# BRITISH CHEMICAL ABSTRACTS

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# I. - GENERAL; PLANT; MACHINERY.

Works' control and laboratory equipment. A. SCHRODER (Ind. Eng. Chem., 1929, 21, 481-485).-The considerable reduction in the number of types of physical and chemical apparatus and the more rigid standardisation of the surviving types is discussed and illustrated in the fields of colorimetry, pyrometry, gas analysis, laboratory glassware, porcelain, and chemicals.

Automatic control in chemical industries. I. GINSBERG (Ind. Eng. Chem., 1929, 21, 410-414).-Automatic pressure and temperature controllers may be self-operated, in which case they are not very precise and only available through a limited range, or worked by compressed air. These can be made to control temperature to 1° and the best are mercury-actuated. Automatic temperature controllers are successfully used on alcohol or benzol stills, oil-cracking units, nitration kettles, dryers, etc., and in the rubber, paper, and textile industries. C. IRWIN.

Conversion of batch into continuous processes. J. V. N. DORR (Ind. Eng. Chem., 1929, 21, 465-471).-The development of the Dorr classifier has afforded a continuous process applicable to the separation of sands and slimes in oil treatment and to the size regulation of pigments. It is also used for the removal of grit from sewage. The Dorr thickener performs continuous countercurrent decantation of slimes, and has also been applied to the manufacture of phosphoric acid, aluminium sulphate, caustic soda, and lithopone. A similar device, the "clarifier," is used for the sedimentation of water and of sewage. The Dorr agitator, combining a revolving mechanism with central air agitation, has made continuous agitation processes C. IRWIN. possible.

Mechanical handling of materials in and about the chemical plant. I. A. K. BURDITT and W. F. SCHAPHORST (Ind. Eng. Chem., 1929, 21, 489-493).-An illustrated account of gravity, belt, screw, and pneu-C. A. KING. matic conveyors.

Progress of industrial heating by oil circulation. A. B. MCKECHNIE (Ind. Eng. Chem., 1929, 21, 496-498). -The heating and circulating of oil at high temperatures (316°) for industrial heating requires plant of careful design and robust construction, properly designed for expansional stresses. A rapid circulation of oil should be maintained to prevent local overheating and carbonisation, and the physical properties of the oil are important, particularly the cold viscosity, the proportion of lighter oils, and the quantity of carbon after distillation to dryness. An oil known as "Meprolene " is recommended for this purpose. C, A, KING.

Calcination. W. S. DICKIE (Ind. Eng. Chem., 1929, 21, 461-464).—The rotary kiln was developed in the cement and lime industries, but has more recently been applied to the undermentioned processes : the reduction of sulphur content of some iron ores, the dehydration of bauxite, the preparation of barium sulphide and lithopone, the revivification of bone black and barium silicate in sugar refining, the calcination of lime sludge and of gypsum, and the dehydration of fuller's earth. C. IRWIN.

Renaissance of the absorption refrigeration cycle. F. G. KEYES (Ind. Eng. Chem., 1929, 21, 477-480).-The typical refrigerating machine functions on the general principle embodying the difference in solubility of gases in cold and hot absorbing liquids of low vapour pressure, the cooling medium usually being water. Increased efficiency has been attempted by the use of a solid salt, e.g., ammonium nitrate or thiocyanate, which is soluble in ammonia liquor, and recent development in adsorption materials has led to machines in which charcoal or silica gel is used as the adsorbent, or even compounds capable of forming associated compounds, e.g., BaCl<sub>2</sub>,8NH<sub>3</sub>, with ammonia. Accelerated evaporation has been induced in a constant-pressure system by circulating air over the liquid ammonia, the same result being attempted without mechanical pumping by the introduction of hydrogen, which has a small density as compared with ammonia. C. A. KING.

Mechanical dispersion by means of the colloid mill. P. M. TRAVIS (Ind. Eng. Chem., 1929, 21, 421 425).-The colloid mill, as distinct from pressure homogenisers, is defined as obtaining dispersion by hydraulic shearing forces. Colloid mills may be of beater type, as the original Plauson mill, or may depend on shearing forces only between either smooth surfaces, or rough surfaces (when turbulence is added), or a combination of the two. It is probable that colloid mills do not actually disintegrate solid particles, but deflocculate aggregates, as in the preparation of paints. In the preparation of emulsions the control of  $p_{\rm H}$  values and temperature and the use of protective colloids are very important. Colloid mills should usually be operated with a clearance of 0.006-0.008 in., and no individual particle of greater diameter should be fed to them.

C. IRWIN.

Crushing and pulverisation. L. T. WORK (Ind. Eng. Chem., 1929, 21, 498-502) .- The overall energy of crushing quartz is proportional to the speed of crushing and probably to the new surface developed, though it is much greater than is theoretically necessary. Fundamental resistance to pulverisation of any material is important, but an unmeasured quantity, and practice

sida are adjustable.

C. A. KING.

has formulated an empirical classification into direct pressure mills, roller mills, tube mills, beater mills, and revolving-plate mills. New developments are in the direction of more wear-resistant materials, simplification of design, and new applications for closed-circuit grinding. C. A. KING.

Present trends in dust recovery. E. P. PARTRIDGE (Ind. Eng. Chem., 1929, 21, 446—451).—A summary is given of the comparative fields of usefulness of cyclone dust separators, Cottrell precipitators, and vacuum airfilters. It is suggested that air-filters may be applied to the cleaning of chimney gases previous to the use of these to minimise explosion risks in grinding as in powdering coal. C. IRWIN.

Trend of filtration. A. WRIGHT (Ind. Eng. Chem., 1929, 21, 493—495).—Progress in construction of filter presses has developed chiefly in the direction of increased filter area per unit of floor space. The present trend is to introduce automatic filters to ensure continuity and uniformity of product with minimum attention. The plant must be designed to suit the objective of the particular process, the complete discharge of the cake without impedance to filter porosity being fundamental to every successful filter. C. A. KING.

Evaporation. P. DE WOLF (Ind. Eng. Chem., 1929, 21, 451-456).—A discussion of the principles of the design of vacuum evaporators. Recent improvements have been mainly in the direction of more rapid circulation. Nickel tubes and tube sheets are increasingly used in the evaporation of caustic soda and of milk.

C. IRWIN.

Apparatus for technical gas analysis. O. BURK-HARDT, A. FISCHER, and F. FRANK (Gas- u. Wasserfach, 1929, 72, 504—505).—A modified type of Orsat apparatus is described. J. S. CARTER.

Errors and illusions in comparison of colours. R. TOUSSAINT (Chim. et Ind., 1929, 21, 924—930).— The difficulties in comparing colours which arise from the different effects produced in light from different sources, and from the imperfections of the eye itself, are discussed. These may be overcome by using the photo-electric colorimeter for the analysis of composite colours. W. J. BOYD.

See also A., June, 642, Ultrafiltration, dialysis, and osmometry (Sigaud). 673, Fractionating columns (MIDGLEY).

#### PATENTS.

Brine evaporation. [Tube] evaporators. Condensers, coolers, or like apparatus having tubular heat-exchanging surfaces. D. A. QUIGGIN (B.P. 309,104-6, 2.1.28).-(A) The brine discharge and blow-down valves are so arranged and screwed plugs so provided that a cleaning rod may be inserted into the brine-discharge pipe without disturbing the valves. (B) The joints between steam-conveying members are constructed with a conical male part and curvilinear female part. (c) In a tubular evaporator in which one tube plate is smaller than the other so that the whole bundle of tubes can be withdrawn through the shell, means are described for making the (detachable) joints between the shell, tube plates, and headers for inner fluid. B. M. VENABLES.

Dryers for various materials. R. DE REYERET (B.P. 293,765, 10.7.28. Belg., 11.7.27).-Fire gases pass from a hearth through a first longitudinal flue of refractory material, then up to a second longitudinal flue of cast-iron plates above the first, then sideways (at the hearth end) to a pair of third longitudinal flues constructed between sheet-iron plates and the outer "stone" wall of the dryer. The material is distributed over the whole length of the furnace and slides over each side of the cast-iron and brick flues ; between them and the sheet-iron flues the iron plates are suitably inclined and the material is drawn out laterally below the third flues. An air flue may be formed between the brick and cast-iron flues; the air heated in this is allowed to permeate the material from under a hood formed over the top of the cast-iron flue. B. M. VENABLES.

Spray drying. W. S. BOWEN (U.S.P. 1,711,306, 30.4.29. Appl., 1.7.27).—The heated gas is admitted to a circumferential passage round the top of the desiccating chamber and descends between radial guides that extend from the circumference part way towards the axis; the liquid is sprayed at the top of the central passage thus left. B. M. VENABLES.

Antifreezing composition. A. H. OSTERLUND (U.S.P. 1,711,324, 30.4.29. Appl., 24.8.25).—A mixture of glycerin with boiling sodium glycerophosphate is cooled, and a clarifying agent added, the latter being finally drawn off with the settled impurities. The mixture is adjusted to freeze below —37°.

B. M. VENABLES.

Grinding mill. D. COLE (U.S.P. 1,711,405, 30.4.29. Appl., 27.7.28).—A form of self-alining roller bearing for rotary drums is described. B. M. VENABLES.

Tubular grinding mills. F. KRUPP GRUSONWERK A.-G. (B.P. 288,259, 13.3.28. Ger., 7.4.27).—A tube mill is provided with a transverse chamber at one end for the collection and discharge, by known methods, of fine material; immediately preceding the transverse chamber is a screening chamber arranged after the manner of a Krupp ball mill, except that the perforated, stepped grinding plates may be inclined to the axis, and that the material passing the screen enters an annular collecting chamber, rotating with the mill, whence it passes to the transverse chamber above mentioned.

B. M. VENABLES. Grinding machine. L. RUPRECHT and A. G. KOLLSTEDE (U.S.P. 1,711,464, 30.4.29. Appl., 10.2.27). —Beaters rotate in a casing in such a manner that they strike upwards the material which is fed in a compact mass by a substantially horizontal worm conveyor.

B. M. VENABLES.

Roller mills for grinding. H. E. Cox and J. R. TORRANCE (B.P. 310,769, 27.1. and 26.10.28).—A mill with 4 rollers and 3 nips is arranged so that the total material passes downwards between an upper pair of slow rolls, then divides and passes outwards between the lower fast rolls and the upper rolls. The lower rolls may run from 6 to 30 times as fast as the upper. The upper rolls may rotate at equal speeds, or, better, one or both may have a continually varying speed so that the relative motion is continually changing. All the nips are adjustable. B. M. VENABLES.

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Crusher roll construction. G. W. JOHNSON. From AMER. ENGINEERING CO. (B.P. 311,534, 23.4.28).— A toothed roll is formed round a prismatic shaft upon the flat sides of which rest the bases of the teeth. The teeth are shouldered and are a loose fit in holes in sleeves which embrace the shaft and teeth ; keys of special form are provided to tighten the teeth in their holes.

B. M. VENABLES.

Pulverising mill. R. S. RILEY, ASST. to SANFORD RILEY STOKER Co. (U.S.P. 1,711,063, 30.4.29. Appl., 3.1.24).—A bull ring and co-acting roll hammers are both driven positively in opposite directions.

