to P. LECHLER (U.S.P. 1,762,069, 3.6.30. Appl., 6.3.25. Ger., 26.4.24).—See B.P. 231,411; B., 1925, 461.

Method and apparatus for making ink [from hydrocarbons]. A. L. MILLER, Assr. to AULT & WIBORG Co. (U.S.P. 1,765,991, 24.6.30. Appl., 28.2.27).—See B.P. 286,207; B., 1929, 196.

Apparatus for preparation of Indian ink and the like. R. H. M. GREEN (B.P. 330,830, 19.8.29).

Atomisers (B.P. 330,022).—See I. **Fabric coating composition** (B.P. 303,368).—See II. **Binding agent** (B.P. 328,908).—See III. **Azo pigments** (B.P. 303,838). **Azo lakes** (B.P. 328,292).—See IV. **Cellulose esters or ethers** (B.P. 328,934).—See V. **Chromium-green** (B.P. 305,588). **Siliceous minerals** (U.S.P. 1,748,989). **Titanium oxides** (B.P. 307,881).—See VII. **Coating asbestos-cement plates** (B.P. 327,871).—See IX. **Insulation of wires etc.** (B.P. 330,552).—See XI.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Inner properties of estate rubber from Java and South Sumatra. R. RIEBL (Arch. Rubbercultuur, 1930, 14, [5]; Med. Proefstat. Rubber, 1930, [57], 223—250).—As the routine tests at the Buitenzorg laboratory relate to rubber prepared on a limited number of estates, three samples taken at random from the product of different days have been drawn from 293 estates, the total number of samples being 963. The proportion of the samples showing a vulcanisation time within 10 and 20 min. of the average (110 min.) ranged from 72% to 93%, and from 93% to 100%, respectively, for various districts. No instance was found of abnormally low tensile strength, *viz.*, more than 10% below the average, the average itself being 1.5 kg./mm.² Slope, viscosity, and plasticity were also measured; the first-named was very constant, whilst the last was distinctly higher, both for crêpe and smoked sheet rubber, than the average observed in 1925. The results of the investigation generally corresponded very closely with the average figures from the routine tests during 1928 and 1929; the restricted number of estates submitting samples for routine testing may consequently be regarded as fairly representative for Java plantation rubber. No sign was detected of deterioration in any respect. (Cf. De Vries and Riebl, B. 1930, 470.)

D. F. TWISS.

Volume increase in vulcanisation [of rubber].

R. DITMAR and C. H. PREUSSE (Chem.-Ztg., 1930, 54, 501—502).—Raw rubber submitted in a mould to the temperature conditions of vulcanisation softens, but does not increase in volume. In the presence of sulphur, however, the volume increases with consequent extrusion of the surplus. The common assumption that the density of the vulcanisate is the same as that of the original mixture is incorrect. Results are given of experiments showing the increase in volume when vulcanised in a spherical mould 2 cm. in diam. of mixtures containing varying proportions of carbon black, whiting, and talc.

D. F. TWISS.

Compressibility of rubber. L. H. ADAMS and R. E. GIBSON (J. Wash. Acad. Sci., 1930, 20, 213—223).—Using a cylinder of rubber, vol. about 20 c.c., immersed in a liquid inside a steel cylinder, applying pressure by means of a piston, and making frequent calibrations with a substance of known compressibility, *e.g.*, steel, measurement was made of the compressibility of rubber at intervals of 1000 bars from 12,000 bars downwards. The compressibility curve for soft rubber resembles that of a liquid such as amyl alcohol and shows little in common with that of a solid; the curve for vulcanite, although different from that for a solid such as sodium chloride, is more like that of a compressible solid than of a liquid and is similar to that of potassium. Soft rubber is almost twice as compressible as vulcanite at atmospheric pressure, but the difference is greatly reduced at pressures above 5000 bars. The large proportion of sulphur in vulcanite has a decided influence on its compressibility, lowering it at the lower pressures and keeping it large at higher pressures. A piece of vulcanite of normal volume 100 c.c. (containing 27.4% by wt. of sulphur) would become 85.4 c.c. at 12,000 bars, whereas a piece of soft rubber containing 10% of sulphur would decrease from 100 c.c. to 82.8 c.c.

D. F. TWISS.

Determination of traces of copper in [rubber] materials. A. RUTHING (Chem.-Ztg., 1930, 54, 403).—Methods for the colorimetric determination of copper (less than 0.005%) in unrubbed and rubbered materials prepared by cold vulcanisation with sulphur chloride are described.

E. H. SHARPLES.

Colour lakes. CLAYTON. **Gutta-percha resins.** STOCK.—See XIII.

PATENTS.

Manufacture of rubber or of polymerisation products of diolefines, which are plastic and/or elastic. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 329,969, 27.2.29).—Plastic and/or elastic polymerisation products with exceptional stability on storage are obtained from diolefines, such as isoprene or butadiene, by adding a dissolved or emulsified anti-ager to the latex or latex-like products from which the polymerised material is to be separated. The anti-ager may be introduced before or during coagulation of the latex-like suspension and in such quantity as to give 0.5—2% in the coagulated product. Compounding ingredients also may be introduced into the latex or latex-like products prior to coagulation.

D. F. TWISS.

Emulsions containing india-rubber. COLAS PRODUCTS, LTD., L. G. GABRIEL, and J. F. BLOTT (B.P.

329,965, 27.2.29).—A mixture of oil, *e.g.*, linseed oil, or molten or liquid bituminous material and rubber latex is subjected to agitation in a mixing apparatus preferably of the colloid-mill type. A preferred mixture is one of bitumen, artificially produced from petroleum, with sufficient latex to introduce 25% by wt. of rubber, calculated on the emulsion. No additional emulsifying agent is necessary, emulsification being effected solely by the latex, but a small proportion of stabilising agent, such as alkali caseinate, may be added to the finished emulsion (cf. B.P. 254,004; B., 1926, 748).

D. F. TWISS.

Manufacture of rubber goods. SOC. ITAL. PIRELLI (B.P. 301,476, 30.11.28. Italy, 30.11.27).—A cylindrical core, heated internally by hot water or electrically, is caused to travel through latex, which may have been preserved and/or compounded, and emerge therefrom coated with a layer of compact coagulated rubber. The thickness of the deposit may be regulated by adjusting the temperature and surface speed of the core and the depth of the latex. In order to prevent thickening or coagulation except at the surface of the core, a coiled pipe through which cold water is circulated may be provided in the latex vessel, the bottom of which may be formed by a resilient membrane with an aperture permitting the liquid-tight passage of the core (cf. B.P. 284,608 and 292,964; B., 1929, 612; 1930, 205).

D. F. TWISS.

Cementing methods and substances. [Attaching rubber to other surfaces.] RUBBER LATEX RES. CORP. (B.P. 309,168, 18.3.29. U.S., 6.4.28).—Laminated articles comprising a base, *e.g.*, metal, Bakelite, wood, and a mass of vulcanised rubber united thereto by means of an intermediate film consisting of a hardened or coagulated water-resistant proteid and rubber are claimed. Heat-coagulable proteins, *e.g.*, egg-albumin, blood, hæmoglobin, are preferred, but dextrin, glue, etc., subsequently treated with formaldehyde, or gelatin or isinglass in dichromate solution, subsequently exposed to light, may be used. After the protein has been rendered water-resistant *in situ*, the vulcanisable rubber mass is superimposed and the assembled article is vulcanised, whereby the rubber of the mass and the film are simultaneously vulcanised and mutually integrated.

S. S. WOOLF.

Oxidisable organic compounds. [Anti-agers for rubber.] GOODYEAR TIRE & RUBBER Co., Assees. of A. M. CLIFFORD (B.P. 316,251, 19.4.29. U.S., 26.7.28).—Alkyl-naphthylamines are used as anti-agers for rubber; examples are *n*-butyl- β -naphthylamine, b.p. 178–183°/4–5 mm., the α -isomeride, heptylnaphthylamines, etc.

C. HOLLINS.

Drying apparatus (U.S.P. 1,750,813).—See I. Rubberised fabric (B.P. 303,368).—See II. Artificial silk products (B.P. 328,627).—See V. Treatment of water-absorbent materials (B.P. 326,940).—See VI. Insulating materials (B.P. 329,683).—See XI.

XV.—LEATHER; GLUE.

Acido-proteolytic enzymes in the tanning industry. C. GORINI (Zymologica, 1930, 5, 41–46).—The use of dung or glandular (pancreatic) enzymes for

the treatment of hides after unhairing may be advantageously replaced by that of the author's acido-proteolytic enzymes, which are capable of carrying out digestion in an acid medium.

T. H. POPE.

Tanning and dyeing of furs. A. DOHOGNE (Boll. Uff. Staz. Sperim. Ind. Pelli, 1930, 8, 168–171).—After steeping for 1–2 days, with several changes of the water, rabbit skins are drained, centrifuged, defleshed, and, tanned. In the Ghent district non-sulphited quebracho is mostly used for tanning, but in Leipzig the skins are pickled and treated on the flesh side with fish oil. Use is sometimes made of formaldehyde, "chrome," alum, oil, synthetic, or combined tanning. The tanned skins are greased, dried at 35°, subjected to fulling etc., defatted, mordanted with potassium dichromate, copper sulphate, ferrous sulphate, or chrome or potash alum, and decolorised by alkaline hydrogen peroxide, hyposulphite, etc., often in presence of a catalyst. Dyeing, either of the whole skins or of the tips only, is mostly effected by means of phenols, amines, phenylamines, such as *p*-phenylenediamine etc., which are capable of giving, on oxidation, colouring matters absorbed by the skins. For dyeing to imitate beaver, use is made of a dye bath containing various Ursol dyes, ammonia, and hydrogen peroxide. The dyed skins are washed in running water, centrifuged, and dressed on the flesh side with a solution of sodium chloride, glycerin, and yolk of egg.

T. H. POPE.

Comparison of the quinhydrone and hydrogen electrodes in solutions containing tannin. E. L. WALLACE and J. BEEK, JUN. (Bur. Stand. J. Res., 1930, 4, 737–745).—Measurements of the p_H of solutions containing chestnut and quebracho tanning extracts and of systems consisting of leather and water in equilibrium have been made with the hydrogen and quinhydrone electrodes and the results are compared graphically. In the leather-water systems the value of $p_{H(H)}$ is slightly less than that of $p_{H(Q)}$, and although no general expression can be formulated for converting one series of results into the other, corrections can be applied to the quinhydrone electrode readings to obtain the corresponding hydrogen electrode readings in certain cases where the nature of the solution is known. In solutions of powdered chestnut-wood extract the values of $p_{H(H)}$ decreased in a smooth curve convex to the axis of composition with increasing tannin content, whereas the values of $p_{H(Q)}$ increased along a curve concave to the same axis under the same conditions. Similar results were obtained for solutions of solid, ordinary quebracho extract, but the differences were more marked, $p_{H(H)}$ decreasing from 4.95 with 0.25% of tannin to 4.75 with 7% of tannin and $p_{H(Q)}$ increasing from 4.1 to 5.25 in the same range.

A. R. POWELL.

Improved Kubelka-Němec sedimentation method of determining the insoluble matter in tannin analysis. V. NĚMEC and E. KUDLAČEK (Collegium, 1930, 72–74; cf. B., 1929, 991).—An inverted bottle (diam. 9 cm.) from which the bottom has been cut away is fitted with a rubber stopper through which pass a wide and a narrow glass tube. The upper end of the narrow tube is bent through 180°, and its lower end is fitted with a rubber connexion, clip, and delivery tube.

The upper end of the wide tube is 1.0—1.1 cm. above the open end of the other tube. The analytical tannin solution is poured into the inverted bottle (any excess runs out through the wide tube), and the bottle is covered with a glass; after being set aside for 4—5 min. particles $>1\mu$ will have settled below the mouth of the narrow tube. The supernatant liquor is then withdrawn through the narrow tube and an aliquot portion evaporated to dryness.