B. M. VENABLES.

Pulverising apparatus. F. H. DANIELS, ASST. to RILEY STOKER CORP. (U.S.P. 1,711,044, 30.4.29. Appl., 12.4.27).—Two pulverising zones are provided within one disintegrator casing; the first reduction of the material is effected by hammers and the second by beaters, the space outside the hammer path forming direct peripheral communication with the beater zone. B. M. VENABLES.

Mixing and grinding device. J. A. MICHAL, Assr. to TURBINATOR Co., INC. (U.S.P. 1,711,154, 30.4.29. Appl., 30.12.26).—A rotor having a diameter substantially greater than its axial length is provided with teeth or blades on the faces which are substantially transverse to the axis. The teeth intercalate with others on a fixed casing. The feed for material is near the axis and outlet at the periphery. B. M. VENABLES.

Machines for kneading, crushing, mixing, etc. H. G. TORULF (B.P. 310,791, 30.1.28).—The machine comprises a drum rotated by sleeve shafts, or trunnions, surrounding a solid shaft which is cranked within the drum; the crank pin carries a pair of links between the free ends of which a kneading roller is journalled. The sleeve trunnions and solid shaft are rotated by power at different speeds, preferably in opposite directions, but the rotation of the roller about its own axis is effected by contact with the material in the interior of the drum. The crankshaft also carries a scraper to discharge the material through a door in the drum when the latter only is held stationary. B. M. VENABLES.

Mixing machines. A. E. WHITE. From HOBART MANUF. Co. (B.P. 311,099, 10.5.29).—A mixing machine having a beater attached to a [vertical] shaft is provided with a conical sleeve at the junction of the beater and its shaft to arrest material creeping upwards. An upwardly inclined extension of the beater arm may also be provided to exert a downward thrust on the material to prevent it rising. B. M. VENABLES.

Apparatus for use in separating, sorting, and/or grading materials. E. M. SAVAGE (B.P. 311,310, 9.2.28).—A tiltable shaking table suitable for the separation of rounded from angular pebbles (*e.g.*, diamonds from rock) is described. B. M. VENABLES.

Centrifugal machine. L. D. JONES, ASST. to SHAR-PLES SPECIALTY CO. (U.S.P. 1,711,468, 30.4.29. Appl., 9.4.26).—A centrifuge which is used to separate concentrated emulsion and pure continuous phase is provided with means to keep constant the ratio of the two products discharged, at any speed or output.

B. M. VENABLES,

Centrifugal extractor. T. A. BRYSON, Assr. to TOL-HURST MACHINE WORKS, INC. (U.S.P. 1,710,447, 23.4.29. Appl., 25.10.21).—A centrifugal separator comprising an imperforate bowl in which the heavier constituent is retained is divided into a number of annular chambers by baffles extending inwards from the wall of the bowl to a greater extent than the overflow rim for separated lighter liquid. The radial flow is practically nil, and the axial flow to the outlet is made very slow compared with the circumferential flow by providing transfer ports in the baffles at points which are far removed circumferentially from their neighbours. If desired, before final outlet the lighter liquid may pass inwardly through a cylindrical screen within the bowl.

B. M. VENABLES.

[Centrifugal] separation of impurities from liquids. C. H. HAPGOOD, Assr. to DE LAVAL SEPARATOR Co. (U.S.P. 1,711,315, 30.4.29. Appl., 24.8.21).—A viscid substance is separated centrifugally, then heated by a medium not in contact with it to render it sufficiently fluid to flow away. B. M. VENABLES.

Separators for treatment of mixtures of mutually insoluble liquids. H. J. HOLFORD, and HARVEY HOL-FORD SEPARATORS, LTD. (B.P. 310,996, 4.2.28).—The mixture is admitted to the lower part of a vessel, and the light liquor travels upwards without much obstruction, the top level being held constant by a ball valve controlling the outlet for light liquid. The heavy liquid passes downwards through slots in an inclined baffle, then upwards in a sub-chamber charged with packing material, the outlet being in the upper part of the subchamber, but not so high as that for the lighter liquid.

B. M. VENABLES.

Rotary filters. BRIT. ROTARY FILTER CO., LTD., and A. G. E. JOYCE (B.P. 311,359, 10.2.28).—A construction of the cells and filtrate discharge pipes within the drum of a rotary filter is described; its object is to reduce the wetted surface and amount of undischarged liquor to a minimum. B. M. VENABLES.

Effecting an intimate contact between two fluids. C. H. FOOTT (B.P. 311,494, 8.3.28).—The fluids may be either a liquid and a gas or two liquids of different sp. gr. One fluid (for convenience assumed the lighter) is introduced in the form of drops or bubbles into the lower part of a comparatively narrow column of the heavier or carrier fluid, the bubbles or drops filling the whole cross-section of the tube and causing a rising broken column of mixed fluids. The actual introduction of the lighter fluid is through a fine jet situated in an enlarged space at the bottom of the column, and the separation of the two fluids is effected in an enlargement at the top, the carrier fluid returning to the bottom through a separate conduit forming a closed circuit; no other means of circulation is provided, and the circulation may be retarded to give any desired time of contact by an adjustable obstruction, such as a tap, in the return conduit. If the carrier fluid has the lower sp. gr. the apparatus must be arranged for reversed circulation. B. M. VENABLES.

Apparatus for treating gases with liquids. CHEM. ENGINEERING & WILTON'S PATENT FURNACE CO., LTD., and N. and T. O. WILTON (B.P. 311,249, 8.11.27).— The gas passes downwards through a number of chambers in succession, and liquid is sprayed into the top of each chamber and passes together with the gas over fixed contact surfaces comprising a number of loose vertical tubes inside which are twisted strips or like helical elements. The liquid is circulated from the bottom of each chamber to the spray of the same chamber, and there is a slower flow from chamber to chamber; this flow is arranged to be countercurrent to the gas flow. B. M. VENABLES.

Method of reciprocal action between gases and finely subdivided materials. METALLGES. A.-G. (B.P. 285,038, 9.2.28. Ger., 9.2.27).—The gas to be treated with powder is caused to flow upwards through a vertical chamber, which preferably expands upwardly, at such a rate that the powder is held in suspension. When the powder is spent the gas current is increased and the powder carried over to a separate collector. If desired, the gas current may be pulsating with intermittent removal of the powder. Two towers may be used alternately, one being used for the reaction while the other is being used for discharge. B. M. VENABLES.

Simultaneous separation and thermic treatment of mixtures of fluids and granular or other substances. P. SCHMIDT (B.P. 285,046, 9.2.28. Ger., 9.2.27).—The mixture is caused to move between surfaces which are maintained at different temperatures and have relative parallel motion. One form of apparatus suitable for drying of dust in dust-laden air comprises an unheated disc rotating above an annular surface heated from below. The mixture is fed upwards through the centre of the heated surface and is discharged at the periphery; the dust then falls downwards and the air passes out upwards. In an apparatus suitable for extracting crystals from hot saturated solutions the rotating disc is cooled. B. M. VENABLES.

Separating gases and liquids under pressure. C. F. R. HARRISON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 310,985, 31.12.27).—The mixed fluid is admitted to a floating chamber within a pressureresisting vessel of about the same shape; the floating chamber is supported by a spring or equivalent means and operates a sleeve valve through which liquid can leave through the lower part of the floating chamber and of the outer pressure-resisting wall. The gas leaves from the upper part of the vessel. The level of the liquid being automatically held constant by the floating chamber, the outflow of gas may be controlled by hand or other means, and will consequently control the inflow of mixture. B. M. VENABLES.

Separation of gaseous and liquid products [under pressure]. C. F. R. HARRISON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 311,193, 31.12.27).— The products from a high-pressure reaction (e.g., the destructive hydrogenation of carbonaceous material) are transferred (without substantial reduction of pressure) to a catch pot which is maintained at a temperature high enough to prevent condensation of vapours. The liquid only is drawn off through a cooler (which may be a heat exchanger) and valve or engine such as a Pelton wheel. The gas and vapour pass through a separate cooler or heat exchanger to a second (cold) catch pot

where the liquefied vapour is separated from the permanent gas, the pressure energy of each product being recovered in an engine if desired. B. M. VENABLES.

Separation of mixtures of liquids, or of liquids and solids, or of liquids and gases. Soc. ANON. DES CHARBONS ACTIFS E. URBAIN (B.P. 283,192, 1.11.27. Fr., 8.1.27).-A capillary siphon is used to transfer the liquid only from one vessel to another. The capillary spaces may be larger than the particles of solids to be separated provided that the capillary material is so chosen as to have an electric charge of the same sign as the phase to be left in the first vessel; this charge may be developed by washing the capillary material with compounds that are used in the mordanting of webs. In some cases the material should be cleaned with detergents, and in all cases it is advisable previously to wet the material with the liquid that is to be transferred. B. M. VENABLES.

Removal of dust from air, gas, and other elastic fluids. C. H. SCHOL (B.P. 311,639, 29.10.28).—A number of inclined baffles are placed alternately along either side of a long chimney, causing repeated slight compression with subsequent eddying of the stream of gas. The dust is thrown out of the eddies and falls down channels left between the baffles and the wall of the chimney, or through separate passages leading from different vertical stages of the chimney.

B. M. VENABLES.

Conditioning of air for ventilation. HEENAN & FROUDE, LTD., and G. H. WALKER (B.P. 311,304, 8.2.28).-In cases, such as mines, where a supply of cooling water is available rather below the temperature of the untreated air which contains considerable moisture, the cooling water is passed through the annular spaces of a heat interchanger constructed of twin concentric tubes; an absorbing liquid such as calcium chloride solution is passed through the inner tubes and afterwards sprayed over the outside of the outer tubes (which may be gilled) in company with the air to be conditioned. The air is thus dried and its heat content reduced, while the consequent rise of temperature of the fluid is prevented. If desired, the device for effecting contact between liquid and air may be separate from the cooler, and, after drying, the air may be again humidified. The absorbing liquid is kept in circulation and part or all is subjected to a concentrating process and returned to the circuit. B. M. VENABLES.

Fluid-storing material. F. G. KEYES, Assr. to NAT. REFRIGERATING CO. (U.S.P. 1,705,482-4, 19.3.29. Appl., [A, B] 6.11.23, [C] 15.1.25).—A hard porous mass for storing gases and liquids comprises a mixture of (A) zinc oxychloride, calcium chloride, and sodium silicate, (B) magnesium oxychloride, calcium chloride, and active charcoal, and (C) a halide capable of forming additive compounds with fluids, an oxychloride cement, and alundum cement. A. R. POWELL.

Conveying and consuming without loss liquid gases such as liquid air, liquid oxygen, liquid nitrogen, etc., boiling at low temperatures. GES. F. INDUSTRIEGASVERWERTUNG M.B.H. (B.P. 280,569, 10.11.27. Ger., 13.11.26).—The storage or transport vessel has no communication with the atmosphere, but communicates with a receiver into which any gas evaporated from the liquid passes and causes a progressive rise of pressure, which, in turn, stifles evaporation. The capacity of the receiver is calculated to contain safely all the vapour produced in the longest period between successive intentional withdrawals of gas for use. B. M. VENABLES.

Means for ascertaining the percentage composition of substances after a given dilution and vice versa. NESTLÉ & ANGLO-SWISS CONDENSED MILK CO. (AUSTRALASIA), LTD. (B.P. 285,383, 13.2.28. Austral., 14.2.27).—The apparatus comprises a chart of the percentage compositions of various concentrated substances (e.g., foods) and a rotatable circle on the principle of the slide rule by which the composition after adding a known proportion of water, or the water to be added to give a desired composition, may be determined.

B. M. VENABLES.

Pulverising apparatus. O. CRAIG, Assr. to RILEY STOKER CORP. (U.S.P. 1,714,080, 21.5.29. Appl., 15.12.27).—See B.P. 302,330; B., 1929, 306.

Filtration. E. A. ALLIOTT, C. BOIS, and A. E. HATFIELD (U.S.P. 1,713,317, 14.5.27. Appl., 19.2.27. U.K., 20.3.26).—See B.P. 270,461; B., 1927, 511.

Centrifugal apparatus [for gases]. H. A. HUM-PHREY, ASST. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,714,370, 21.5.29. Appl., 26.11.27. U.K., 15.1.27).—See B.P. 276,557; B., 1927, 842.

Absorption refrigerating apparatus. G. BEAU-MONT and G. MANSIO (B.P. 290,585, 14.5.28. Fr., 13.5.27).

Refrigerating apparatus of absorption type. H. D. FITZPATRICK. From N. V. KODOWA REFRIGERA-TOR Co. (B.P. 311,891, 3.4.28).

Evaporators for refrigerating. H. R. VAN DEVEN-TER and J. A. GRIER (B.P. 299,728, 29.10.28. U.S., 27.10.27).

Thermostats [of float-valve type]. J. L. CATON (B.P. 311,901, 3.1.29).

Apparatus for filling solid articles in layers into receptacles. I. G. FARBENIND. A.-G. (B.P. 299,887, 1.11.28. Ger., 3.11.27).

Heat-treatment of pulverulent materials (B.P. 310,907).—See II. Moisture-absorbent (B.P. 311,578).—See VII.

# II.—FUEL; GAS; TAR; MINERAL OILS.

Determination of hygroscopic moisture in coal. H. Löffler (Chem.-Ztg., 1929, 53, 411).—The coal is dried at 60—70° in a vacuum. A convenient form of apparatus, in which heating is effected by a vapour jacket, is described and illustrated. J. S. CARTER.

Waxes and boghead coals as parents of petroleum. IV. N. D. ZELINSKI and K. P. LAVROVSKI (Ber., 1929, 62, 1264—1266; cf. A., 1928, 731, 865).—Distillation of purified beeswax with aluminium chloride affords carbon dioxide and inflammable gases with a mixture of liquid and solid hydrocarbons of paraffinoid nature. Two substances, m.p. 59° and 70—71°, respectively, are characterised. Boghead coal when heated with aluminium chloride gives a liquid volatile with steam in which unsaturated cyclic hydrocarbons appear to predominate; the portion of the distillate not volatile with steam gives a crystalline paraffin, m.p. 63—64°. H. WREN.

Chemical composition of peat. IV. Chemical studies of highmoor peat from Maine. S. A. WAKSMAN and K. R. STEVENS (Soil Sci., 1929, 27, 389–398; cf. B., 1929, 421).—Examination of some sphagnum peats is recorded. The sphagnum horizon was acid  $(p_{\rm H} 4 \cdot 0)$  and had low ash, nitrogen, and lignin contents and high cellulose, hemicellulose, fat, and wax contents. With the transition to the lower or sedimentary peat layers there is a rise in  $p_{\rm H}$  value, an increase in ash, protein, and lignin values, and a decrease in cellulose and hemicellulose. A. G. POLLARD.

Economic test of low-temperature coking. R. S. McBride (Chem. Met. Eng., 1929, 36, 288-291).-The K.S.G. process, which has been in large-scale operation at Essen for 5 years, has been adopted in a plant now starting work in New Jersey. It consists of 8 retorts consisting of two concentric drums 85 ft. long. These are externally heated by producer gas, the coal travelling first through the inner drum and back through the outer. The transference takes place as the coal is becoming plastic, and the rapid subsequent heating reduces the plastic period to a minimum. Soot deposits are minimised by tangential steam-jets. The gas is to be used for public supply. It is anticipated that the tar will yield 5 gals. per ton of light products and a high content of phenols. Pea-size semi-coke is to be used for generating producer gas. The steam used is generated by the products of combustion, and the total heat used in carbonisation is estimated at 800 B.Th.U. per lb. of coal. C. IRWIN.

Phenol recovery [from coke-oven effluent] and treatment. Works of the Hamilton Coke and Iron Company. B. F. HATCH (Ind. Eng. Chem., 1929, 21, 431-433).—The first large-scale plant operating the Seaboard, N.J., process of phenol recovery is described. Ammonia liquor is withdrawn from the bottom of the free ammonia still before liming and sprayed down a high steel tower packed with tiles against a mixture of 90% of steam and 10% of air, the temperature being kept just below the b.p. of the liquor. The lower three fourths of the tower form dephenolising sections, being packed with steel turnings and fed with caustic soda. Both gas and caustic soda are recirculated. The heat consumption is low owing to good insulation, and the phenol recovery may reach 95%. C. IRWIN.

Occurrence of pyridine bases in the tar oils from Russian bituminous schists. J. DODONOV and E. SOSCHESTWENSKAJA (Ber., 1929, 62, 1348—1352; cf. B., 1926, 1006).—Fractions of the oil, b.p.  $140-250^{\circ}/$ 650 mm., are freed from phenols by treatment with alkali and shaken with sulphuric acid (d 1·3) whereby 1·1% of basic substances are isolated. The bases are further fractionated and the individual fractions are transformed into their hydrochlorides and thence into their additive compounds with mercuric chloride. The presence of pyridine, 2- and 3-methyl-, 2:4-, 2:5-, and 2:6dimethyl-pyridine is established. H. WREN.

Oil-testing apparatus. H. HERBST (Chem.-Ztg., 1929, 53, 344-345).-By means of the apparatus the surface tension of liquids (especially of lubricating oils), the lubricating power of oils in contact with metals, the angle of contact of these oils and metals, the viscosity of pitch, rosins, size, etc., and the thickness of bodies (e.g., the dimensions of metal blocks) may be measured. The apparatus consists of a rectangular table along two adjacent sides of which is fixed a vertical framework in which sliding carriages may be moved vertically up or down by means of micrometer screws. Each carriage carries a horizontal arm; to one of these is fixed a reading telescope or lens, and to the other a. holder supporting a capillary tube in which the height of capillary rise etc. of the liquids under examination may be measured by means of the telescope and the scales on the micrometer screws. For measuring, e.q., the lubricating power of oils in contact with metals, standard rectangular blocks of the same or of different. metals are fixed by means of clamps and distance pieces so that their inner faces are parallel and at a standard distance apart. The blocks are then placed centrally under a capillary tube containing the required oil, and the distance through which the surface of the oil in the tube falls is measured. In order to determine: the dimensions of the metal blocks referred to, the: capillary tube is replaced by a steel ruler suitably graduated. The apparatus can also be used for measurement of small pressure differences by substituting a. manometer for the capillary tube. B. P. RIDGE.

Oxidation of motor fuels. E. BERL, K. HEISE, and K. WINNACKER (Z. physikal. Chem., 1929), 144, 223; cf. B., 1929, 158).—Acknowledgment is made that the "peroxide" theory, which explains the knocking of certain fuels in internal-combustion engines, was put forward by Moureu and his co-workers in 1925, and by Callendar (B., 1927, 272) only in 1927. A. JI MEE.

Determination of unsaturated and aromatic hydrocarbons in light oils and motor spirits. A. B. MANNING (J.C.S., 1929, 1014-1020).-0.2-0.5 g. of the oil is vaporised in a current of air and passed through a bubbler charged with a 10% mixture of concentrated nitric acid (or 16% of potassium nitrate) in concentrated sulphuric acid. The increase in weight of the bubbler gives the total unsaturated and aromatic hydrocarbons in the oil. This solution in the acid mixture is heated on the water-bath for 2-3 hrs. to complete both the nitration of the aromatic hydrocarbons. and the oxidation of the unsaturated hydrocarbons. The cooled acid solution is poured into excess of water and the nitro-compounds are extracted with three 50-c.e. portions of benzene. The benzene extract is itself extracted with 10% sodium hydroxide, and is finally evaporated to dryness. The weight of aromatic hydrocarbons in the original oil is calculated by multiplying the weight of mixed nitro-compounds obtained by the factor 0.452; the result so obtained is liable to an error of about one unit on the percentage for every 20% of unsaturated hydrocarbons present.

R. J. W. LE FÈVRE,

See also A., June, 655, Low-temperature oxidation of hydrocarbons (LEWIS). 658, Activity of charcoal (ALEXSEEVSKI and AVGASTINIK). 673, Fractionating columns (MIDGLEY). 687, Action of acetylene on benzene in presence of aluminium chloride (BÖESE-KEN and ADLEB).

Oil for heating purposes. McKechnie.—See I. Wet carbonisation of wood. Schwalbe.—See V. Sulphuric acid. Spangler.—See VII. Cast-iron pipes. Bradshaw.—See X.

# PATENTS.

Briquetting of fuels. CHEM.-TECHN. GES.M.B.H. (B.P. 300,195, 28.9.28. Ger., 8.11.27).—Briquettes are manufactured from a blend of a caking and a noncaking or poorly-caking fuel; the two fuels are first heated separately, then mixed and briquetted under pressure. The caking fuel is heated to a temperature below its softening point, whilst the non-caking fuel is raised to a higher temperature, so that on admixture the interchange of heat causes the former fuel to cake. The caked product may be subjected to further heattreatment before being allowed to cool.

#### A. B. MANNING.

Destructive hydrogenation of carbonaceous materials. H. G. WATTS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 311,197—8, [A] 28.1.28, [B] 30.1.28).— (A) The separation of the solid and liquid products of the destructive hydrogenation of coal etc. by filtration is facilitated by the preliminary addition thereto of finely-divided, insoluble, carbonaceous material, e.g., coal or coke. The product is preferably filtered hot, *i.e.*, at about 100°, after the removal of the lowerboiling constituents. (B) The separation is further facilitated by dilution of the material with an oil capable of dissolving pitch, preferably an oil of b.p. 200—300° derived from the process itself. The solid matter may be washed with light oil and then briquetted.

#### A. B. MANNING.

Heat treatment of pulverulent [carbonaceous] materials. J. N. BALLEY, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 310,907, I.2.28).—A retort for the fractional distillation of carbonaceous material is so shaped that the material falls through one elongated chamber or a succession of separate vortex chambers, the heating gas being admitted tangentially at different regions or to different chambers; portions of the contents of the chambers are withdrawn from points in different regions, but all adjacent to the axis of the vortices, by means of a number of concentric conduits. B. M. VENABLES.