D. WOODROFFE.

Action of water on vegetable-tanned leather.
II. Resistance of the collagen-tannin compound to hydrolysis on long washing. H. B. MERRILL (J. Amer. Leather Chem. Assoc., 1930, 25, 173—182; cf. B., 1929, 567).—Vegetable-tanned calfskin samples were extracted first with chloroform, then in a Wilson-Kern extractor with distilled water for 1—121 days. Water, hide substance, and ash were determined in the original and the dried, extracted leathers, and from these results and the weight of the leathers before and after extraction the amount of tannin combined with 100 g. of hide substance was calculated. The amount of residual tannin calculated by means of the residual weight of leather was less than that obtained by the analysis of the residue. After 65 days' washing, the amount of material removed thereby was so small that the residue must have been a tannin-collagen compound which was hydrolysed only to a negligible extent. In similar tests with vegetable-tanned hide powder similar results were obtained, but equilibrium was reached more quickly. The quebracho-collagen compound was more resistant to hydrolysis than the oak-collagen, whilst the gambier-collagen was much less resistant. The same initial tannin content of the leather was obtained when the pelt or powder was tanned with a 2% solution as with a 4% solution of tannin, but less resistance to hydrolysis was shown by the product tanned in the stronger solution. The initial ratio of tannin to hide substance was increased by increasing the time of tanning. Hide substance combines irreversibly with a limited amount of tannin, after which it combines with more tannin, which is removable by washing, the amount depending on the amount originally fixed by the leather and on the kind of tanning material used.

D. WOODROFFE.

Skin- and bone-glues. H. MAIER-BODE (Kunst-dünger- u. Leim-Ind., 1929, 26, 327—329, 339—341, 351—354; Chem. Zentr., 1930, i, 927).—It is confirmed that the viscosity of fresh glue liquor is smaller than that of a sol of equal concentration prepared from the solid material; it is considered that, on drying, reaggregation of the gluten takes place. Skin and bone glues are conveniently tested by determining the viscosity of a 17.75 or 20% solution at 30° and 40°. A modification of Ostwald and Köhler's test differentiates skin glue from bone glue.

A. A. ELDRIDGE.

Colour lakes. CLAYTON.—See XIII.

PATENTS.

Imparting a blue colour to leather polish. S. H. DONNINGTON (B.P. 330,014, 7.3.29).—Ferric ferrocyanide is well stirred into the usual mixture of oils and fats (except linseed oil) in the liquid or semi-liquid state. [Stat. ref.]

H. ROYAL-DAWSON.

Depilating hides and skins. M. BERGMANN (U.S.P. 1,765,199, 17.6.30. Appl., 27.7.25. Ger., 12.2.25).—See B.P. 247,826; B., 1926, 377.

Dehairing hides and skins and preparing them for tanning. C. J. M. M. LE PETIT, Assr. to RÖHM & HAAS Co. (U.S.P. 1,767,536, 24.6.30. Appl., 21.5.26. Fr., 18.4.25).—See B.P. 250,907; B., 1926, 1022.

Preparation of gelatin. A. PANSKY (U.S.P. 1,761,362, 3.6.30. Appl., 14.12.23. Fr., 24.11.23).—See B.P. 221,702; B., 1924, 956.

Adhesive agent (B.P. 328,908).—See III. **Bleaching of skins etc.** (B.P. 310,030). **Treatment of fur** (B.P. 330,598).—See VI.

XVI.—AGRICULTURE.

“Build-up and break-down” of soil zeolites as influenced by reaction. P. S. BURGESS (Arizona Agric. Exp. Sta. Tech. Bull., 1929, No. 28, 101—135).—The base-exchange capacity of many arable soils can be increased by treatment with solutions of alkalis or alkali salts, the increase being closely proportional to the hydroxyl-ion concentration of the solution. The zeolitic complex can be partly (1.2—18.4%) broken down by leaching with relatively large amounts of dilute solutions of organic acids (including carbonic). Appreciable amounts of hydrogen zeolite were formed. The break-down was more rapid and complete if solutions of aluminium chloride or sulphate were used. It is probable that active build-up of soil zeolites takes place largely in the calcareous soils of arid and semi-arid regions where alkaline soil solutions prevail, and that disintegration is more common in the more or less acid soils of humid regions.

CHEMICAL ABSTRACTS.

Comparison of soil-liming materials. A. T. WIANCKO, G. P. WALKER, and S. D. CONNER (Indiana Agric. Exp. Sta. Bull., 1929, No. 329, 3—24).—Ground limestone and marl are preferred for Indiana sandy loam and silt loam. The coarse limestone particles have a definite, although delayed, action. Moderate applications affect the acidity of the surface layer only.

CHEMICAL ABSTRACTS.

Impoverishment of forest soils by use of the litter. A. NĚMEC (Forstarchiv, 1929, 497—503; Bied. Zentr., 1930, 59, 257—258).—Low proportions of nutrients in many forest soils are attributed to the annual removal of mineral matter in the forest litter. In this respect the available potash is least affected.

A. G. POLLARD.

Phosphoric acid content of forest humus soils. A. NĚMEC (Forstwiss. Zentr., 1929, 721—732; Bied. Zentr., 1930, 59, 247—248).—The citric-soluble phosphate content of forest soils is generally smaller than that of similar arable soils. The humus layer of forest soils is the most abundant source of easily soluble phosphate, and in lower layers there is a general parallelism between the citric solubility of phosphates and the humus content. The humus layer under pines has a relatively low citric-soluble phosphate content. The impoverishment of forest soils by removal of the *débris* of leaves etc. for litter is emphasised.

A. G. POLLARD

Nutrient contents of Rhineland soils. G. HAGER, (Landw. z. Rheinprovinz, 1929, No. 47; Bied. Zentr. 1930, 59, 256—257).—A survey of Rhineland soils is recorded. The productivity of soils is not governed by the nutrient content alone, but is dependent on many other factors, notably the proportions of clay and humus, tilth, soil depth, and climatic conditions. With due consideration of these factors, the Neubauer test gives valuable information as to soil fertility.

A. G. POLLARD.

Determination of the nutrient requirement and inoculability of soils by Niklas' *Azotobacter* method. L. KREYBIG (Forts. landw., 1929, 4, 12; Bied. Zentr., 1930, 59, 253—256).—Growth of *Azotobacter* in soils leads to increased citric solubility of the phosphates. In soils deficient in phosphate Rhenania phosphate increased the growth of *Azotobacter* to a greater extent than did superphosphate, particularly in humus soils with low exchange acidity. The application of phosphatic fertilisers increased the nitrogen fixation in phosphate-deficient soils. The *Azotobacter* method is considered satisfactory for the practical determination of the phosphate requirement of soils.

A. G. POLLARD.

Determination of nitrogen in soils by the Mitscherlich vegetation method. A. Y. SAMOILOVA (Udobr. Urozhai, 1929, 355—358).—Mitscherlich's equation, $\log(A - Y) = \log A - cx$, where Y is the yield, A the possible maximum yield obtained with an optimum quantity of the factor studied, c the activity coefficient of the nutrient factor, and x the amount of nutrient added, was employed. In determining the nitrogen resources of the soil the use of a coefficient calculated from experimental data gives more trustworthy results than that of Mitscherlich's coefficient 0.122. The establishment of the working formulæ is described.

CHEMICAL ABSTRACTS.

"Kalkammonsalpeter." O. NOLTE and H. MÜNZBERG (Mitt. Deut. landw. Ges., 1930, 45, 37; Bied. Zentr., 1930, 59, 258—259).—Comparison is made of the efficiency of "Kalkammonsalpeter," ammonium sulphate, and sodium nitrate in field trials. In dry seasons sodium nitrate was particularly effective, but in wet summers, when leaching losses were considerable, ammonium sulphate proved the more profitable. "Kalkammonsalpeter" is preferable to either of the above for cereals and root crops.

A. G. POLLARD.

Changes in soil reaction produced by ammonium, potash, and potash ammonium superphosphate GERLACH (Superphosphat, 1929, 5, 282; Bied. Zentr., 1930, 59, 248—249).—The increasing acidity of many German soils is more definitely attributable to the normal processes of cropping and leaching than to the use of physiologically acid fertilisers. Systematic liming or marling is preferable to the withholding of valuable, if slightly acid, fertilisers.

A. G. POLLARD.

Influence of roasting raw phosphates on the availability of phosphoric acid. N. D. SMIRNOV (Udobr. Urozhai, 1929, 359—363).—High-grade phosphate (23—30% P_2O_5) showed a slight increase of citrate-soluble phosphorus on roasting. Pot experiments with

oats on podsol and degraded chernozem revealed no superiority.

CHEMICAL ABSTRACTS.

Obtaining a concentrated triple fertiliser. N. E. PESTOV and E. A. KALABEKOVA (Udobr. Urozhai, 1929, No. 7, 421—425).—For the preparation of a fertiliser containing nitrogen, phosphorus, and potassium, but little chlorine, the optimal mol. ratio of phosphoric acid:potassium chloride:ammonia is 1:1.25:1. This gives a salt of composition KH_2PO_4 78.63, $NH_4H_2PO_4$ 19.73, KCl 1.64%.

CHEMICAL ABSTRACTS.

Apatite and nepheline rock of the Khibin Mts. as a direct fertiliser. I. P. SOMOV (Udobr. Urozhai, 1929, 412—418).—Soils of pH 4.4—4.1 responded, and of pH 5.3—5.2 did not respond well, to treatment with apatite. Addition of ammonium sulphate rendered the apatite more effective. The nepheline is a satisfactory source of potassium.

CHEMICAL ABSTRACTS.

Fertilising action of bone meal. E. TRUNINGER and F. KELLER (Landw. Jahrb. Schweiz, 1929, 43, 931—945; Chem. Zentr., 1930, i, 1028).—Bone meal, preferably defatted, gives good results only on acid soil. With one application raw bone meal gave 0.33, or with a double quantity 0.5, of the increased yield afforded by superphosphate. The activity depends largely on the fineness of division. The value of bone-meal nitrogen is estimated to be 50% of that of nitrate nitrogen.

A. A. ELDRIDGE.

Conditions of application and the effect of phosphate on chernozem. VIII. Reversion. M. A. EGOROV (Udobr. Urozhai, 1929, 401—412).—The sedimentation of soil extracts on treatment with calcium carbonate followed by carbon dioxide, with sodium phosphate and calcium salts, was studied. Various types of lime do not act on soils in the same way.

CHEMICAL ABSTRACTS.

Effect of fertilisers on the quality of the juices of sugar cane. R. F. GARCIA (Ann. Rep. Insular Exp. Sta. Porto Rico, 1929, 68—88).—In general, nitrogenous fertilisers gave the highest yields. Soluble manganese salts exercised a negligible effect.

CHEMICAL ABSTRACTS.

Effect of nitrogenous fertilisers on pastures. O. NOLTE, H. MÜNZBERG, and H. KOCH (Mitt. Deut. landw. Ges., 1929, 44, 385; Bied. Zentr., 1930, 59, 263).—Results of meat and milk trials on fertilised pastures are recorded.

A. G. POLLARD.

Comparison of the fertilising value of manure and mineral fertilisers. S. V. SHTSCHERBA (Udobr. Urozhai, 1929, 418—420).—Experiments with sugar beet and potatoes are recorded. Values for the starch content of potatoes are: without manure or mineral fertilisers 14.4, with manure 14.6, with mineral fertilisers 18.45, with both 18.8%.

CHEMICAL ABSTRACTS.

Composting barnyard manure with sulphur and rock phosphate. W. G. FRIEDMANN (Georgia Agric. Exp. Sta. Bull., 1929, No. 154, 1—14).—Untreated composts suffered a greater loss of nitrogen than did those containing sulphur; with 2% S only 10% or less of the nitrogen was lost. Superphosphate was more effective than sulphur in preventing loss of nitrogen from liquid

manures. Part of the phosphorus of a manure-rock phosphate-sulphur mixture becomes available during composting.

CHEMICAL ABSTRACTS.