Liquefying and solubilising coals by extraction with solvents under pressure. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 311,031, 1.3.28).— Solid carbonaceous materials are heated under pressure with a suitable solvent, *e.g.*, tetralin, high-boiling mineral oil fractions, in the presence of small quantities of halogens, halogen hydrides, or compounds which set free these substances under the conditions of the reaction.

A. B. MANNING.

Manufacture of valuable liquid products from varieties of coal, tars, mineral oils, etc. I. G. FARBENIND. A.-G. (B.P. 311,628, 3.7.28. Addn. to B.P. 282,384; B., 1929, 465).—The apparatus described in the main patent is modified by constructing of silver or its alloys those parts which come into contact with sulphur-contaminated hydrocarbons at elevated temperatures. A. B. MANNING.

Manufacture of valuable liquid hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 311,251, 5.12.27).—Coals, tars, mineral oils, etc. are subjected to destructive hydrogenation or cracking in the presence of catalysts, *e.g.*, tungstic acid, molybdic acid, either alone or mixed with chromium or other metallic oxides, cobalt oxide, etc. which have been subjected to a preliminary treatment at an elevated temperature with gases such as carbon dioxide, sulphur dioxide, nitric oxide, nitrogen, or oxygen, which have no reducing action on them. A. B. MANNING.

Control of combustion. W. M. HEPBURN, Assr. to SURFACE COMBUSTION CO. (U.S.P. 1,710,772, 30.4.29. Appl., 10.12.23).—A furnace is provided with a regulator for controlling the rate of supply of air, which is automatically increased or decreased in accordance with the presence in the flue gases of unconsumed fuel or unconsumed air respectively. A. B. MANNING.

Production of activated carbon. W. M. WILLIAMS, R. S. CLAYTOR, (SIR) J. P. FRY, and A. R. HARPER (B.P. 310,908, 1.2.28).-Powdered carbonaceous material, e.g., coal dust, mixed with a limited supply of air, is injected into a retort maintained at 800-1000°, and the partially burnt material is kept in suspension in the gaseous combustion products until it is activated. The retort may take the form of a vertical cylindrical chamber into the upper end of which the material and air are injected tangentially, the retort having first been heated by the combustion of a gas-air mixture therein. An acid atmosphere is maintained within the retort either by addition of acid to the raw material or its introduction directly into the combustion chamber. A water spray may be introduced into the lower part of the chamber in order to quench the product as soon as activation is complete. A. B. MANNING.

Manufacture of activated carbon. A. B. RAY, E. G. DOYING, and J. J. BUTKOVSKY, ASSIS. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,694,040, 4.12.28. Appl., 22.10.25).—Cellulose material, e.g., nut shell, is impregnated with 50% of phosphoric acid or 100% of zinc chloride, or other dehydrating agent, and heated to 350°, leached, and reheated to 600° in steam or other oxidising atmosphere. R. BRIGHTMAN.

Gas producers. F. L. BROUGHTON and D. HAD-LINGTON (B.P. 311,587, 3.7.28).—A producer with a rotatable base has attached thereto an ash box, a regular discharge of ashes from the producer to the box being caused by arms rotating on a shaft within the latter. A scraper on the underside of the base gathers together the ashes discharged from the box and delivers them to a truck or conveyor. Vertical rods attached to the base prevent the formation of masses of clinker within the producer. Air and steam are admitted through a central stationary pipe, above the open end of which is a cap, and attached to which are guards or scrapers to prevent blocking of the inlet. Additional steam is preferably supplied through the sides of the producer at a slightly higher level than the main steam and air inlet. A. B. MANNING.

Production of gas. SILAMIT-WERKE DR. STRASS-MANN & Co., FABR. FEUER- & SÄUREFESTER PROD. M.B.H. (Swiss P. 122,317, 12.5.26).—Part of the distillation retort is heated directly by combustion gases during the whole process, while the remainder is heated directly until the gas consumption is at a maximum; it is then heated by the waste gases from the heating of the first part. L. A. COLES.

Apparatus for production of oil gas. A. SCHILLING, R. SACHSE, D. LIAMIN, and T. CALLAERT (U.S.P. 1,710,900, 30.4.29. Appl., 23.11.26).—A generating chamber with a chequered hearth has a wall of refractory material rising therefrom and dividing the interior into a number of concentrically disposed chambers, which are connected by apertures in the lower part of the wall. A combustible mixture of oil, air, and steam is projected axially downwards into the inner chamber, while jets of oil and steam are projected against the outer surface of the refractory wall. The gases produced pass through the chequered hearth, and are then withdrawn from the apparatus.

A. B. MANNING.

Gas scrubber. G. FAST (U.S.P. 1,713,175, 14.5.29. Appl., 28.5.25).—The gas passes through a number of rotating sprays of liquid moving in opposite directions. J. A. SUGDEN.

Extraction of acetylene from gases. I. G. FARB-ENIND. A.-G. (B.P. 303,068, 27.12.28. Ger., 27.12.27). —Gases poor in acetylene are treated with acetonitrile cooled to about —10°; the acetylene is recovered on boiling. H. ROYAL-DAWSON.

Still for use in oil refining, and method of making same by electric arc welding. E. C. R. MARKS. From A. O. SMITH CORP. (B.P. 310,761, 24.12.27; cf. B., 1929, 385).—Two end heads in the form of hollow hemispheres and a number of thick, seamless, annular sections of cast steel of the same thickness and diameter, having the circumferential edges partially cut away are alined end to end and fused together by an electric arc in the region of the grooves to constitute an integral tubular structure without longitudinal joints.

H. S. GARLICK.

Extraction of oils from materials containing the same. I. G. FARBENIND. A.-G. (B.P. 283,159, 4.1.28. Ger., 5.1.27).—Oils, mixed with solid coaly materials and inorganic matter, obtained as residues in the destructive hydrogenation of carbonaceous materials, are completely extracted by treatment with liquid sulphur dioxide. H. S. GARLICK.

Manufacture of natural gasoline. H. E. THOMPSON, Assr. to CARBIDE & CARBON CHEM. CORP. (U.S.P.1,695,162, 11.12.28. Appl., 12.4.24).—Liquid gasoline prepared from natural gas, after stablisation by removal of undesirable volatile fractions, is cooled below its normal b.p. for example, first with water, and then to at least 0° with the cold vapours (-20°) from the rectifying column. Alternatively, vapour may be withdrawn

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from the liquid gasoline, compressed, cooled, and expanded, and the cold expanded material returned.

R. BRIGHTMAN.

Cracking of hydrocarbons. E. C. HERTHEL and H. L. PELZER, ASSTS. to SINCLAIR REFINING CO. (U.S.P. 1,703,528, 26.2.29. Appl., 17.1.25).—The bulk supply tank is provided with a filter bed containing metallic oxides, and oil circulates relatively slowly through the tank and bed and rapidly through an externally-heated cracking coil from which the hot oil is discharged to the supply tank by a submerged pipe above the bed. Vapours escape to a reflux tower, and thence to condenser and receiver. Crude oil is fed into the top of the reflux tower, and the reflux is returned to the tank or direct to the circulating pipe to the heating coil. Tar oil may be withdrawn from this circulating pipe before it discharges to the heating coil. R. BRIGHTMAN.

Conversion [cracking] of hydrocarbon oils. C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,703,100, 26.2.29. Appl., 31.10.21).—The crude oil passes downward through closed coils forming the condensing medium in a reflux tower up which the cracked vapours from the vaporising chamber pass to the water condenser. Pressure distillate may also be sprayed in at the top of the reflux tower in contact with the ascending vapours, and the phlegms collect in a well into which the crude oil from the coil discharges and from which the oil thus preheated is pumped to the heating tubes. Part of the oil may be by-passed from the well, through the cooler, to the crude oil feed to the tower. R. BRIGHTMAN.

Cracking of hydrocarbon oil and apparatus therefor. C. ARNOLD. From STANDARD OIL DEVELOP-MENT Co. (B.P. 311,362, 10.2.28).—A stream of oil is heated to cracking temperature in a single passage through a heating zone and passed to a digestion zone in which partial vaporisation occurs. The vaporised portion is partially condensed in a condensing zone under substantially the same pressure as that of the heating and digestion zones. The condensate is run wholly or partially under the action of gravity into the stream of hot oil passing from the heating to the digestion zone at a position anterior to that at which it enters the digestion zone and in sufficient quantity to control the temperature therein. H. S. GARLICK.

Treatment [cracking] of petroleum oils. W. M. CROSS, ASST. to GASOLINE PRODUCTS Co., INC. (U.S.P. 1,696,030, 18,12.28. Appl., 14.10.22).—Petroleum oil under 400—750 lb./in.<sup>2</sup> pressure is heated above its flash point and passed into a reaction chamber, where it is maintained at 375—485° by the controlled introduction, through a perforated pipe, of a combustible gas. The converted oil is passed into a still, where the pressure is released, and the lighter fractions distil off. The reflux condensate is returned to the heating coil and the residuum in the still, containing the carbonaceous matter, is drawn off. R. BRIGHTMAN.

Emulsifiable oil. D. R. MERRILL, Assr. to UNION OIL CO. OF CALIFORNIA (U.S.P. 1,695,197, 11.12.28. Appl., 14.6.23).—Emulsions for treating wools, spraying fruit trees, or for use as sheep dip, lubricants, or Turkeyred oil substitute, are obtained by mixing 70 pts. of

sodium naphthenate, of about 8% water content, with 17 pts. by vol. of butyl alcohol or other aliphatic alcohol containing more than two carbon atoms, and dissolving 30 pts. of this mixture in about 70 pts. by vol. of a mineral lubricating oil. R. BRIGHTMAN.

Decolorisation of kerosene, gasoline, and similar light petroleum distillates. V. A. KALICHEVSKY, ASST. to UNION OIL CO. OF CALIFORNIA (U.S.P. 1,695,251, 11.12.28. Appl., 19.9.27).—When washed with sodium hydroxide, *e.g.*, with 5% of a 10% solution, before agitation with acid and clay or with clay alone, light petroleum distillate gives a product equal in colour to, but more stable than, that obtained by air oxidation. R. BRIGHTMAN.

Manufacture of pure mineral oil sulphonates. C. FISCHER, JUN., and W. T. REDDISH, ASSTS. to TWITCHELL PROCESS CO. (U.S.P. 1,703,838, 26.2.29. Appl., 11.10.27).—Mahogany soap, containing, e.g., 20—25% of oil, is dried and mixed with an aqueous solvent, e.g., alcohol above 75% concentration, saturated with sodium carbonate or other water-soluble salt to keep the sulphonates out of the lower layer. The upper layer on decantation and evaporation of the solvent affords pure mineral oil sulphonates. R. BRIGHTMAN.

Manufacture of carbonaceous material. H. N. GILBERT, ASST. to ROESSLER & HASSLACHER CHEM. Co. (U.S.P. 1,714,165, 21.5.29. Appl., 9.5.25).—See B.P. 251,982; B., 1927, 805.

Plant for utilising the sensible heat of hot coke. O. HELLER (U.S.P. 1,714,168, 21.5.29. Appl., 29.9.24. Ger., 3.10.23).—See B.P. 222,886 ; B., 1925, 580.