Manuring and crop quality in root crops. KLEE-BERGER (Superphosphat, 1929, 5, 74—76; Bied. Zentr., 1930, 59, 259—260).—The effect of unbalanced fertilisation on the quality of sugar beet and potatoes is examined. In this respect nitrogenous fertilisers are of first importance. The use of phosphate fertilisers in amounts based on Neubauer trials may be unsatisfactory, since this method of examination allows of no consideration of quality. There is no foundation for the opinion that phosphate fertilisers are of minor importance for potatoes.

A. G. POLLARD.

Manurial trials with potassium ammonium superphosphate on root crops. DENSCH (Superphosphat, 1929, 5, 53; Bied. Zentr., 1930, 59, 259).—The efficiency of the nitrogen of "Kali-ammon-superphosphat" is similar to that of the simpler nitrogenous materials, and the fertiliser proved suitable for use on acid soils and for acid-sensitive plants.

A. G. POLLARD.

Fineness of ground sulphur sold for dusting and spraying. L. R. STREETER and W. H. RANKIN (New York State Agric. Exp. Sta. Tech. Bull. No. 160, 1930, 16 pp.).—A consideration of methods, including a description of a micro-projection method, of determining the degree of subdivision of ground sulphur, together with details of an examination of five commercial brands.

E. HOLMES.

Supplements for copper fungicides. E. B. HOLLAND, C. O. DUNBAR, and G. M. GILLIGAN (Mass. Agric. Exp. Sta. Bull., 1929, No. 252, 94—112).—The efficiency of insoluble copper fungicides depends largely on the degree of dispersion and other physical characteristics. "Soluble copper" is not a criterion, since various organic compounds produce filterable copper of low activity. The efficiency of the spray is increased by wheat flour, gelatin, soap, or tannic acid.

CHEMICAL ABSTRACTS.

Effect on plants of cyanide fumigation following spraying with Bordeaux mixture. O. BUTLER and R. R. JENKINS (Phytopath., 1930, 20, 419—429).—Injury to foliage following the fumigation with hydrocyanic acid of plants previously sprayed with Bordeaux mixture depends on the proportions of lime and copper sulphate used in preparing the latter. Exposure of various Bordeaux mixtures to hydrocyanic acid showed that no cupric cyanide was formed when the ratio of copper sulphate to quicklime used was 1:0.2 and in negligible amounts only when the ratio was 1: >4. In a 1:0.2 mixture insoluble cuprous cyanide was formed, and in a 1:6 mixture a soluble double cyanide. In mixtures between 1:1 and 1:6 the amount of cupric cyanide formed decreased and that of the double cyanide increased with increasing proportions of lime used. The double cyanide is injurious to foliage. A neutral Bordeaux mixture is the only type suitable for use when cyanide fumigation is to follow.

A. G. POLLARD.

Tank-mixture method of using oil spray. R. H. SMITH (J. Econ. Entom., 1930, 23, 376—382).—Uniform pine oil-water mixtures may be maintained in spray tanks and in the spray by the use of suitable paddle

agitators revolving at 200—250 r.p.m. More oil is retained by sprayed surfaces at a distance from the nozzle than close to it. The quantity of oil deposited is largely determined by the nature and quantity of the emulsifier used, soap causing a smaller deposit than other commonly used materials. The effect of an emulsifier in oil-water mixtures cannot be judged by its effect in other spray mixtures.

A. G. POLLARD.

Borax as an insecticide for protecting seed. H. H. SCHWARDT (J. Econ. Entom., 1930, 23, 401—404).—Powdered borax controlled the rice weevil when used at the rate of 10 oz. per bushel of grain, and the four-spotted bean weevil when 20 oz. per bushel of cow peas were used. Treated grain cannot be fed to cattle.

A. G. POLLARD.

Differentiation between good and poor germinative capacity of seeds by chemical means. H. O. PAECH (Diss. Agric.-Chem. Inst. Univ. Breslau, 1929; Bied. Zentr., 1930, 59, 268—270).—The germinative activity of seeds is closely related to their dehydrogenase activity. Of numerous colorimetric methods for the determination of the latter, the most satisfactory is that using *m*-dinitrobenzene. Comparative tests should be made on the same day with freshly-ground material.

A. G. POLLARD.

Seed treatments for the control of barley stripe. C. S. REDDY and L. C. BURNETT (Phytopath., 1930, 20, 367—390).—Results of trials with a number of mercurial fungicides are recorded and discussed.

A. G. POLLARD.

Nitrophosphates. CALCAGNI.—See VII. **Drinking water for cattle.** McLACHLAN.—See XXIII.

PATENTS.

Fertilisers. A. A. J. and C. A. A. VILAIN (VILAIN FRÈRES) B.P. 309,175, 2.4.29. Fr., 7.4.28).—The product obtained by treating dolomite with nitric acid is claimed.

H. ROYAL-DAWSON.

Product for promoting plant growth. R. A. BAKER and C. C. CARPENTER (U.S.P. 1,747,281, 18.2.30. Appl., 30.6.27).—The product consists of a mixture of an alkaline-earth carbonate (*e.g.*, limestone or dolomite) and aluminium sulphate.

H. ROYAL-DAWSON.

Freezing soils by means of carbonic acid. H. DEHOTAY (B.P. 308,310, 20.3.29. Belg., 21.3.28).

Production of mixed manure containing phosphoric acid and nitrogen. F. G. LILJENROTH (U.S.P. 1,761,400, 3.6.30. Appl., 23.7.27. Swed., 13.8.26).—See B.P. 275,843; B., 1927, 826.

XVII.—SUGARS; STARCHES; GUMS.

Absorption of atmospheric oxygen by limed cane juice. J. A. AMBLER (Ind. Eng. Chem., 1930, 22, 357—362).—Quantitative studies of the rates of absorption of oxygen by cane and beet juices and pure sugar solutions containing up to 0.06% CaO, aerated under constant conditions at the ordinary temperature, showed that cane tannin and invert sugar undergo oxidation under these conditions, with formation of calcium salts of organic acids and consequent lowering of alkalinity. The rate of oxidation is greater at high than at low alkalinities. It is concluded that under technical conditions of liming and defecation of cane juices considerable

oxidation must occur in view of the high temperatures employed, the common use of air for mixing the limed juices, and the high alkalinities which occur locally before the lime is uniformly mixed with the juice.

J. H. LANE.

Comparison of some white-sugar methods.

H. A. FABER (Ind. Eng. Chem., 1930, 22, 366—367).—In American beet-sugar factories fully 85% of the available sugar in the thick juice is usually obtained as standard white granulated without remelting. This is due to the use of live steam in the vacuum-pan coils. In Javan and Continental European white-sugar factories the vacuum pans are heated by exhaust steam or vapour from the first effect of the evaporators, and in consequence circulation of the massecuite is sluggish, crystal growth is retarded, and water has often to be drawn into the pan to remedy supersaturation and false grain formation. A soft type of crystal is produced, centrifugal work is inefficient, and the mother-liquors are highly viscous. Only about 15% of the total available sugar is obtained as white sugar without remelting; to recover the whole, four boilings are necessary, as compared with two in American beet-sugar factories. The author strongly advocates the use of live steam for pan boiling in cane-sugar factories, for the production of white sugar.

J. H. LANE.

Determination of polarisation, ash, alkalinity, p_H value, and colour of molasses with one weighing. V. KONN (Z. Zuckerind. Czechoslov., 1930, 54, 325—331).—For factory analyses of beet molasses the author proposes to weigh out 26 g., make up to 200 c.c. with neutral distilled water, and use aliquot portions for the various determinations, viz., 20 c.c. for alkalinity by titration, 10 c.c. for p_H value by Clark's method, 20 c.c. diluted to 200 c.c. for colour degrees (Stammer), 10.4 c.c. diluted to 100 c.c. for carbonate ash by Sandera's conductimetric method, and 100 c.c. clarified with 3.5 g. of dry basic lead acetate for polarisation. With ordinary beet-factory or refinery molasses the results obtained agree satisfactorily with those found by the conventional Czechoslovakian methods, according to which the carbonate ash is determined by incineration. Rather wide discrepancies in ash values, up to 1.13%, were found, however, with molasses 40 years old and also with molasses containing much raffinose, and with a sample of Javan cane molasses. In general, with all these molasses the carbonate ash was only about four fifths of the sulphated ash, even after the usual subtraction of one tenth from the latter.

J. H. LANE.

Influence of the reaction on the technique of the preparation of lactose, condensed milk, and ice-cream. P. PARISI (Giorn. Chim. Ind. Appl., 1930, 12, 225—235).—The "sandiness" of condensed milk and of ice-cream and the low yield in lactose from concentrated whey depend essentially on crystallisation of the lactose, which is closely connected with the phenomenon of mutarotation and this with the temperature and hydrogen- and hydroxyl-ion concentrations of the solution. The velocity of transformation of the β -stereoisomeride of lactose into the α -form at a given temperature and a given p_H is the resultant of the separate velocities due to the hydrogen and hydroxyl ions. The actions

of these ions are represented by geometrical progressions having as exponents the p_H and p_{OH} values, the velocity coefficient k_2 being given by $ax^{p_H} + by^{p_{OH}}$. The logarithm of the common ratio of the geometrical progression as a function of the hydroxyl ions is seven times as great as that of the corresponding ratio for the hydrogen ions. The two curves intersect at p_H 6.7—6.8 and the minimum value of k_2 occurs at about p_H 5.0.

T. H. POPE.

Fertilisers and sugar cane. GARCIA.—See XVI. **Fermentation of sugar.** TOMODA and KOCHI. **Fermentation of molasses.** TOMODA.—See XVIII. **Lactose from skim milk.** NABENHAUER. **Test for sugars in jams.** LEWIS. **Dextrins in food materials.** GROSSFELD and HOLLATZ.—See XIX.

PATENTS.

Manufacture of sugars and polyhydric alcohols.

J. Y. JOHNSON. FROM I. G. FARBERIND. A.-G. (B.P. 327,193, 3.12.28).—In the synthetic production of sugars from formaldehyde or its polymerides by heating with oxide catalysts, greatly increased yields of sugars are obtained by carrying out the reaction in presence of aliphatic alcohols, such as methyl or ethyl alcohol. Thus 500 pts. of a 30% formaldehyde solution, boiled under reflux for 10 hrs. with 500 pts. of methyl alcohol and 10 pts. of lead oxide, yield 140 pts. of syrupy sugars. Mixtures of polyhydric alcohols may be obtained from the synthetic sugar mixtures by catalytic reduction (e.g., with hydrogen in the presence of a platinum catalyst).

J. H. LANE.

Manufacture of sugar solutions suitable for fermentation purposes. J. Y. JOHNSON. FROM I. G. FARBERIND. A.-G. (B.P. 330,275, 25.2. and 22.3.29).—Peat is hydrolysed by acids and the separated solution treated, before or after neutralisation, with steam, or aerated while hot. [Stat. ref.] C. RANKEN.

Crystallisation (U.S.P. 1,749,588).—See I.

XVIII.—FERMENTATION INDUSTRIES.

Water for brewing. G. CAPRINO (Zymologica, 1930, 5, 47—55).—In carrying out the decarbonation of water for brewing, the nature of the raw materials used and of the beer to be produced must be considered. With brown beers, for which long-grown, well-kilned malt is used, a harder water is required than for pale beers. Substitutes such as rice give an alkaline reaction, softer water being hence required. For making beer of the Pilsen type, the liquor should be treated with lime water, this being followed, when necessary, by addition of sufficient hydrochloric or sulphuric acid to neutralise 1 or 2 degrees of hardness. The optimum p_H is 5.2, this corresponding with the most rapid and complete saccharification, the highest extract, and the greatest attenuation. Decarbonation of the brewing water necessitates considerable increase in the amount of hops used.

T. H. POPE.

Efficiency of alcoholic fermentation of sugar.

Y. TOMODA and H. KOCHI (J. Soc. Chem. Ind., Japan, 1930, 33, 75—76 B).—The relation between yeast reproduction and the efficiency of the alcoholic fermenta-

tion of sugar has been studied. The possibility of reducing rather than increasing the yield of alcohol by adding ammonium sulphate to the molasses mash is demonstrated, as more sugar is expended for yeast multiplication in the presence of the salt. The highest fermentation efficiency may be obtained by restricting the yeast multiplication during fermentation as far as possible: a high pitching rate of yeast usually increases the efficiency on account of the reduced reproduction.

E. LEWKOWITSCH.

Production of glycerin by fermentation. IX. Separation of glycerin from fermented waste molasses. Y. TOMODA (J. Soc. Chem. Ind., Japan, 1929, 32, 271—272 B).—The residue from the fermentation of molasses is evaporated to a thick paste from which the glycerin is extracted with 95% alcohol. In this way 70% of the glycerin is recovered. A. A. GOLDBERG.

[Detection of fruit wines in grape wines by means of the] conversion of dibenzylidenesorbitol to sorbitol hexa-acetate. H. JAHR (Z. Unters. Lebensm., 1930, 59, 285—288).—Werder's method (cf. B., 1929, 619) for the detection of cider in wine depends on the isolation of sorbitol as its dibenzylidene derivative. This alcohol is said only to occur in cider. As the hexa-acetyl compound is better crystallised and has a sharp m.p., it is preferred as a means of identification. The benzylidene derivative is decomposed by heating on a water-bath with hydrochloric acid in the presence of benzaldehyde. The benzaldehyde is extracted with ether and the acid removed with zinc oxide. The neutralised solution is then heated with acetic anhydride and zinc chloride. On taking up in water and keeping, the hexa-acetate crystallises out, seeding being sometimes necessary. Sorbitol hexa-acetate has m.p. 98—99°; mannitol hexa-acetate has m.p. 120°. B. W. TOWN.

Removal of cream of tartar from grape juice by freezing storage. M. A. JOSLYN and D. A. TUCKER (Ind. Eng. Chem., 1930, 22, 614—615).—Separation of excess cream of tartar from fresh grape juice by freezing at —18° and subsequent thawing is much more rapid than, and as thorough as, storage at 0°, but in the case of juice previously stored at room temperature and consequently containing less cream of tartar the small crystals produced by freezing redissolve to a considerable extent during thawing. W. J. BOYD.

Evaluation of potable spirits, especially "Kirschwasser" and rum, by determination of the higher oxidation potential by means of chloramine-T. A. MIERMEISTER (Z. Unters. Lebensm., 1930, 59, 289—300).—A method is described for the evaluation of the aromatic constituents to which the bouquet is due. A standard quantity of the spirits is carefully distilled, using a double fraction-head, into seven fractions of equal volume, and each fraction is treated with chloramine-T and set aside. Excess chloramine is back-titrated iodometrically, the chloramine value of the fraction being expressed as the number of c.c. of 0.01N-chloramine-T used up. The sums of the values for the fractions give characteristic numbers which can be used to differentiate genuine rum from the artificial product. The behaviour of alcohol-water mixtures con-

taining artificial aroma constituents, such as benzaldehyde and bitter-almond water, has been investigated.

B. W. TOWN.

The extract question in the liqueur industry. C. LUCKOW (Z. Unters. Lebensm., 1930, 59, 301—304).—It is suggested that only such sweet spirits as contain at least 22 g. of extract per 100 c.c. should be considered as liqueurs.

B. W. TOWN.

Improvement of the quality of alcoholic liquids by treatment with charcoal. C. LUCKOW (Z. Spiritus-ind., 1930, 53, 174—175).—The undesirable flavours and aromas which brandy, rum, arrack, and other alcoholic liquids had acquired by contact with oil, rubber, inferior pitch, etc. were removed by treatment of the liquids with 0.1—1% of charcoal for periods varying from 1 to 6 hrs. If this treatment diminished the characteristic flavour of the liquid, the treated liquid was blended with a further quantity which had not been contaminated. Where the liquid had no characteristic aroma or flavour, the amount of charcoal was increased to 3% and the period extended to 2—3 days.

C. RANKEN.

Acido-proteolytic enzymes and tanning. GORINI.—See XV.

PATENTS.

Treatment of top-fermentation pale ale, mild ale, and stout after the primary fermentation for bottling and draught purposes. H. E. DRYDEN (B.P. 330,327, 19.3.29).—After the conclusion of the primary fermentation an extract of fresh hops is added to the chilled ale or stout, and the liquid filtered to remove primary yeast, proteins, resins, etc. A quantity of priming sugar solution is added and the cold, bright, fermented liquor pitched with a small amount of pure yeast sufficient to ensure the generation of carbon dioxide to produce suitable condition at the end of two weeks.

C. RANKEN.

Dehydration of impure ethyl alcohol. E. RICARD, Assr. to U.S. INDUSTRIAL ALCOHOL CO. (U.S.P. 1,763,722, 17.6.30. Appl., 20.11.25. Belg., 20.11.24).—See F.P. 614,913; B., 1927, 857.

Ketone fuels (F.P. 637,993).—See II. **Sugar solutions** (B.P. 330,275).—See XVII.

XIX.—FOODS.

Atypical corrosion of the starch of American winter barley attacked by *Fusarium roseum* and *Gibberella Saubinetii*. D. STRILCIUC (Z. Unters. Lebensm., 1930, 59, 258—265).—It is established that the starch of the attacked barley is corroded atypically. The manner of the corrosion can be distinguished from that in germinating grain and that produced by moulds. The reason for this atypical corrosion is the local ferment produced by the fungal hyphae. This atypical corrosion can be used for diagnosis of the fungus in groats and flour.

B. W. TOWN.

Colloid chemistry of gluten. II. H. L. B. DE JONG and W. J. KLAAR (Cereal Chem., 1930, 7, 222—244; cf. B., 1930, 31).—The effect of varying alcohol concentration on the viscosity of positive and

negative gliadin sols is examined. The relative viscosity increases up to a concentration of alcohol of 48% by wt., but in higher alcohol concentrations a rapid fall in these values occurs with both positive and negative gliadin. It is suggested that the maximum in the relative viscosity is caused by the formation of an alcohol hydrate layer around the particles, and this is gradually removed in higher alcohol concentrations, causing a fall in the viscosity. The effect of changing hydrogen-ion concentration is to cause separation on both sides of the point of maximum hydration of the aqueous sol, and this separation is a gradual process. Whether an emulsoid will flocculate, separate, or remain in solution by discharging is dependent only on the isoelectric hydration. B. W. TOWN.

Comparative study of the non-gluten constituents of soft and hard wheat flours. H. HALL (Cereal Chem., 1930, 7, 270—279).—In bread-baking the gluten is the "frame-work" of the loaf, but this does not apply in cake-making. A bread batch contains nearly three times the amount of dry gluten that a normal cake batch contains. In cakes the starches are more completely gelatinised than in bread, and the texture of cakes is intimately bound up with this gelatinisation. High viscosities of the gelatinised non-gluten constituents are associated with high swelling powers and high water-imbibing powers. Soft wheat flours contain a greater proportion of non-gluten constituents than hard wheat flours, and have a proportionately higher swelling power. B. W. TOWN.

Comparison between commercially and experimentally milled flours. T. A. PASCOE, R. A. GORTNER, and R. C. SHERWOOD (Cereal Chem., 1930, 7, 195—221).—The saccharogenic activity of experimentally milled flour does not represent the potential activity of the same flour milled commercially, for the commercial product was found to have an activity from one to four times that of the experimental product. This is due to the finer state of division of the commercial article, for the saccharogenic activity of a commercial flour was increased by 35% by grinding for 20 hrs. in a ball-mill. In the baking test the only significant difference was the consistently lower "absorption" of the experimental flours. There was found a linear relation between the total protein content and the loaf volume when the sample of wheat was milled from several different lots of wheat, but not apparently when milled from one sample of wheat. A lyotropic series was demonstrated in the peptising effect of potassium halide salts on flour proteins, potassium bromide showing the highest peptisation, with the least variability. B. W. TOWN.

Possibilities of standardising the granulation test for flour. J. MICKA and K. VRANA (Cereal Chem., 1930, 7, 280—306).—To promote standardisation, it is recommended (a) that the flours should be treated so as to have the same percentage (13.00—13.50%) of moisture; (b) that the method of continuous sifting should be used, one lot of 100 g. sufficing for the whole experiment; (c) to use 20 g. of wheat as a cleaner; (d) to perform the test under standard conditions of temperature and humidity (21°, and 70% R.H.);

(e) that the flour and wheat be at the same temperature as the room (21°); and (f) that the percentage of flour passing through the silks be recorded in a standard manner. B. W. TOWN.

Fat: its determination in wheaten products. C. W. HERD and A. J. AMOS (Cereal Chem., 1930, 7, 251—269).—It is suggested that direct ether extraction gives a measure of the free fat existing in the product, whilst acid hydrolysis gives the total fat content, i.e., free fat + combined fat after its liberation. The major portion of the lipid material is obtained unhydrolysed by the alkaline and alcoholic hydrolysis methods. B. W. TOWN.

Effect of acid potassium tartrate as an ingredient in angel cake. E. GREWE and A. M. CHILD (Cereal Chem., 1930, 7, 245—250).—The change in colour and grain of angel cake by using acid potassium tartrate is largely due to acidity. This ingredient gives a fine-grained, white cake, which otherwise is yellow and coarse-grained. The same effect is produced by using citric, malic, or tartaric acid in place of the tartrate. B. W. TOWN.

Hydrocyanic acid in Lima beans. II. Influence of heat on the toxicity of the beans. S. K. HAGEN (Z. Unters. Lebensm., 1930, 59, 211—216; cf. B., 1928, 689).—Long heating at 125° does not influence the toxicity, i.e., the glucosidic content, of the beans, although it destroys the enzyme present in the bean, which is very specific in its action. B. W. TOWN.

Effect of mineral oil treatment on the composition of milk. M. B. MACDONALD, E. C. ANDES, and F. A. BRIGGS (J. Home Econ., 1930, 22, 213—218).—On addition of mineral oil to remove "onion flavour" the butter fat absorbs less than 1% of oil, and less than 0.5% of butter fat is lost. There is no detectable loss of vitamin-A. CHEMICAL ABSTRACTS.

α - and β -Lactose in milk products. H. C. TROY and P. F. SHARP (J. Dairy Sci., 1930, 13, 140—157).—The rate of change of α - to β -lactose is minimal at p_H 2.0—7.0, but approaches infinity at p_H 0 and 9; the rate of attainment of final solubility or of precipitation of α -lactose is also affected by the p_H . High concentrations of sucrose, as in condensed milk or ice-cream, have no appreciable effect on the equilibrium rate. Skim milk prepared by pressure or centrifugal spray, or by atmospheric or vacuum roll showed a non-crystalline equilibrium mixture of α - and β -forms, whilst skim-milk flakes contained much crystallised α -hydrate. The caking of milk powder and the sandiness of ice-cream are discussed. In the determination of total solids in milk products the residue contains an equilibrium mixture of non-crystalline α - and β -lactose. CHEMICAL ABSTRACTS.

Manufacture of casein and lactose from skim milk. F. P. NABENHAUER (Ind. Eng. Chem., 1930, 22, 54—56).—Methods of manufacture of rennet casein, self-soured casein, artificially-soured casein, and lactose are described. Rennet casein is prepared by adding rennet to the milk and maintaining the mixture at 36° for 15—20 min. The curd is allowed to settle, whey drained off, the curd washed with water, pressed, and

dried. This type of casein requires fresh skim milk, thorough washing and pressing, and rapid drying at a low temperature. Self-soured casein is prepared by allowing spontaneous curdling at 38°. Soft curd forms, and is clumped by direct heating with steam jets. The curd is strained off, washed with cold water, pressed, shredded, dried on trays or in a tunnel drier, and then ground. "Muriatic" casein is prepared by adding sufficient sulphuric or hydrochloric acid just to curdle milk at 41–49°. Curd separates as tough lumps, which are drained off, heated with steam to form one large doughy lump, kneaded, cooled, cut into small chunks, dried, and ground. This gives a hard, flinty product which does not "wet" rapidly. The grain-curd method of producing casein is to add slowly, with stirring, dilute hydrochloric acid to give p_H 4.6–4.8. The curd, precipitated in fine grains, is drained off. The more acid is added the lower are the ash content of the resulting casein and the viscosity of its solution. This treatment is limited by the fact that a very acid curd is difficult to handle. Sulphuric acid yields the hardest curd and lactic acid the softest. An outline of the removal of the albumin from the whey, its concentration, and crystallisation of the lactose produced is also given.