Apparatus for drying and distilling lignite, peat, non-coking coals, and other similar carbonaceous matter. H. DEBAUCHE (U.S.P. 1,713,032, 14.5.29. Appl., 6.7.27. U.K., 20.7.26).—See B.P. 270,921; B., 1927, 547.

Production of mixed coal gas and water-gas. F. D. MARSHALL (U.S.P. 1,713,189, 14.5.29. Appl., 10.4.26).—See B.P. 261,975; B., 1927, 244.

Conversion of hydrocarbon oils. J. F. DONNELLY (U.S.P. 1,712,789, 14.5.29. Appl., 21.11.24).—See B.P. 243,339; B., 1927, 356.

Manufacture of synthetic liquid fuels. E. A. PRUDHOMME, ASST. to SOC. INTERNAT. DES PROC. PRUD-HOMME (S.I.P.P.) (U.S.P. 1,711,856, 7.5.29. Appl., 7.4.26. Fr., 9.3.26).—See B.P. 267,512; B., 1928, 470.

Low-compression fuel. E. V. BERESLAVSKY, Assr. to ETHYL GASOLINE CORP. (U.S.P. 1,713,589, 21.5.29. Appl., 17.9.25).—See B.P. 258,550; B.,1927, 900.

Apparatus for treating [hydrocarbon] oils. G. W. WALLACE (U.S.P. 1,714,198, 21.5.29. Appl., 24.10.24. U.K., 1.8.24).—See B.P. 233,395; B., 1925, 662.

Apparatus for [oil-]separation of combustible material from its associated non-combustible material. W. L. REMICK (U.S.P. 1,711,326, 30.4.29. Appl., 18.2.26).

Quenching of coke. J. PINTSCH A.-G. (B.P. 299,315, 22.10.28. Ger., 21.10.27).

[Mixing device for] preparation of charges for

use in internal-combustion engines. A. S. JOHN (B.P. 311,847, 9.8.28).

Separation of gaseous and liquid products (B.P. 311,193).—See I. Hydrogen and gases containing it (B.P. 311,299). Sulphur from gases (U.S.P. 1,695,068).—See VII. Improvement of ferrous metals (U.S.P. 1,712,879).—See X. Sulphur product (U.S.P. 1,690,335).—See XIII.

# III.—ORGANIC INTERMEDIATES.

Amination by ammonolysis [the substitution of the amino-group for other radicals by means of ammonia]. P. H. GROGGINS (Chem. Met. Eng., 1929, 36, 273-275).-The type of reaction referred to is illustrated by the preparation of aniline from chlorobenzene,  $\beta$ -naphthylamine from  $\beta$ -naphthol, etc. It is affected by possible variables as follows. Excess of ammonia of a given concentration minimises the formation of imino-compounds and phenols. Increase of ammonia concentration acts similarly. The reaction velocity is increased by rise in temperature within the working limits for the reaction considered. Stirring sufficiently to mix the materials thoroughly is necessary. In the case of water-insoluble aromatic halogen compounds the use of nitrobenzene, amyl alcohol, etc. as a common solvent is desirable. The use of a copper catalyst should be limited to the production of volatile amines or those that will remain in solution in the ammonia. The value of such catalysts has been much exaggerated.

C. IRWIN.

See also A., June, 693, Esters of aminoazobenzenesulphonic acids (HANTZSCH). Aminoazobenzenes and their salts (HANTZSCH). Aminoazobenzenes and their salts (HANTZSCH and VOIGT). 701, Polyiodoanthraquinones (ECKERT and KLINGER). 702, Reduction products of 1:1'-dianthraquinonyl (ECKERT). 712, Determination of carbon and carbon dioxide (FRIEDEMANN and KENDALL). 713, Determination of amino-acids (ROSENTHALER). 724, Acetaldehyde by fermentation (KISCH and LEIBOWITZ). Conversion of acetic acid into succinic and fumaric acids by *Mucor stolonifer* (BUTKEVITSCH and FEDOROV). 730, Purification of picric acid (BENEDICT).

Phenol from coke-oven effluent. HATCH.—See II. Detection of *iso*propyl alcohol in presence of acetone. MEYER.—See XVIII.

#### PATENTS.

Manufacture of methyl alcohol. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 308,181, 19.9.27).— Almost pure methyl alcohol is obtained by hydrogenation of oxides of carbon at ordinary or slightly increased pressure and at 100—250° by using a copper catalyst to which one or more oxides of metals of groups II to VII have been added; metals of group VIII are excluded. A catalyst, prepared by precipitating a solution of copper nitrate (2 mols.) and magnesium nitrate (1 mol.) with sodium hydroxide at 20°, followed by reduction with dilute (5%) hydrogen at 100—250°, converts a mixture of carbon monoxide and hydrogen at 150° and at ordinary pressure into colourless methyl alcohol,  $d^{15}$  0.792, practically free from unsaturated compounds.

C. HOLLINS.

Manufacture of mono- and poly-hydric alcohols. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 309,200, 28.9.27).—The crude reaction product from aldolisation of aldehydes or ketones (or mixtures of either or both), without separation of the alkaline condensing agent, is hydrogenated in presence of diluents (water, alcohols) and of a catalyst (nickel, cobalt, copper, or mixtures). Aldol from acetaldehyde gives  $\alpha\gamma$ -butylene glycol; from *n*-butaldehyde,  $\gamma$ -octanol. The alkaline condensation products from formaldehyde or from formaldehyde and aldol yield syrups on hydrogenation. [Stat. ref.] C. HOLLINS.

Manufacture of esters and other valuable organic compounds. E. I. DU PONT DE NEMOURS & Co. (B.P. 287,846, 12.3.28. U.S., 26.3.27).—Vapour of ethyl (or higher) alcohol is led over a dehydrogenating catalyst, *e.g.*, copper oxide containing oxides of manganese and magnesium, at 250—500° under high pressure (*e.g.*, 275 atm.). Ethyl alcohol gives ethyl acetate, *n*-butyl alcohol, and acetaldehyde. C. HOLLINS.

Manufacture of new compounds containing sulphur. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 307,728, 12.12.27).—Secondary alicyclic amines are converted by the usual methods into dithiocarbamates. Examples of amines used are: cyclohexylethylamine, 2-methylcyclohexylethylamine, and cyclohexylmethylamine. C. HOLLINS.

**Production of aldehyde-sulphoxylates.** J. Y. JOHNSON, From I. G. FARBENIND, A.-G. (B.P. 308,229, 19.9.27).—Solutions of aldehyde or ketone bisulphite compounds or aldehyde hyposulphite compounds are treated with hydrogen in presence of an activated or unactivated hydrogenation catalyst. To avoid hydrolysis the  $p_{\rm H}$  should be 7 or a little above. In the reduction of formaldehyde bisulphite an unactivated catalyst and a high pressure (40—140 atm.) must be used. The preparation of sulphoxylates of formaldehyde, benzaldehyde, *m*-tolualdehyde, acetone, and methyl ethyl ketone is described. The catalysts are nickel, nickel-cobalt, nickel-tungsten, nickel-ormium-tungsten, on kieselguhr. C. HOLLINS.

Manufacture of methylol [hydroxymethyl] derivatives of urethanes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 309,108, 3.1.28).—Paraformaldehyde is stirred into a molten urethane (alkyl carbamate) to which a little barium or calcium hydroxide or oxide has been added; nearly quantitative yields of N-hydroxymethylurethanes are obtained. Methyl Nhydroxymethylcarbamate, m.p. 61—62°, and the corresponding n-propyl (m.p. 63—64°), n-butyl (m.p. 62—63°), and  $\beta$ -ethoxyethyl, m.p. 59—60°, derivatives are described. C. HOLLINS.

cycloHexyl compounds and their manufacture. R. ADAMS, ASST. to ABBOTT LABORATORIES (U.S.P. 1,703,186, 26.2.29. Appl., 8.8.27).—cycloHexyl alkyl halides are condensed with substituted malonic esters, or  $\omega$ -cyclohexylalkylmalonic esters or cyclohexylmalonic esters with alkyl halides and the product is hydrolysed; the malonic acid derivative fused affords bactericidal compounds (cf. A., 1928, 62). R. BRIGHTMAN. Catalytic oxidation of organic compounds. A. O. JAEGER, ASST. to SELDEN CO. (U.S.P. 1,694,122, 4.12.28. Appl., 24.11.26).—Zeolites containing catalytically active elements, such as vanadium, obtained by base exchange methods are used alone or diluted with silica as catalysts, *e.g.*, for vapour-phase organic oxidations. Alternatively, an inert artificial or natural zeolite may be impregnated with a catalyst. Thirty-one examples are given. R. BRIGHTMAN.

Purification of benzoic acid and its derivatives. A. O. JAEGER, ASST. to SELDEN CO. (U.S.P. 1,694,124, 4.12.28. Appl., 17.10.27).—Benzoic acid or its derivatives are separated from the corresponding phthalic anhydride by treating the mixture with the vapours of an organic solvent for benzoic acid at temperatures at which the benzoic acids are volatile, but below those at which the phthalic acids are transformed into anhydrides. The operation is effected in presence of water or steam and may be continuous.

R. BRIGHTMAN. Manufacture of benzoates. W. S. CALCOTT and H. W. DAUDT, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,692,927, 27.11.28. Appl., 13.9.24. Renewed 27.2.28).—Coloured impurities in benzoates made from benzoic acid from phthalic acid are removed by oxidation with permanganate. C. HOLLINS.

Benzoic acid salt of aminobenzoic esters. H. SEYDEL (U.S.P. 1,690,705, 6.11.28. Appl., 5.4.27).— Aminobenzoic esters in dilute hydrochloric acid are added to sodium benzoate solution, giving, *e.g.*, *ethyl* p*aminobenzoate benzoate*, m.p. 70—71°. R. BRIGHTMAN.

Purification of nitroaniline. H. MERRILL, Assr. to TOWER MANUF. Co. (U.S.P. 1,692,308, 20.11.28. Appl., 1.7.25).—p-Nitroaniline made from chloronitrobenzene is stirred with hot 10% caustic alkali, filtered at 40—50°, and allowed to crystallise. C. HOLLINS.

Hydrogenation of pyridine and/or its homologues. TECHN. RES. WORKS, LTD., and E. J. LUSH (B.P. 309,300, 31.3.28).—Pyridine etc. is passed in the liquid state over active nickel at 180° in a hydrogen atmosphere at 300 lb./in.<sup>2</sup> The products are piperidine and a little tetrahydropyridine, with no amylamine.

C. HOLLINS.

Manufacture of 8-amino-6-alkoxyquinolines. I. G. FARBENIND. A.-G., and A. CARPMAEL (B.P. 307,727, 12.12.27. Addn. to B.P. 267,457; B., 1927, 379).— The 8-amino-group is introduced by Hoffmann decomposition of 6-alkoxyquinoline-8-carboxylamides. *E.g.*, 6-methoxyquinoline-8-carboxylamide, m.p. 169—170°, obtained from the methyl ester, is treated with potassium hydroxide and bromine. C. HOLLINS.