E. B. HUGHES.

Solubility method of classifying acid caseins.

W. R. MUMMERY and F. BISHOP (Analyst, 1930, 55, 367–369).—Caseins may be classified by their solubility in borax. The casein (2 g.), ground if necessary to pass 30-mesh (opening 0.0223 in.) is placed in a $\frac{3}{4}$ -in. test-tube, 12 c.c. of borax solution (20.833 g. made up to 1 litre) are added, and the solution is frequently stirred while kept at 70° for $\frac{3}{4}$ hr. Incomplete solubility is then demonstrated if the casein is not fully dissolved. When dissolved, the solution is made up to 100 c.c., 1 c.c. of which with 10 c.c. of water are placed in another tube, to this are added 5 drops of bromothymol-blue indicator (0.1 g. macerated with 3.2 c.c. of 0.05N-sodium hydroxide and made up to 250 c.c.), and the colour is compared with that of the standard buffer solution (Clark and Lubs' formula; B., 1916, 980). The p_H of the solution after dilution (solubility index) indicates the solubility of the casein, which above 6.8 (blue) is termed very good; from 6.8 to 6.4 (green), good; and below 6.4 (yellow), passable. If the casein has not completely dissolved, the p_H is 6 or less, provided that the undissolved particles are casein. Determinations on 230 samples of lactic acid caseins showed that 5.2% had solubility indices of 6.5, 37.8% of 6.6; 41.7% of 6.7; 13.5% of 6.8; 1.3% of 6.9; and 0.5% of 7.0.

D. G. HEWER.

Substances causing heating in pears. E. BOTTINI (Annali Chim. Appl., 1930, 20, 181–187).—The so-called heating of pears may be reproduced artificially by storing the fruit in an atmosphere containing the vapour of ether, amyl or ethyl acetate, acetaldehyde, light petroleum, toluene, amyl or ethyl alcohol, chloroform, carbon tetrachloride, or carbon disulphide. These compounds kill the epidermal tissues and lead to the appearance of an enzymic action, which destructively oxidises aromatic compounds such as phenols and glucosides and causes browning of the vegetable tissues. Death of the epidermal tissues, accompanied by entry

of air, allows of the rapid diffusion of the heating throughout the interior of the fruit and of rapid invasion by moulds. The enzymic oxidising action is greatly hindered by the acidity of the fruit. Obviously the phenomenon cannot be combatted effectively by wrapping each pear in paper, as this maintains a stagnant atmosphere round the fruit. Adequate ventilation of the cold store and the use of a means of packing which favours rapid elimination of the ethereal metabolic products should reduce to a minimum losses due to this cause.

T. H. POPE.

Hydrogen-ion concentration of the juice of the pear during ripening. D. CORTESE (Annali Chim. Appl., 1930, 20, 197–204).—During the growth period of pears the p_H value for the juice falls gradually from 3.26 to the minimum 2.07, corresponding with the maximum size of the fruit. During the subsequent ripening period the value increases to about 4.5.

T. H. POPE.

F.p. of pears and apples. M. AGGRADI (Annali Chim. Appl., 1930, 20, 188–196).—The mean f.p., which represent the lowest temperatures to which fruit may be exposed without danger from alteration, vary from –1.4° to –3.3° for different varieties of pears and from –1.6° to –2.58° for different varieties of apples.

T. H. POPE.

Acidity and corrosion in canned fruit. E. F. KOHMAN and N. H. SANBORN (Ind. Eng. Chem., 1930, 22, 615–617; cf. B., 1928, 159).—Additional data are given to show that tin becomes less noble compared with iron as the p_H decreases, although the effect of such decrease varies according to the particular acid present. The greater tendency of enamelled cans containing fruit to perforate and to swell owing to development of hydrogen is due to the protective action of the large area of less noble tin on the small area of exposed iron in the plain can, whereas in the enamelled can the small area of exposed surface is rapidly detinned, thereby increasing the area of exposed iron and reducing its protection by the anodic tin. Addition of citric acid to sweet black cherries inhibits the tendency of these to perforate the can. The greater corrosive effect of dehydrated prunes over that of fresh prunes is similarly due to the dipping of the prunes into lye before dehydration in order to permit rapid evaporation through the skin, the p_H being thereby increased. Citric acid had a greater effect in rendering tin anodic to iron than a commercial grade of malic acid examined, although the range of p_H was the same in both cases. The presence of anthocyanin pigments increases corrosion in enamelled cans.

W. J. BOYD.

Microscopical diagnosis of marmalades. W. OETKER (Z. Unters. Lebensm., 1930, 59, 132–184).—After making an extensive anatomical and histological examination of the seeds and fruit of the species *Ananas*, *Ribes*, and *Vaccinium*, the author deals with the principles of determination and methods for the investigation of marmalades. It is considered that especial value should be attached to the detection of starch, large quantities of which were found in unripe fruit, in stone fruits, and in tomato, rhubarb, swedes, and carrots. Only apple marmalade contains large quantities of

starch that is of diagnostic value. But diagnosis merely on the presence of starch is not possible. The vascular bundles are of value in diagnosis. Coarse adulterants such as turnips are easily shown by their large vascular bundles, but the differentiation of fruits from one another is difficult. The absence of sclerenchymatous elements, as well as the kind and quantity of vessels present, supply important hints in diagnosis. The various fruits give fluorescent colours in ultra-violet light, but this is not yet of any value.

B. W. TOWN.

Simple polarimetric test for sugars in jams.

S. J. LEWIS (Analyst, 1930, 55, 384).—The test first introduced by Juckenack and Pasternack (Z. Unters. Nahr. Genussm., 1904, 8, 10), depending on the fact that the specific rotation of the inverted extract of all fruit juices is approx. -20° , is used for a rapid sorting test for jams. The jam (25 g.) is carefully stirred with 120 c.c. of water for 1 min. without breaking the fruit, strained, 2 g. of nitric acid are added, and, after keeping in boiling water for 30–35 min., the mixture is cooled and just sufficient (about 12 c.c.) of a concentrated solution of lead subacetate added; after shaking, leaving for 5 min., and filtering, the inverted sugar solution, lead-free and nearly citric acid-free, is polarised at 20° . The total solids are determined in 5 c.c. of solution, their acidity is found and calculated as citric acid, and this weight is deducted from the solids, giving the weight of "saccharine solids" (w). The sp. rotation is calculated from: $[\alpha] = 100\alpha/20 \cdot 1w$, i.e., $5\alpha/w$ for a 100-mm. tube and $2 \cdot 5\alpha/w$ for a 200-mm. tube. Results obtained for various jams are: raspberry, from $-20 \cdot 8^\circ$ to $-22 \cdot 9^\circ$; for strawberry, $-20 \cdot 5^\circ$, $-20 \cdot 8^\circ$; stoneless plum, $-22 \cdot 4^\circ$, $-21 \cdot 9^\circ$; strawberry with 10% of added glucose, $+1 \cdot 21^\circ$. D. G. HEWER.

Detection of dried plums in plum jam. G. KAPPELLER, G. PRANGE, and W. REIDEMEISTER (Z. Unters. Lebensm., 1930, 59, 191–197).—The method employed depends on the differing colours of the precipitate obtained with lead acetate, and also with acid. Jam made with dried plums sometimes has a low acid content. A microscopical examination also helps in the differentiation, as does the occasional presence of mites in the jam made with dried fruit.

B. W. TOWN.

Determination of the alkalinity of fruit produce by conductivity measurements. P. HIRSCH and K. RICHTER (Z. Unters. Lebensm., 1930, 59, 184–191).—From apple juice, cider, etc. values were found for the alkalinity that harmonised with those found by titration of the ash. The method is more rapid and the values are more sharply defined, and 4–5 determinations can be carried out per hour. The solution to be investigated is mixed with an equal volume of standard hydrochloric acid and the conductivity measured. The conductivity of the mixture is denoted by x_m , that of a 1:1 mixture of standard hydrochloric acid and water by x_a , and of a 1:1 mixture of solution and water by x_s . Then the decrease in the conductivity $dx = (x_a + x_s) - x_m$. The value for the alkalinity is then determined by reference to a curve showing the value of dx for known mixtures of the fruit acids and their sodium salts.

B. W. TOWN.

Caffeine content of natural and treated coffee. A. RINCK and E. KAEMPF (Z. Unters. Lebensm., 1930, 59, 281–285).—The caffeine from treated coffee does not differ in its solubility or physiological action from that from natural coffee.

B. W. TOWN.

Technical aids in the manufacture of foods.

Preservatives. A. BEHRE (Chem.-Ztg., 1930, 54, 325–327, 346–347).—The literature of food preservation by chemical methods is reviewed and discussed. A table of some 18 materials has been prepared showing compositions, proprietary names, applications, concentrations, physiological effects, and action on bacteria, moulds, etc. A further table gives the doses which produce symptoms of poisoning or death in dogs and puppies. Such materials as sugar, alcohol, oils, nitrates, nitrites, phosphates, acetates, and tartrates are considered unobjectionable, and are not included in the discussion. Mention is made of the difficulty of selecting for each and every food a material that is efficient but harmless. The use of 0.2% of benzoic acid in margarine and of sulphurous acid for preserved fruits appears to be satisfactory.

H. J. DOWDEN.

Detection and determination of dextrans in food materials. J. GROSSFELD and G. HOLLATZ (Z. Unters. Lebensm., 1930, 59, 216–236).—It was found that the colloids and colloidal starch could be precipitated from food materials by treatment with potassium ferrocyanide and zinc acetate without precipitating any of the dextrin. The dextrin was precipitated with alcohol, and it was found that considerably more dextrin was precipitated from a concentrated than from a dilute solution. The quantity of dextrin precipitated (x) is related to the quantity of syrupy starch (y) by the equation $y = ax^n$, where a and n are constants. The mean value of a was found to be 2.50, and of n was 2/3. A table is set out from these values from which x can be calculated.

B. W. TOWN.

Iodometry of food products. I. K. FUCHS, W. RUZICZKA, and E. KOHN (Z. Unters. Lebensm., 1930, 59, 266–281).—The material is treated with standard alcoholic solution of iodine, following the procedure of Margosches and co-workers (B., 1925, 639), and the excess of iodine titrated with thiosulphate. In the case of food products containing starch, the usual direct titration is difficult to carry out because the starch grains absorb iodine so strongly that the emulsion shows blue long after the end-point is reached. This difficulty is removed by adding excess of thiosulphate and back-titrating with standard iodine, when a sharp end-point is obtained.

B. W. TOWN.

Separation and determination of the solid fatty acids in food fats. J. GROSSFELD and A. SIMMER (Z. Unters. Lebensm., 1930, 59, 237–258).—A new procedure is given for the separation of the solid from the liquid fatty acids, depending on the crystallisation of the lead salts from dilute alcohol. The method gives a complete separation of oleic, linoleic, and linolenic acid. It was further found that hardened fats contained different isooleic acids, with different solubilities of their lead salts, of which one is probably n -elaidic acid. By adsorption on lead palmitate one can detect down to 2% of rape oil in olive oil. An addition of 5–10%

of rape oil was detected in linseed oil, peanut oil, and in sesamé oil by the raising of the iodine value of the solid fatty acids. B. W. TOWN.

Determination of minute amounts of lead and copper in food, beverages, etc. F. W. RICHARDSON (Analyst, 1930, 55, 323—325).—If the material yields very little ash 0.2 g. of calcium hydroxide is added as fixative. For liquids, 100 c.c. are evaporated in a silica dish, incinerated by an iron burner from the top and later also from below. The ash is heated almost to dryness with 2 c.c. of hydrochloric acid (d 1.16), then 1 g. of citric acid, 2 g. of ammonium acetate, and 5 c.c. of water are added, and, after boiling for $\frac{1}{2}$ min. and filtering, the filtrate is made slightly alkaline to alcoholic phenolphthalein with 10% ammonia, and then decolorised by acetic acid, allowing 0.03 c.c. excess of acid. Hydrogen sulphide solution is added and, after keeping warm for 1 hr., any sulphides and sulphur are collected on a small filter, washed, and the whole is transferred to a test tube to which are added 2 c.c. of a mixture of 50 c.c. of 10 vol. hydrogen peroxide, 10 c.c. of dilute sulphuric acid (1:1), and 60 c.c. of methylated spirit (64 O.P.). The tube is kept warm till the contents are bleached, and, after 1 hr., the filter paper is removed and washed first with the contents of the tube and then with 1 c.c. of peroxide mixture. After addition of 5 c.c. of water the hydrogen peroxide is expelled, the solution cooled, and the process continued as with the first filtrate (but leaving only 0.1 c.c. of excess acetic acid). The filtrate is made up to 10 c.c. and compared in miniature Nessler glasses with a standard containing 0.1 mg. Cu per c.c. Lead is determined in the contents of the small filter by pouring 2 c.c. of a 40% solution of ammonium acetate over the filter and washing with 3 c.c. hot water, whereby the usual procedure with phenolphthalein and ammonia solution gives a liquid with a small excess of acetic acid. The solution is made up to 10 c.c., hydrogen sulphide water added to 5 c.c., and a comparison made with a lead nitrate solution containing 0.1 mg. Pb per c.c. Modifications are necessary in treating materials leaving much phosphatic ash. D. G. HEWER.

Homogenisers. MÖLLERING.—See I. **Preparation of condensed milk and ice-cream.** PARISI.—See XVII. **Air-free water for food analysis.** FREELAND.—See XXIII.

PATENTS.

Preparation or substance for use in the manufacture of bread and such like. A. ARCHIBALD (B.P. 330,779, 22.6. and 26.7.29).—Cotton oil for use in bread baking is replaced by an emulsion of water, cottonseed oil, rice, saponin, and glycerin. The saponin-glycerin mixture may be replaced by an infusion of Irish moss. E. B. HUGHES.

Treatment of flour, meal, milling products, and the like. N.V. INTERNAT. OXYGENIUM MAATS. "NOVADEL" (B.P. 312,182, 21.5.29. Holl., 19.5.28).—Flour etc. is improved by treatment with small quantities of mono- or di-chloroamine or a mixture of the two. Air is passed through a mixture of sodium hypochlorite (or hypochlorous acid) and ammonia, thus becoming

charged with monochloroamine, and is brought into intimate contact with the substances to be treated. The use also of nitrogen trichloride or of per-compounds is not excluded. E. B. HUGHES.

Preparation of cereal food. W. D. and B. A. SMITH (B.P. 327,811, 14.2.29).—Wheat grains are cooked by heating under pressure with water, heating the product by steam and hot air, immersing it in vegetable oil heated at about 200°, and then centrifuging. E. B. HUGHES.

Butter-composition test. C. C. BOMBERGER (U.S.P. 1,748,668, 25.2.30. Appl., 14.4.27).—A rough method of determining moisture, fat, curd, and ash of butter by the application of heat only is described. The butter (10 g.) is weighed between the various heat-treatments which are calculated to remove (a) moisture; (b) fat by burning; (c) curd, by heating until nearly white, leaving the ash. J. H. BUSHILL.

Manufacture of cream and like fatty products. J. E. NYROP (B.P. 324,276, 5.3.29. Addn. to B.P. 297,256; B., 1928, 825. Cf. also B.P. 307,167 and 312,875; B., 1929, 376, 659).—The gaseous medium for spray-drying of cream etc. is first purified by being passed through material such as active charcoal, silica gel, chalk, etc. Instead of inert gas, air may be used as drying medium if deprived of its oxygen content by combustion and subsequent purification. E. B. HUGHES.

Production of artificial cream. S. E. HEMBERG (B.P. 330,307, 6.3.29).—Pasteurised milk freed from butter fat is mixed with melted vegetable or animal, vitamin-containing fats freed from free fatty acids, emulsified by whipping, and homogenised. H. ROYAL-DAWSON.

Treatment of creamery waste. J. T. TRAVERS, ASSR. to OHIO SANITARY ENG. CORP. (U.S.P. 1,747,802, 18.2.30. Appl., 2.8.27).—A process for clarifying effluent from creameries in which 1000 gals. are treated with 3—18 lb. of solid comprising roughly 80 pts. of magnesium sulphate, cement dust, or various waste products from alkali plants, 15 pts. of alkali, usually slaked lime, and 5 pts. of ferrous sulphate, aluminium sulphate, or calcium monophosphate. The proportion of the ingredients is varied to suit the particular requirements. The alkali neutralises any excess acid, making a slightly alkaline solution; the precipitant (e.g., ferrous sulphate) together with the electrolyte present then precipitates the protein, carrying with it the fat. J. H. BUSHILL.

Making cheese. L. A. THOMPSON (U.S.P. 1,745,962, 4.2.30. Appl., 21.10.27).—By maintaining the temperature of milk at 46.1—48.9° for 30 min. in the presence of 20% of malt wort, cooling to 90°, and adding rennet, a cheese may be produced of the Cheddar variety containing partially digested casein and possessing additional nutritional value. J. H. BUSHILL.

Egg products and their production. A. K. EPSTEIN (B.P. 327,701, 9.10.28).—If sucrose or other carbohydrate and common salt or other edible salt together with an edible acid (e.g., acetic) are added to liquid egg before freezing, various physical consistencies of the product after thawing can be obtained, depending

on the amounts and proportions of the above-mentioned ingredients that are added. E. B. HUGHES.

Treatment of cacao beans. G. DEFREN (U.S.P. 1,750,795, 18.3.20. Appl., 4.1.26).—The raw beans are soaked for 2–12 hrs. in water at 60° to extract most of the volatile constituents (acids) and bitter (non-volatile) constituents, thereby enhancing the true chocolate flavour, permitting more uniform roasting subsequently, and facilitating more complete removal of shell during winnowing. E. B. HUGHES.

Fermentation of cacao beans. Further preparing cacao beans after fermentation. B. MÜLLER (B.P. 305,236 and 329,015, [A] 2.2.29, [B] 8.2.29. Ger., [A] 2.2.28).—(A) Complete alcoholic fermentation of beans and pulp to prevent germination is obtained in 3 days by placing them, free from the pods, directly into transportable fermenting boxes having ventilating pipes; the necessity for turning the beans to prevent overheating and undesirable acetic fermentation is thus avoided. (B) The fermented beans, heated on trays in a closed space below 50°, swell, change in colour, and by oxidation lose their bitterness, and are partly dried by a current of air below 60°. The warm beans are finally dried without further heating, by continuous circulation of outside air through perforated pipes, thus avoiding destruction of enzymes. E. B. HUGHES.

Production of chocolate. H. BOLLMANN and B. REWALD (B.P. 330,450, 30.7.29. Ger., 8.7.29).—Deterioration of chocolate by heat, light, and air is obviated by the addition of 0.1–0.5% of animal or vegetable lecithin, previously dissolved in cacao butter at 60°.

E. B. HUGHES.

Sterilisation and preservation of fruit juices. W. DOERBECKER (B.P. 330,368, 29.4.29).—A preservative, *e.g.*, sodium benzoate solution, is added to fruit juices, and the mixture then subjected to the action of ultra-violet rays.

H. ROYAL-DAWSON.

Bleaching of foodstuffs. L. MELLERSH-JACKSON. From PILOT LAB., INC. (B.P. 327,670, 3.10.28).—The bleaching agent claimed consists of a higher fatty acid, peroxide, or a mixture of such peroxides, and is produced from a fat, such as coconut oil, by first forming the fatty acid chlorides and then peroxidising with hydrogen peroxide in alkaline solution. Suitable foodstuffs are those containing chromophoric oil-bearing materials (*e.g.*, flour, meals, egg yolk).

E. B. HUGHES.

Treating comestibles. ATLANTIC COAST FISHERIES Co., Assees. of H. F. TAYLOR (B.P. 311,317, 1.3.29. U.S., 9.5.28).—Before quick-freezing, flesh foods are immersed in a buffer solution to give p_H 7, thus preventing drip on thawing, and in common salt solution to prevent absorption of water from the buffer. Nitrite is also added to preserve colour. Suitable buffers are tri-sodium citrate or phosphate, sodium carbonate or hydroxide, or the corresponding potassium or ammonium derivatives.

E. B. HUGHES.

Treating comestibles with smoke. ATLANTIC COAST FISHERIES Co., Assees. of H. F. TAYLOR (B.P. 309,405 and 316,283, 1.3.29. U.S., [A] 9.4.28, [B] 27.7.28).

—(A) In the smoking of fish and meat, ammonia gas is mixed with the smoke to combine with aldehydes, neutralise acids, and favour colour development (oxidation of pyrogallol-like substances). (B) Apparatus is described for treating fish etc. in a closed room with smoke at about 30°, saturated with water vapour to prevent shrinkage. [Stat. ref.] E. B. HUGHES.

Treatment of comestibles with smoke. H. F. TAYLOR, Assr. to ATLANTIC COAST FISHERIES Co. (U.S.P. 1,760,091, 27.5.30. Appl., 9.4.28).—See B.P. 309,405; preceding.

Production of puffed, popped, or expanded cereal food products. KELLOGG Co., Assees. of E. H. MCKAY (B.P. 300,261, 8.10.28. U.S., 10.11.27).

Method and apparatus for continuous drying and roasting of cacao beans and the like. D. G. STEELY and F. W. BLAKE (B.P. 330,826, 14.8.29).

Smoke-producing apparatus for use in smoking comestibles. ATLANTIC COAST FISHERIES Co., Assees. of H. F. TAYLOR (B.P. 318,194, 1.3.29. U.S., 30.8.28).

Kneading machine (U.S.P. 1,750,558).—See I. **Acid calcium phosphate** (B.P. 330,777).—See VII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Rapid determination of phenolphthalein in aperients. G. ROSENBERGER (Chem.-Ztg., 1930, 54, 345).—The pastille types of aperient generally contain phenolphthalein as the active constituent (0.1–0.15 g. in each) compounded with sugar and suitably flavoured and coloured. Agar-agar jelly is frequently used as a base. The analysis of these products is based on the insolubility in water of phenolphthalein. A weighed pastille is boiled with 25 c.c. of water and, after cooling to 80°, the residue is collected and washed with about 75 c.c. of water at 75–80°. The residue is then extracted on the filter with boiling absolute alcohol and washed with 95% alcohol until the washings are free from phenolphthalein. The extract and united washings are evaporated to dryness and the dried residue is weighed. To compensate for the partial solubility in water of phenolphthalein a correction is applied to the weight by adding 0.0035 g. for each 100 c.c. of aqueous filtrate and washing water. When agar-agar is present in the material, the initial disintegration is effected by boiling with 50 c.c. of water containing 2 c.c. of hydrochloric acid (d 1.19), and, after cooling and filtering, the residue is washed until the washings are free from chloride. The correction to be applied is 0.0035 g. per 100 c.c. of water used, and is the mean of 50 determinations using 1.5–2.5 g. of phenolphthalein and about 100 c.c. of water at 80°.

H. J. DOWDEN.

Colorimetric determination of novocaine and anaesthesin with β -naphthol. W. EISSNER (Arch. Pharm., 1930, 268, 322–323).—To 0.05–1 c.c. of the approx. 0.2% solution to be analysed are added, successively at 0°, 0.1 c.c. of 2*N*-sulphuric acid, 0.5 c.c. of 1% sodium nitrate, 1.0 c.c. of 2*N*-sodium hydroxide, and 0.2 c.c. of 4% alkaline β -naphthol solution. After 1 hr. each at 0° and at the ordinary temperature, the red colour developed is compared with that of a solution

prepared from the pure drug, compensating, if necessary, for the yellow colour of the β -naphthol. The solutions obtained from novocaine obey Beer's law; those from anæsthesin do not. Cocaine, small amounts of adrenaline, and serum do not interfere. H. E. F. NOTTON.