Manufacture of alkali salts of nitrosoamines of primary aromatic amines. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 307,965, 15.12.27).—A diazo solution is run into cooled alkali and the resulting syn-diazotate is added to alkali at the appropriate temperature for conversion into *iso*diazotate. The optimum temperatures for this conversion are given for the following amines: *m*-chloroaniline (105°), 3:4dichloroaniline (110°), 3:5-dichloroaniline (100°), 2:4:5- and 2:4:6-trichloroanilines (100°), 2-chloro-5bromoaniline (100°), 5-chloro-o-toluidine (115°), 4chloro-o-toluidine (110°), 5 : 6-dichloro-o-toluidine (120°), 2 : 6-dichloro-m-4-xylidine (120°), 4- and 5bromo-o-toluidines (110—115°), 2 : 5- and 5 : 2-chlorobromo-p-toluidines (105°), 4-chloro-m-toluidine (110°), 4 : 5-dichloro-o-toluidine (120°), 2 : 5- and 2 : 3-dichlorop-toluidines (105°), 6-chloro-o-toluidine (115°), 3-chlorop-toluidine (105°), 3 : 6-dichloro-o-toluidine (105°), 4 : 6-dichloro-m-toluidine (105°), 3 : 5-dichloro- and -dibromo-p-toluidines (100°), 4 : 6-dichloro-manisidine (100°), 4 : 5-dichloro-o-anisidine (115— 120°), 5-chloro-o-anisidine (120°), 4 : 6-dichloro-manisidine (100°), 4 : 5-dichloro-o-anisidine (110°), 6chloro-3-methoxy-p-toluidine (115°),  $\alpha$ -aminoanthraquinone (120—125°), 2 : 2'-dichlorobenzidine (100°).

C. HOLLINS.

Manufacture of monohalogenated naphthastyril compounds. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 309,107, 3.1.28. Cf. B.P. 278,100; B., 1927, 102).—Naphthastyril, boiled with sodium hypochlorite solution, yields the N-chloro-compound, m.p. 132°, which is converted by heat or by treatment with acid or in boiling solvents into 6-chloronaphthastyril (annexed formula), m.p. about 270°; this is also obtained

directly by the action of chlorine in presence of iodine at  $15-20^{\circ}$ , or from an acid suspension of naphthastyril hydrochloride and sodium chlorate, or by the action of sulphuryl chloride in tetrachloroethane at  $50-60^{\circ}$ . Bromine in tetrachloroethane at  $50-60^{\circ}$  in presence of iodine gives 6(?)-bromonaphthastyril, m.p.

256—257°. N-Chloro- (m.p. 117°) and 6-chloro- (m.p. 250—252) derivatives of 5-ethoxynaphthastyril are also described. C. Hollins.

Production of anthraquinone colouring matters. W. M. MURCH, Assr. to NAT. ANILINE & CHEM. Co. (U.S.P. 1,692,854, 27.11.28. Appl., 2.4.26).—The alkaline fusion of  $\beta$ -aminoanthraquinone for indanthrone is performed in a nickel-steel vessel.

C. HOLLINS.

Manufacture of highly-chlorinated perylenes. A. ZINKE and K. FUNKE, Assrs. to F. BENSA (U.S.P. 1,713,362, 14.5.29. Appl., 11.4.28. Austr., 2.4.27).— See B.P. 288,171; B., 1928, 921.

Preparation of phthaloyl-2: 3-thionaphthens. F. MAYER, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,713,670, 21.5.29. Appl., 25.1.27. Ger., 29.1.26).— See B.P. 265,193; B., 1928, 635.

Purification of crude anthracene. SELDEN Co., Assees. of A. O. JAEGER (B.P. 304,179, 19.3.28. U.S., 16.1.28).—See U.S.P. 1,693,713; B., 1929, 350.

Manufacture of 1:4-diarylamino-5:8-dihydroxyanthraquinone. K. WEINAND, Assr. to GRAS-SELLI DYESTUFF CORP. (U.S.P. 1,713,576, 21.5.29. Appl., 26.6.26. Ger., 3.7.25).—See B.P. 263,370; B., 1927, 136.

Manufacture of benzanthrone compounds. B. STEIN, W. TRAUTNER, and R. BERLINER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,713,565, 21.5.29. Appl., 16.9.26. Ger., 8.10.25).—See B.P. 297,129; B., 1928, 847. Manufacture of Bz-methylbenzanthrones. A. WOLFRAM and H. GREUNE, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,695,626, 18.12.28. Appl., 28.11.25. Ger., 4.12.24).—See B.P. 244,120; B., 1927, 326.

1-Phenylbenzanthrone compounds. Cinnamylideneanthrones. 10-Phenylbenzanthone and its derivatives. R. BERLINER, B. STEIN, and W. TRAUTNER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,713,571 and 1,713,590—1, 21.5.29. Appl., [A—c] 16.9.26. Ger., [A, B] 8.10.25, [C] 20.10.25).—See B.P. 297,129; B., 1928, 847.

Preparation of flavanthrone. W. SMITH and J. THOMAS, ASSTS. to SCOTTISH DYES, LTD. (U.S.P. 1,714,249, 21.5.29. Appl., 16.5.27. U.K., 26.5.26).—See B.P. 287,039; B., 1927, 902.

Base-exchange bodies (U.S.P. 1,694,620).—See VII. Reduction of sugars to alcohols (U.S.P. 1,712,952).—See XI. Pinacol (U.S.P. 1,703,220).—See XX.

#### IV.—DYESTUFFS.

Analysis of dyestuffs. H. E. FIERZ-DAVID (J. Soc. Dyers and Col., 1929, 45, 133—142).—The dye is first purified and the group to which it belongs ascertained by means of Green's tables. The ultra-violet lamp is valuable for this purpose, and it has been found that dyes containing an -S- group or a thiazole ring invariably show a very strong fluorescence in ultraviolet light. Flourescence is thus a general property of some classes of dyes. It is shown that a study of the literature, especially the patent literature, is of great assistance when dealing with unknown dyes. The above methods have been applied in the case of such dyes as Brilliant Wool Blue FFR, Alizarine Fast Grey 2BL, and Indocyanine BF. The value of the spectroscope in dye analysis is also emphasised.

#### L. G. LAWRIE.

See also A., June, 671, Device for identifying colours (HOLMES). 694, Absorption spectra and composition of azo dyes (BRODE). 702, Reactions for cantharidin (VAN URK). 706, Pyrimidine dyes (JOHNSON). 712, Eosinates (HOLMES).

#### PATENTS.

Manufacture of [black benzanthrone] vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,723, 10.12.27).—Black vat dyes are obtained by alkaline fusion of 2-aminobenzanthrones carrying as N-substituent a 1-anthraquinonyl residue. Examples are 2-benzanthronyl derivatives of 1-amino-, 1-amino-4methoxy-, 1:6- or 1:5-diamino-anthraquinone, and condensation products from 2:9-dichlorobenzanthrone and 1-aminoanthraquinone, or from 2:10-dichlorobenzanthrone and 1-amino-4-methoxyanthraquinone.

#### C. HOLLINS.

Manufacture of [black dibenzanthrone] vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,947, 12.9.27, 16.3. and 19.3.28; cf. B.P. 307,364 and 285,502; B., 1929, 388).—N-Substituted amino-dibenzanthrones or *-iso*dibenzanthrones in which the N-substituent is an aromatic or heterocyclic residue and may be complex (e.g., residues of perylene, dibenz-

pyrene, anthanthrone, ms-anthradianthrone, etc.) and/or may link together two or more aminodibenzanthrone molecules, are valuable black vat dyes. They may be prepared, for example, by condensing the aminodibenzanthrone obtained by reduction of the nitro-compound (B.P. 1818 of 1905; B., 1905, 963) with 1- or 2-chloroanthraquinone, dibromoanthanthrone, trichloroanthraquinoneacridone, dibromoisodibenzanthrone, dichloroallo-ms-naphthadianthrone, tri- or tetra-bromopyranthrone, nitrobenzanthrone (cf. B.P.220,212), a-nitronaphthalene, 1: 5-dinitroanthraquinone, chloranil, cyanuric chloride, or with cyanuric chloride and 1: 4-aminomethoxyanthraquinone; or of aminodibenzanthrone from the nitro-compound of B.P. 220,212 (B., 1924, 862) with chloranil, or cyanuric chloride and 1 aminoanthraquinone; or of nitrodibenzanthrone with hydrazine, hydrazine and tetrabromopyranthrone, or hydrazine and 1:5-dichloroanthraquinone; or aminodibenzanthrone, from hydroxylamine and dibenzanthrone, with tribromopyranthrone. [Stat. ref.]

#### C. HOLLINS.

Manufacture of sulphur dye pastes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 309,061, 31.12.27).— Addition of a carbohydrate (starch, flour) which is swelled by alkali to sulphide dye pastes prevents separation of crystals or water even on long keeping or on addition of the sodium sulphide required for the dyeing operation. Bactericidal agents may be added to prevent mildew ; if Chloramine T is used the starch is solubilised during the heating in the dye-bath. The amount of carbohydrate added should be less than 12½% of the weight of dye. C. HOLLINS.

Manufacture of monoazo dyes [for wool and acetate silk]. I. G. FARBENIND. A.-G. (B.P. 282,683, 20.12.27. Ger., 23.12.26) .- Violet to black wool dyes (which when they contain only one sulphonic group also dye acetate silk) are obtained by coupling p-hydroxyphenyl-B-naphthylamine (or derivatives) with diazotised p-nitroaniline-o-sulphonic acid, sulphonic acids of 4-nitroα-naphthylamine, or 5-nitro-o-toluidine-ω-sulphonic acid, or derivatives of these. Examples of coupling components used are p-hydroxyphenyl-B-naphthylamine, its 7-sulphonic acid, 2-p-hydroxyphenylamino-3-naphthoic acid, and 4'-hydroxy-3'-sulphophenyl-B-naphthylamine. The diazo components are p-nitroaniline-osulphonic acid, 5-nitro-o-anisidine-3-sulphonic acid, p-nitroaniline-2: 5-disulphonic acid, 4-nitro-a-naphthylamine-5- and -6-sulphonic acids, and 5-nitro-o-toluidine-C. HOLLINS. ω-sulphonic acid.

Manufacture of [direct green] azo dyes. A. CARPMAEL. From I. G. FARBENIND, A.-G. (B.P. 308,958, 30.12.27).—An aminoazo compound is diazotised and coupled with 1:2-aminonaphthyl ethyl ether (or derivative) and the product is rediazotised and coupled with an acylated 1:8-aminonaphtholsulphonic acid. Examples are: sulphanilic acid  $\rightarrow$  o-toluidine  $\rightarrow$  2ethoxy-Cleve acid  $\rightarrow$  N-benzoyl-H-acid; o-toluidine-5sulphonic acid  $\rightarrow$  o-toluidine  $\rightarrow$  2-ethoxy-Cleve acid  $\rightarrow$  n-o-chlorobenzoyl-H-acid; 2:8-naphthylaminesulphonic acid  $\rightarrow$  aniline (as its methane- $\omega$ -sulphonic acid) or m-5-xylidine  $\rightarrow$  2-ethoxy-Cleve acid  $\rightarrow$  N-acetyl-Hacid. C. HOLLINS. Manufacture of fast azo dyes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 309,216, 31.12.27).— Dyes of good fastness to light are obtained in substance or on the fibre by coupling diazotised 2:4:5-trichloroaniline with a 2:3-hydroxynaphthoic arylamide. [Stat. ref.] C. HOLLINS.