Seventh Report of the Essential Oil Sub-Committee to the Standing Committee on Uniformity of Analytical Methods. Determination of solubilities [of essential oils] (Analyst, 1930, 55, 386; cf. B., 1930, 639).—It is recommended that tests should be carried out, unless otherwise stated, at 15.5°, and that the concentrations of alcohol used should be given as vol.-%. The terms solubility, opalescence, and turbidity as applied to essential oils are also defined.

D. G. HEWER.

Ambergris and how to recognise it. F. R. MORRISON (Bull. Tech. Mus., Sydney, 1929, No. 15, 9 pp.).—The different varieties of ambergris are described together with their values, uses, and characteristics. All ambergris will float on sea-water and, if the latter is slowly heated, the material will melt to a brown or black liquid before the water reaches its b.p. A piece of ambergris about the size of a pea is almost completely soluble in about $\frac{1}{2}$ oz. of warm alcohol, giving a fluorescent solution. A needle, previously heated in a candle-flame for 10–20 sec., on being pressed into a sample of ambergris to a depth of $\frac{1}{8}$ in., forms a dark brown resinous liquid, which, when touched by the finger, leaves pitch-like "strings" adhering to the skin. If the needle be withdrawn and placed in the flame, the ambergris burns; if the flame be extinguished the odour of the "smoke" is somewhat fatty or resinous and resembles that of burning rubber.

E. H. SHARPLES.

Enzymic processes in the preparation of perfumes from plants. A. ELLMER (Reichstoffind., 1929, 4, 105–109, 139–142, 160–162; Chem. Zentr., 1930, i, 758).—A discussion. A. A. ELDRIDGE.

Homogenisers. MÖLLERING.—See I. **Detection of ethyl o-phthalate.** SUCHODOLSKI.—See III. **Solubility of sulphur.** HENVILLE.—See VII.

PATENTS.

Production of anæsthetics. W. SCHOELLER and H. G. ALLARDT, Assrs. to SCHERING-KAHLBAUM A.-G. (U.S.P. 1,765,621, 24.6.30. Appl., 18.8.28. Ger., 6.8.27).—See B.P. 321,968; B., 1930, 348.

Manufacture of santalol compounds. H. WALTER, Assr. to VER. F. CHEM. IND. A.-G. (U.S.P. 1,761,376, 3.6.30. Appl., 3.1.28. Ger., 17.7.25).—See B.P. 278,982; B., 1928, 36.

Stable [medicinal acetyl]salicylate composition. M. COPLANS (U.S.P. 1,764,933, 17.6.30. Appl., 20.12.28. U.K., 12.1.28).—See B.P. 315,330; B., 1929, 835.

Basic oxime ethers of cyclic compounds. W. KROPP, Assr. to WINTROP CHEM. CO., INC. (U.S.P. 1,733,462, 29.10.29. Appl., 25.8.27. Ger., 23.9.26).—See B.P. 301,956; B., 1929, 149.

Apparatus for vaporising chemicals [e.g., medicaments] in rooms [for inhalation purposes].

"AIROSANA" TROCKENINHALATIONS-GES.M.B.H. (B.P. 311,733, 4.5.29. Ger., 15.5.28).

Processes and devices for [air-]drying of tobacco and similar materials. E. QUESTER (B.P. 330,778, 21.6.29).

Sanitary agent (B.P. 330,099).—See XXIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Spectral sensitisation of photographic emulsions. Bathing with pinacyanol-pinaflavol mixtures. B. H. CARROLL and D. HUBBARD (Bur. Stand. J. Res., 1930, 4, 693–701).—Pyridine increases the effectiveness of sensitisation and reduces the tendency to flocculation, which may be a serious difficulty when pinaflavol is mixed with other sensitising dyes. Optimum conditions are: bathing for 1 hr. at not above 10° in a vertical position with an aqueous bath containing, per litre, 4 mg. each of pinaflavol and pinacyanol and 10 g. of pyridine. Good results may also be obtained with some emulsions by bathing for 3 min., with agitation, in a bath containing, per litre, 10 mg. of each of the dyes and 10 g. of pyridine. The pyridine must be free from reducing impurities. W. E. DOWNEY.

Hypersensitising with peroxide and silver salts. U. SCHMIESCHEK (Brit. J. Phot., 1930, 77, 276–277).—Efficient hypersensitising baths for panchromatic materials may be prepared from a combination of a soluble silver salt with hydrogen peroxide, e.g., 1% solution of silver nitrate 4 c.c., 30% hydrogen peroxide 1 c.c., water 400 c.c. The action of such baths is almost independent of the pH , since good hypersensitisation can be obtained in the presence of either acetic acid or ammonia, the increase in sensitivity being slightly the greater with the acid bath. To test the efficiency of various silver salts and their effect on the keeping properties of the plate, baths were prepared with the nitrate, chloride, chromate, carbonate, phosphate, oxalate, arsenate, iodate, molybdate, and tungstate, according to the following formula (in which the silver is present as the ammonium complex salt): 25% ammonia solution 1 c.c., 30% hydrogen peroxide 1 c.c., water 400 c.c., and silver salt equivalent to 0.025 g. Ag. The nature of the acid radical had very little effect on the hypersensitisation, but the molybdate and tungstate showed a marked effect in preventing fog. Non-colour-sensitised and orthochromatic plates were scarcely affected by the silver tungstate bath, and the slow, fine-grain, panchromatic plates showed a greater percentage increase in sensitivity than the faster panchromatic plates. J. W. GLASSETT.

PATENTS.

Manufacture of silver halide emulsions. I. G. FARBENIND. A.-G. (B.P. 305,143, 31.1.29. Ger., 31.1.28. Addn. to B.P. 271,475; B., 1928, 69).—In place of the glyoxaline of the prior patent any other organic reagent capable of forming a stable silver salt of solubility not greater than 1–2 mg. per litre at 18°, e.g., thioglycolic acid, thiolacetic acid, tetrazole, 2:6-dithiol-4-keto-3:5-diphenylpenthiophen, and allylaminodithiazole hydrogen sulphide, may be used. C. HOLLINS.

Sensitised coating and method of making and applying same and photographic plate or film produced thereby. E. DE STUBNER (U.S.P. 1,752,069, 25.3.30. Appl., 14.3.27).—The danger of displacement or stripping of a sensitised film from a celluloid base is overcome by eliminating the special cementing agent by using as sensitised coating a soluble cellulose ester which forms a homogeneous union between the coating and film directly. Silver nitrate and potassium iodide solutions are mixed in a tank containing a soluble cellulose ester such as nitro- or acetyl-cellulose, very finely divided silver iodide thus being precipitated on the ester. The cellulose ester is washed free from potassium nitrate, dried with alcohol, and dissolved in a suitable solvent, forming a lacquer containing silver iodide in suspension. This is applied directly to the celluloid film.

J. W. SMITH.

[Resist for] photographic etching. A. B. DAVIS, Assr. to KEYSTONE WATCH CASE CORP. (U.S.P. 1,751,908, 25.3.30. Appl., 16.12.26).—A stable resist is prepared by the action of sulphur chloride dissolved in carbon disulphide on asphalt. The carbon disulphide is removed on a water-bath, and the residue dissolved in benzene, with addition of 2% of lavender oil as accelerator and sensitiser. This solution is applied to the metal plate and dried. Before and after exposure the film is dipped for 15 sec. in ether, which causes the film to become more adherent to the plate. Development is carried out with turpentine and petroleum benzine (3:1).

J. W. SMITH.

Photographic resist and photographic etching. A. B. DAVIS, Assr. to KEYSTONE WATCH CASE CORP. (U.S.P. 1,751,909, 25.3.30. Appl., 28.1.27).—The resist is prepared by dissolving 15 g. of shellac in a solution of 5 g. of borax in 100 c.c. of water; 5 c.c. of 28% ammonia solution and 15 c.c. of 20% ammonium dichromate solution are added. The metal plate is coated with this mixture, dried, exposed, and developed by washing in methylated spirit containing a little dye to facilitate judging the degree of development. After development the plate is dried and placed in alcoholic tannic acid (50 g. of tannic acid dissolved in a mixture of 50 c.c. each of water and methylated spirit), which renders the shellac insoluble after heating. The plate is dried and baked and finally electro-etched in the usual way.

J. W. SMITH.

Preparation of transparent pictures. J. HEIDENHAIN (B.P. 327,068, 18.6.29).—A transparent support is coated with a layer of lead sulphide by immersion in a suspension of the latter prepared by adding a 2% aqueous solution of potassium hydroxide to a mixture of 2% aqueous solutions of lead acetate and thiourea. This is then coated with a light-sensitive layer, e.g., sensitised albumin, which is then printed, developed, coated with asphaltum, and etched to give a negative. When used for screen negatives, the dots may be intensified or reduced by suitable chemical agents or mechanical means.

J. W. GLASSETT.

Production of photographs in natural colours. C. RUZICKA (B.P. 326,764 and 326,779—326,781, [A—D] 13.12.28).—The following methods are described for the

preparation of colour screens. (A) The particles comprising the screen are prepared by hardening a suitably dyed gelatin solution with formaldehyde, allowing this to set to a stiff jelly, and, after breaking up into small particles by pressure through a fine mesh, drying the particles very slowly with agitation. Equal quantities of red, yellow, and blue particles are intimately mixed and incorporated in a nitrocellulose solution which is applied in a thin layer to the support for the sensitive emulsion. (B) The screen is prepared from differently coloured particles of glass, transparent metallic or metalloidal compounds, transparent minerals, starch, or cellulose ester. Equal quantities of the particles are incorporated in a vehicle such as nitrocellulose solution and applied to the emulsion support. (C) A dyed gelatin solution is treated with formaldehyde and, before gelatinisation occurs, the gelatin is precipitated in the form of fine particles by the gradual addition of alcohol with rapid agitation. These particles are then further hardened and dried. Alternatively, a dyed solution of a resin or gum in a suitable solvent is subjected to an increase in pressure sufficient to precipitate the resin or gum in fine particles. Coloured particles from either of these methods are thoroughly mixed and strewn upon a thin sheet of nitrocellulose or cellulose acetate, the surface of which has been made tacky by treatment with a solvent. A similar sheet of material is then placed over the particles and the whole firmly joined under pressure. (D) Before the colour screen is applied, the support is coated with a highly reflecting metallic layer of silver or other suitable metal either by the metal-spraying process or by electrodeposition.

J. W. GLASSETT.

Preparation of pictures to be produced by tanning action. M. P. SCHMIDT, R. ZAHN, and W. KRIEGER, Assrs. to KALLE & Co. A.-G. (U.S.P. 1,762,033, 3.6.30. Appl., 7.8.28. Ger., 22.8.27).—See B.P. 296,008; B., 1930, 218.

[Combined taking screen and panchromatic plate for] colour photography. C. L. FINLAY, and FINLAY PHOTOGRAPHIC PROCESSES, LTD. (B.P. 330,508, 9.2.29).

Light filter (U.S.P. 1,751,220).—See I. Decolorising film (U.S.P. 1,743,155).—See VI.

XXII.—EXPLOSIVES; MATCHES.

Manufacture of nitrocellulose of very high nitrogen content for B.F.P. powder. DEMOUGIN (Mém. Poudres, 1928, 23, 262—267).—Investigation of the maximum degree of nitration of linters, consistent with stability of the product, indicates the necessity for using mixed acid containing about 22% HNO_3 and 10.3—10.5% H_2O ; the nitrocellulose produced yields approx. 213.5 c.c. NO . A series of curves is given which show the variation in nitrogen of the nitrocellulose for varying amounts of water and nitric acid in the nitration acids. These curves have the same form as those obtained by Lunge, but do not show a maximum nitrogen content at 13.8%, equivalent to 220 c.c. NO , found by him. For nitrocellulose for B.F.P. powder, the mixed acid should contain 21.0—21.5% HNO_3 and 15.6—15.7% H_2O , the yield of nitric oxide being then 199 c.c.;

it is not advisable to exceed this if a completely soluble nitrocellulose is required. In the stabilisation of nitrocellulose for B.F.P. powder, it is possible to increase the solubility by long boilings of slight acidity without detriment to the stability or the nitrogen content.