Manufacture of insoluble azo dyes. I. G. FARB-ENIND. A.-G. (B.P. 282,682, 20.12.27. Ger., 22.12.26). -2:3-Hydroxynaphthoic arylamides are coupled in substance or on the fibre with a diazotised unsulphonated aminocarbazole to give violet pigments or dyeings. Examples are: the  $\beta$ -naphthylamide with 2-aminocarbazole, and the dianisidide with 3:6-dichloro-2aminocarbazole. C. HOLLINS.

Manufacture of [solubilised o-hydroxy] azo dyes. I. G. FARBENIND. A.-G. (B.P. 282,107, 12.12.27. Ger., 13.12.26).—o-Hydroxyazo dyes are treated with chlorosulphonic acid or esters, especially in presence of pyridine or dimethylaniline, to give soluble sulphuric esters. The product from 4-chloro-o-aminophenol  $\rightarrow \beta$ -naphthol dyes wool orange, becoming reddish-brown on chroming. The sulphuric ester of 5-nitro-o-aminophenol  $\rightarrow$ 2: 3-hydroxynaphthoic acid or phenylmethylpyrazolone may be reduced and phosgenated to give a direct cotton dye (blue-violet or red, respectively, after coppering). C. HOLLINS.

Manufacture of [violet] indigoid vat dyes. I. G-FARBENIND. A.-G. (B.P. 283,118, 3.1.28. Ger., 3.1.27. Addn. to B.P. 282,805; B., 1928, 849).— $\alpha$ -Arylamides of 7-methylisatins, carrying a 5-methyl group or, in positions 4 and 5, two methyl, two chlorine, or chlorine and methyl substituents, are condensed with thioindoxyls to give violet vat dyes. The isatin anils are made by Sandmeyer's method from *m*-4-xylidine,  $\psi$ -cumidine, 6-chloro-*m*-4-xylidine, 4 : 5-dichloro-*o*-toluidine, 5-chloro-*p*-2-xylidine. C. HOLLINS.

Manufacture of brown dyes suitable for dyeing wool and leather. R. SCHMIDLIN, ASST. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,711,860, 7.5.29. Appl., 13.7.24. Ger., 23.7.23).—See B.P. 219,673; B., 1925, 538.

Manufacture of azo dyes. A. L. LASKA and A. ZIT-SCHER, ASSTS. to I. G. FARBENIND. A.-G. (Re-issue 17,306, 28.5.29, of U.S.P. 1,667,667, 24.4.28).—See B., 1928, 400.

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

Composition of jute fibre with special reference to the use of chlorine dioxide as an analytical reagent. J. K. CHOWDHURY and P. C. MAJUMDAR (J. Indian Chem. Soc., 1929, **6**, 239–251).—Successive treatment of jute fibre with a  $2\cdot5\%$  chlorine dioxide solution (Schmidt and Graumann, A., 1921, i, 912) removes almost the whole of the lignone (15%) but does not affect the raw cellulose (about 85%). The value for the lignone content agrees with that obtained using Willstätter and Zechmeister's method, whilst the cellulose content is about 10% higher than is indicated by Cross and Bevan's method. The delignified fibre is of a good tensile strength, but is disrupted by boiling with sodium sulphite or alkalis; with Schweitzer's

reagent swelling first occurs with ultimate dissolution. Removal of the hemicelluloses from the raw cellulose by washing with 17.5% sodium hydroxide solution gives about 71.5% of a-cellulose (60.6% of dry jute), which contains only small amounts of ash and furfuraldehyde. Determinations of the pentosans in raw jute and raw cellulose give values which are almost identical (about 10%), showing that they are unaffected by the chlorine dioxide treatment. Washing the raw cellulose with 5% sodium hydroxide solution removes the gummy material (13.56%), which is isolated by diluting the washings with much alcohol and subsequently acidifying with a slight excess of hydrochloric acid. Partially delignified jute (lignone content about 5%) is less susceptible to washing with alkali than the completely delignified fibre. H. BURTON.

Wood chemistry. V. Mechanical and chemical pulps from "Ezomatsu" (*Picea jezoensis*). Y. UVEDA and I. MITSUHASHI (J. Cellulose Inst., Tokyo, 1929, 5, 98—99).—The results of analyses of the original wood and of the mechanical and sulphite pulps prepared therefrom are given. D. J. NORMAN.

Wet carbonisation of wood and sulphite waste C. G. SCHWALBE (Papier-Fabr., 1929, 27, liquor. 309-311).-The production by Germany alone of about a million tons of cellulose is accompanied by the loss of roughly an equal weight of organic matter in the waste liquors. If this were used on a large scale (e.g., as a source of fuel) considerable saving would be effected. Methods are discussed whereby such recovery may be undertaken which depend on precipitation of the organic matter on absorbent material produced by swelling bark chips with magnesium chloride solution. The concentrated waste liquor is heated under pressures of about 10 atm. with water, or at lower pressure if salt solution is used. Work carried out during the war on the production of sugar and fodder from cellulose showed that on treatment of the latter with sulphuric acid at 70-80° carbonisation occurred readily. Magnesium chloride solutions may be used instead, thereby causing simultaneous swelling and hydrolysis of the material, whilst the wet distillation obtained by use of these solutions under pressure is much more rapid than the ordinary dry distillation. Carbon, acetic acid, and wood spirit are obtained as in the latter process, but no tar or From coniferous woods the yields (%) acetone. obtained by wet and dry distillation, respectively, are: carbon 55, 30 (of calorific values 6800, 8000 kg.-cal.); acetic acid 6, 2; wood spirit  $1 \cdot 2, 0 \cdot 6$ ; and wood oil -, 2%. Deciduous woods may also be used. The lower calorific value of the carbon from the wet process is of minor importance since the yields of wood spirit and acetic acid are higher. The carbon obtained may be used in the carbonisation of sulphite waste liquors in the proportion of 1 pt. of carbon to 3 pts. of concentrated liquor. Carbonisation of the waste liquor is less satisfactory if the material has previously been submitted to a process of removal of sugars, but proceeds better if the basic material present is first removed by the addition of sulphuric acid, whilst the ash content of the resulting carbon is reduced from 20-22% to 9-12%. Fuel obtained by the methods described may

be converted into briquettes which do not powder on burning. The adaptation of the methods for use on a technical scale is discussed. B. P. RIDGE.

Sclerenchema in straw cellulose. KORN (Papier-Fabr., 1929, 27, 311-314).-In an attempt to find whether the cellulose or the paper manufacturer is responsible for the appearance of sclerenchema particles in paper, experiments were carried out on samples of straw cellulose made by different processes at 12 different works. The particles were collected under standardised conditions, identified under the microscope, and counted. Out of all the samples, which had been prepared under the ordinary conditions of commercial manufacture, only one showed absence of the sclerenchema. The remainder showed numbers ranging from 9 to 93 particles per  $m^2$  of test paper; the greater the number, the greater, in general, was their size. Of the test samples, 2 were of high content, 5 of medium, and 4 of small. The presence of large or small amounts cannot be correlated with any particular process of preparation of the cellulose. Cleaning the mincers and the manner of preparation of the cooked straw must be of importance. B. P. RIDGE.

Catalytic action of selenium on calcium bisulphite solution in cellulose production. N. F. JERMOLENKO (Chem.-Ztg., 1929, 53, 343-344).-Under the conditions obtaining during sulphite cooking, free sulphur, which is always present in the cooking liquor, combines with the sulphurous acid to form thiosulphuric and polythionic acids which are themselves decomposed, yielding sulphuric acid and sulphur. Sulphuric acid is especially dangerous in this process since it causes hydrolysis and charring of the cellulose and renders it useless. Pyrites (the starting material for the preparation of the calcium bisulphite used) always contains selenium, which behaves in a similar manner to sulphur but has a catalytic action about 500 times as great. Experiments carried out in an autoclave under normal conditions of cooking, except that increasing amounts of selenium were added, show that a concentration of 0.7 mg. of selenium per litre of cooking liquid renders the cellulose unfit for papermaking after 10 hrs.' cooking at about 140°. At about 130° this concentration causes darkening of the liquor but no charring of the wood shavings used, whereas 1.4 mg./litre causes charring. The higher the concentration of selenium present the greater is the amount of free acid formed, and the greater is the calcium sulphate content of the precipitate formed and the less its calcium sulphite content, thus illustrating B. P. RIDGE. the catalytic action of the selenium.

Viscose. XXIII. Surface tension of viscose during ripening. G. KITA, S. IWASAKI, and S. MASUDA (J. Cellulose Inst., Tokyo, 1929, 5, 122-124).—According to Fukushima and Atsuki the surface tension changes during ripening in a manner similar to the viscosity but to a smaller extent, whilst Mukoyama states (B., 1927, 810) that no change occurs. The authors have examined viscose solutions prepared from three different kinds of cellulose in both 6% and 0.5% cellulose concentration by means of du Noüy's apparatus as used by Fukushima and by the stalagmometer method as used by Mukoyama. In both cases the drop number does not change-as

asserted by the latter worker-but with du Noüy's apparatus a slight fluctuation was observed except in one case. If the difficulty of measuring the surface tension of a 6% solution by means of this apparatus, and that of preparing a homogeneous 0.5% viscose solution, are taken into account, however, it is rational to assume that no change of surface tension takes place.

B. P. RIDGE. Transverse sections of artificial silk. III. The coagulating bath. I. Y. KAMI and M. NOZAKI (J. Cellulose Inst., Tokyo, 1929, 5, 117-121; cf. B., 1928, 809).-Viscose prepared by the usual process, of cellulose content 8.1-8.15%, sodium content 7-7.05%, and ripeness (ammonium chloride) 8.7-9, was spun under standard conditions into coagulating baths of different concentrations of various acids at 36.5-40°. The following properties of the threads : denier, extensibility (%), strength per denier, cross-sectional area, fullness (%), etc. were measured and correlated with the kind and concentration of acid used. Generally speaking, viscose spun from a bath containing acid alone has a circular cross-section. Concentrated acid coagulates strongly and rapidly, hence the thread is white but matt; its cross-section has many boundary fissures and a non-uniform, uneven circumference. These cracks are the smaller the more dilute is the acid, until finally they disappear and the silk has the normal lustre and good tearing strength and extensibility like the commercial material. The percentage "fullness" of the sections increases with increasing dilution of the acid. Threads spun in solutions of acids which react with the sodium in the xanthate have a greater percentage fullness than is obtained for solutions of other acids; thus the percentage fullness given by the use of acetic acid is greater than that for sulphuric, hydrochloric, or nitric acid. Relatively concentrated nitric acid baths give lustrous threads of high strength with a somewhat different cross-section. Extensibility increases, and tearing strength diminishes very slowly, with increasing B. P. RIDGE. dilution of the acid in the bath.