W. J. WRIGHT.

Manufacture and stabilisation of nitrocellulose. A. H. BRESSER (Ind. Chemist, 1930, 6, 249—251).—Disintegrated cellulose is weighed and nitrated with 50 pts. of mixed acid in either stoneware pots or cast-iron centrifuges with acid-resistant internal lining. The acid may be removed in the same centrifuge or in another one, but in the latter case the management of the connexions and cocks is troublesome and dangerous. A table gives the relation between the composition of the mixed acid and the resulting nitrocellulose. The temperature should not exceed 68°, but has no influence on the composition of the product. The nitrocellulose is washed several times with cold water and then boiled with two changes of hot water and live steam. It is then ground in beaters with addition of a little soda and agitated for 8 hrs. in iron stabilisers lined with stoneware. Bleaching is best effected with hypochlorite.

C. IRWIN.

Elimination of dextrose in the stabilisation of nitrocellulose. H. LÉCORCHÉ (Mém. Poudres, 1928, 23, 275—284).—In experiments to determine the elimination of sulphuric acid and dextrose during the stabilisation of nitrocellulose, the nitrocellulose was kierié at 130°, 8 hrs. being sufficient to ensure stability. On extending this time, increasing amounts of dextrose were found in the wash-waters, and after 24 hrs., decomposition of the nitrocellulose was complete and the amount of glucose was 25%. The origin of the dextrose and the effect of nitrates of sugar on the stability of the product are discussed, and two hypotheses are formulated: (A) the existence of sugars in the cellulose before nitration, (B) the existence of two phases, imbibition and nitration proper, in the nitration. It would appear that if sugars exist in the linters an appreciable amount of nitrated sugars is produced during nitration, depending on the amount of acid imbibed. In regard to the sulphuric acid that is eliminated during stabilisation, this probably existed originally as mixed sulphuric and nitric esters of cellulose, or even of sugars. The sulphuric impurities are more stable than those containing nitric acid.

W. J. WRIGHT.

Nitration of wood and other forms of cellulose. DEMOUGIN (Mém. Poudres, 1928, 23, 268—274).—Experiments were carried out by the Poudrerie d'Angoulême on the nitration of wood, alfa, and straw, the wood being in the form of wool, unsized paper, and wadding. In the Selwig plant, a product was obtained which yielded 207—210 c.c. NO, had a solubility of 20%, and was suitable for use in powder manufacture. The Thomson process caused difficulties with the wood-wool, and was quite inapplicable with the paper and wadding; the wood-wool was the most satisfactory form of cellulose. Notwithstanding its lower price as compared with linters, the cost of the nitrated product from wood was higher on account of the higher acid concentrations and the greater losses of acid. Nitration of alfa gave

a product intermediate in properties between that from cotton and from wood, but the tendency to form hard lumps renders the material unsuitable. Straw has only the advantage of availability, but has not been tested on an industrial scale.

W. J. WRIGHT.

Hygroscopicity of powder B and various nitroglycerin powders. M. MARQUEYROL (Mém. Poudres, 1928, 23, 300—303).—Three nitroglycerin powders containing, respectively, amyl phthalate, vaseline, and diphenylamine, and vaseline alone were tested for hygroscopicity in comparison with a thick B powder, BM₁₇, by exposure over sulphuric acid, and further tests were made to determine the effect of various periods of stoving. Results are tabulated. The nitroglycerin powders containing vaseline absorbed moisture less rapidly than the powder that contained amyl phthalate. The humidities of these powders in the condition of equilibrium were approximately of the same order of magnitude and much lower than that of BM₁₇.

W. J. WRIGHT.

Loss on heating of powder B and nitroglycerin powders. M. MARQUEYROL (Mém. Poudres, 1928, 23, 304—308).—BM₁₇ powder and three nitroglycerin powders, which had been preheated, were heated at 60° for prolonged periods and their loss in weight was determined at various times. BM₁₇ showed little loss and considerably less than the nitroglycerin powders; the curves for the latter coincided. The results give rise to the theory that in the period of rest between the two stovings the nitroglycerin diffused from the centre to the surface of the grains. Then, during the later heating, it evaporated from the surface. The nitroglycerin is therefore present in the form of a solid solution and not as a nitrocellulose complex.

W. J. WRIGHT.

Determination of residual solvent in B powders. DESMAROUX (Mém. Poudres, 1928, 23, 285—299).—The influence of the time of drying, the intensity of the immersion treatment, the content of soluble nitrocellulose, and the composition of the original solvent on the amount of residual has been investigated. No conclusion could be drawn regarding the effect of varying periods of drying, although generally the ether was retained more tenaciously. Examination of the effect of prolonged immersion in water at 80° showed the same irregularity; the alcohol chiefly was expelled. Increase in the thickness of the sample increased the amount of residual solvent. With increasing nitrocellulose content from 18 to 65% a regular increase in the residual alcohol and decrease in the residual ether were found; up to 98.8% nitrocellulose, however, the reverse took place. The composition of the original solvent has a direct influence on the amount of residual solvent. Analyses of various powders of various ages are given and show the persistence of the solvent.

W. J. WRIGHT.

Graphic method for determining the explosion temperature of SD powders. H. MURAOUR (Mém. Poudres, 1928, 23, 256—261).—Under certain conditions the determination of the explosion temperature from a measurement of pressure in experiments with the bomb gives results in agreement with those calculated from the heat evolved and the composition of the gases. The

latter method is preferable on account of its simplicity. Calculations of the explosion temperature of SD powders by Aunis show that it is a function of the nitric nitrogen content and the centralite content. By plotting the results, a graph has been constructed by means of which the explosion temperature may be calculated for any given nitric nitrogen or centralite content.

W. J. WRIGHT.

PATENTS.

Ammonium nitrate explosive and its manufacture. W. O. SNELLING and J. A. WYLER, Assrs. to TROJAN POWDER CO. (U.S.P. 1,749,613, 4.3.30. Appl., 14.10.22).—Ammonium nitrate is allowed to react with nitrated toluene, lead, and water, preferably at temperatures above 100°, *e.g.*, 115°. The reaction is hastened by adding an alcohol as well, and it is possible to use a lead salt in place of lead. The product may be used in conjunction with nitrostarch.

S. K. TWEEDY.

[Self]-combustible mixture and its preparation. J. THIECKE, Assr. to MINIMAX A.-G., and to DEUTS. PYROTECHNISCHE FABRIKEN A.-G. (U.S.P. 1,766,269, 24.6.30. Appl., 20.12.28. Ger., 25.10.27).—See B.P. 299,396; B., 1930, 488.

XXIII.—SANITATION; WATER PURIFICATION.

Removal and determination of nitrites in sewage effluents and waters. J. W. H. JOHNSON (Analyst, 1930, 55, 325—326).—The fact that nitrites can be easily and efficiently removed by boiling the slightly acidified liquid, and that, when distilled off completely, they can be collected and determined in the distillate is confirmed. The author attempts to explain the conflicting statement, appearing in "Methods of Chemical Analysis as Applied to Sewage and Sewage Effluents," published by the Ministry of Health, and taken from Report IV of the Royal Commission, Appendix V, 33—35, that "after reduction from 100 c.c. to 25 c.c.," apparently under similar conditions, "much nitrite remained," by suggesting that the boiled residues were repeatedly examined, in which case the loss observed would represent finality, half the original nitrite being lost during distillation. If a solution containing 0.001 g. or more of nitrous nitrogen is distilled, brown fumes appear which may be absorbed in sodium hydroxide, nitrites and nitrates both appearing in the distillate: $\text{N}_2\text{O}_4 + 2\text{NaOH} = \text{NaNO}_2 + \text{NaNO}_3 + \text{H}_2\text{O}$. In the case of the *iso*-form (*cf.* Thorpe, "Dictionary of Applied Chemistry," 1912, p. 688) the whole of the nitrites are present in the distillate: $\text{N}_2\text{O}_4 + 2\text{NaOH} = 2\text{NaNO}_2 + \text{H}_2\text{O} + \text{O}$.

D. G. HEWER.

Water purification [in the paper-mill]. J. W. BERRIMAN (Proc. Tech. Sect. Papermakers' Assoc., 1930, 10, 360—379).—Filtration by means of gravity and pressure filters is dealt with, and the plant described. The results of experiments on the treatment of water with aluminium sulphate are given. It is found that the optimum condition for the formation of filterable flocs lies between p_H 6.3 and 7.1, according to the turbidity of the water to be treated.

T. T. POTTS.

Air-free water for sulphur dioxide determinations in foods. D. M. FREELAND (Analyst, 1930, 55,

383).—The method of Magnus and Herd (B., 1930, 215) and of the Preservatives Determination Committee of the Chemists of the Manufacturing Confectioners' Alliance and of the Food Manufacturers' Federation (B., 1928, 346) both require the use of air-free water. A "Sparklet" siphon is filled with cooled de-aerated distilled water, a wash-bottle siphon inserted in the neck with the shorter limb at the level of the red line, and water displaced to this level by carbon dioxide. The wash-bottle fitting is removed, the components of the "Sparklet" are rapidly assembled, and the water is charged with carbon dioxide. Water stored thus yields results in sulphur dioxide determinations similar to those obtained with freshly de-aerated water.

D. G. HEWER.

Drinking waters for cattle. T. MCLACHLAN (Analyst, 1930, 55, 372—375).—Comparison of samples of water from five ponds used for years by healthy cattle with samples of fourteen other waters shows that it is not safe to condemn such waters for cattle-drinking on the results of chemical analysis unless a definite chemical poison is present, such as may be derived from sheep dip, minerals introduced from the neighbourhood, an excess of hydrogen sulphide, free chlorine, or possibly certain weeds. A recommended water should not show an oxygen absorption figure of more than 1 pt. per 100,000 in 3 hrs., but even a figure of 7.8 may be perfectly safe. A water should only be condemned on the results of bacteriological examination when bacteria pathogenic to cattle can be isolated. If contagious disease, which may be water-borne, is present in a herd, the water supply should be very good, and no surface water allowed to be drunk.

D. G. HEWER.

Water for brewing. CAPRINO.—See XVIII.

PATENTS.

Sanitary cleansing and deodorising agent. H. PICKUP and W. E. CLARINGBOLD (B.P. 330,099, 3.5.29).—Pine, cedarwood, or clove oil, or a mixture of two or more of these oils, is added to the material described in B.P. 318,344 (B., 1929, 912).

L. A. COLES.

Purification of water. J. MUCHKA (B.P. 316,965, 20.6.29. Austr., 7.8.28).—Filtering apparatus is described in which chlorinated water is freed from suspended organic matter, iron, and manganese before passing forward to the dechlorinator.

C. JEPSON.

Softening of water by base exchange. K. MORAWE (G.P. 460,743, 1.11.24).—The water is filtered through disintegrated lignite which has been treated with solutions of alkali silicate and acid salt solutions or dilute acids. The softening action of spent lignite may be regenerated by means of an alkali salt (sodium chloride) solution.

S. K. TWEEDY.

Disinfecting, bactericidal, insecticidal, fungicidal, and vermin-destroying preparation. R. LIESKE and W. SCHEPSS, Assrs. to WINTHROP CHEM. CO., INC. (U.S.P. 1,766,441, 24.6.30. Appl., 14.2.25. Ger., 10.4.24).—See B.P. 232,249; B., 1925, 940.

Air filter (U.S.P. 1,751,999).—See I. **Preparations for respirators** (B.P. 305,101).—See VII. **Creamery waste** (U.S.P. 1,747,802).—See XIX.