Analytical detection of the metal-corrosive capacity of papers. L. KALB and F. FRH. VON FALKENHAUSEN (Papier-Fabr., 1929, 27, 330-333).-The behaviour of papers placed in contact with metals depends on the content of acidic ingredients of the paper; the latter can be determined more quickly by titration or  $p_{\rm H}$  measurement than by measurement of metal-corrosive capacity. The latter has been determined for various parchment and parchment-substitute papers, and the results are correlated with those obtained by titration and  $p_{\rm H}$  experiments. Titration was carried out with standard sodium hydroxide solution on the aqueous extract obtained after steeping a sample of the paper for 24 hrs., and the number of c.c. of 0.1Nsolution used per 100 g. of paper is called its "acid coefficient." For the determination of  $p_{\rm H}$  the spotting method of Schleicher and Rössler (B., 1924, 553) was found less satisfactory than the Wulff foil colorimeter method. In the metal contact tests copper was used, since it has the advantage of showing characteristic temper colours according to the severity of attack. Strips of the paper were clamped between strips of clean copper foil and exposed in a glass vessel to a

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controlled atmospheric humidity of 90-95% until the first appearance of a colour patch corresponding with a certain stage of attack. Four such stages are identified and described which vary in intensity with the acidity of the paper and the time of treatment. The test is generally completed in 7-10 weeks. All papers showing a  $p_{\rm H}$  less than 5 by Wulff's test are injurious to metal and all of 5 or over are non-injurious. (One exception is quoted in which a paper of  $p_{\rm H}$  5.4 showed a very slight corrosive effect.) Whilst low acidity coefficients correspond, in general, with no corrosion, high coefficients do not necessarily indicate excessive corrosion effects. The corrosive capacity of sulphide sulphur has been investigated by the use of zinc sulphide. This substance alone is corrosive and its action is increased by the presence of alum; addition of zinc oxide, however, prevents such attack. Papers which contain filling material of a basic nature, in spite of having a high acidity coefficient, may on account of their small hydrogen-ion concentration be non-corrosive to metals. In these cases the titration indications are valueless and a  $p_{\rm H}$  method gives the best results. B. P. RIDGE.

See also A., June, 684, Lignin and cellulose (HESS). Cellulose benzyl ethers (NAKASHIMA). Celluloseamine and celluloseaniline (SAKURADA).

Dyeing of tissue paper. LANDOLT.—See VI. Sugar factory filter-cloths. ŠANDERA.—See XVII.

#### PATENTS.

Artificial wool. G. M. ROSSATI and G. DE BLASIO (U.S.P. 1,703,693, 26.2.29. Appl., 18.1.28).—Vegetable fibres, preferably jute or other fibre of the *Tiliaceae* family, are immersed for 2 hrs. in clear 1% line water and treated with sulphur dioxide for 1 hr. After 12 hrs.' rest the fibre is washed, dehydrated, treated with sodium hydroxide solution ( $d \ 1.075$ ), and dried. The dried fibre is washed, bleached, fixed in 0.25% sodium bisulphite solution, and, after washing in 0.5% hydrochloric acid, is immersed in a sulphonated fatty acid bath. R. BRIGHTMAN.

Aircraft covering. Gas-retaining fabric. I. M. JACOBSOHN and S. TRUSCOTT (U.S.P. 1,706,294-5, 19.3.28. Appl., [A] 11.4.25, [B] 30.9.25).—(A) A fabric treated with a mixture of regenerated cellulose and rubber dispersed in an organic solvent is claimed. (B) The fabric is treated with an aqueous solution of a soluble compound of cellulose glycollate. A. R. POWELL,

Manufacture of chloroform-soluble cellulose acetate. H. LE B. GRAY and C. J. STAUD, Assrs. to EASTMAN KODAK CO. (U.S.P. 1,690,632, 6.11.28. Appl., 6.6.27).—Cellulose is treated below 30° with less than  $2 \cdot 5$  pts. of acetic anhydride, but more than the theoretical quantity, in the presence of a catalyst, preferably sulphuric acid and phosphoric acid. The anhydride is of at least 85% concentration. R. BRIGHTMAN.

Cellulose esters containing nitro-groups and halogen-substituted acyl groups. H. T. CLARKE and C. J. MALM, ASSTS. to EASTMAN KODAK Co. (U.S.P. 1,690,621, 6.11.28. Appl., 14.5.27).—Nitrocellulose is treated with an unsaturated acid, and the mixed cellulose is halogenated. As example, nitrocellulose (11% N) in chloroacetic anhydride is treated at  $60-65^{\circ}$  with crotonic acid in presence of magnesium perchlorate, and the nitrocellulose crotonate is treated with bromine in acetic acid giving a product containing 17.8% Br and 6.8% N. R. BRIGHTMAN.

Composition containing cellulose derivatives. J. G. DAVIDSON, ASST. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,693,746, 4.12.28. Appl., 29.4.26).—Polyglycols, e.g., diethylene glycol, b.p. 250°, and triethylene glycol, b.p. 290°, are used as solvents for cellulose esters, e.g., nitrocellulose. R. BRIGHTMAN.

Treatment of fibres. A. E. MILLINGTON (U.S.P. 1,708,586, 9.4.29. Appl., 18.8.26).—Waste wood products which have been disintegrated and screened are softened by cooking in a solution of salt and soda ash with steam under pressure; the mass is refined by shredding and made into building boards.

F. R. ENNOS.

Recovery of valuable products from spent sulphite liquors. G. A. RICHTER, ASST. to BROWN CO. (U.S.P. 1,710,272, 23.4.29. Appl., 6.1.27).—Spent liquor from the digestion of cellulose material in a sulphurous acid solution of ammonium salts is neutralised with basic calcium phosphate and, after addition, if desired, of potash in a suitable form, is evaporated, and the dry residue containing all the organic matter removed from the cellulose material is used as a fertiliser. F. R. ENNOS.

Wood fibre for high-grade paper and derivative manufacture. G. A. RICHTER, ASST. to BROWN CO. (U.S.P. 1,709,322, 16.4.29. Appl., 7.11.24).—By suitable treatment of unbleached sulphite pulp, a cellulose wood pulp of high whiteness is produced containing  $93\cdot5-96\%$  of  $\alpha$ -cellulose, 2-4% of  $\beta$ -cellulose, 2-3% of  $\gamma$ -cellulose, not more than  $2\cdot4\%$  of pentosans, and giving a copper number of  $1\cdot0-2\cdot3$ , a reddishpurple colour when subjected to Herzberg fibre stain, and a solubility of not over 9% in 3% boiling sodium hydroxide. F. R. ENNOS.

Sizing of paper. CONTINENTALE A.-G. F. CHEMIE, Assees. of A. ILLOCH (G.P. 454,005, 28,1.26).—An emulsion obtained by the saponification of pinabietic acid is used. *E.g.*, the acid is saponified by means of sodium hydroxide or carbonate solution, and the mixture, after being boiled for about  $\frac{1}{2}$  hr. until free from acid crystals, is poured into water and forms a rosin milk.

B. P. RIDGE.

Liquidation of natural silk threads. T. MUTO and S. HIDA, ASSIS. to KANEGA-FUCHI BOSEKI KABUSHIKI KWAISHA (U.S.P. 1,714,039, 21.5.29. Appl., 21.2.28. Japan, 15.12.27).—See B.P. 306,699; B., 1928, 352.

Production of textile fibres of high quality from skins of fish such as, in particular, those of the *Chondropterygii selachii*. A. EHRENREICH (U.S.P. 1,713,036, 14.5.29. Appl., 24.1.28. Belg., 27.1.27).— See B.P. 284,297; B., 1928, 668.

Industrial treatment of coconuts and their constituents, particularly coconut fibres. B. G. H. VAN DER JAGT, ASST. of Z. VAN DER BERGH and F. A. J. VAN KUYK (U.S.P. 1,713,681, 21.5.29. Appl., 29.1.26. Holl., 2.2.25).—See B.P. 246,837; B., 1927, 472.

[Mechanical] preparation of flax, hemp, and like fibrous materials for spinning. LINEN INDUSTRY Res. Assoc., and J. A. MATTHEW (B.P. 311,883, 30.3.28)

[Pumping] apparatus for use in manufacture of artificial silk or other operations in which liquids are supplied under pressure. BRIT. CELANESE, LTD., and E. KINSELLA (B.P. 311,670, 9.2.28).

Drying paper or the like in the form of continuous webs, and papermaking machinery therefor. E. A. Ohlin (B.P. 285,914, 23.2.28. Swed., 26.2.27).

Emulsifiable oil (U.S.P. 1,695,197).—See II. Wood fibre-asbestos product (Swiss P. 122,820).—See IX. Oils for fabrics, paper, etc. (B.P. 311,786).—See XII.

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

Effect threads. J. G. GRUNDY (J. Soc. Dyers and Col., 1929, 45, 142—148).—A tabular synopsis is given classifying the properties of the main groups of dyes suitable for the production of effect threads on mixed fibres. These tables include lists of acid, Neolan, and chrome colours dyed on wool with resists on cotton, viscose, passivated and immunised cotton, acetate silk, and silk, together with direct colours dyed on cotton with resists of wool, silk, acetate silk, and passivated and immunised cotton. Formulæ are also given for dyeing the various classes of dyes in order to obtain the best resist with various effect threads.

# L. G. LAWRIE.

Dyeing of tissue paper with acid dyes fast to water. A. LANDOLT (Papier-Fabr., 1929, 27, 357-362). -Direct dyes on tissue paper give, as a rule, dull shades, whilst acid dyes, although bright and fast to light, in many instances give dyeings which are not fast to water. Many acid dyes, however, can be made fast to water by dyeing the paper by the dipping method and aftertreating the dyed paper with aluminium acetate. The amount of the acetate to be added should be sufficient to ensure fixation of the dye, but insufficient to cause precipitation of dyestuff in the dyebath; suitable proportions are: 10 g. of dyestuff, 400 c.c. of 5% aluminium acetate solution, and 1 litre of water. Lake formation can be prevented with many dyes by increasing the hydrogen-ion concentration by the addition of 20-50 c.c. of acetic acid per litre of dyebath. Suitable dyes are Kiton Yellow S, Kiton Yellow 3GN, Paper Red PSN, Benzyl Red B, Brilliant Benzyl Violet 3B, Cloth Fast Brilliant Red 4B, Pure Blue BSI, Benzyl Green B, and Acid Black HA. The fastness to water is tested by soaking the dyed paper in distilled water for 10 min. and comparing the result with an untreated portion. A dyeing fast to water should neither change in shade nor stain the water. The handle of paper treated with aluminium acetate is somewhat firmer.

# L. G. LAWRIE.

See also A., June, 641, Adsorption of soap solutions (NONAKA; MIKUMO). 645, Diffusibility and dispersity of dyes (PISCHINGER). 671, Device for identifying colours (HOLMES).

Comparison of colours. TOUSSAINT.—See I. Dyewood extracts. BRECHT.—See XV.

# PATENTS.

Weighting of natural silk. I. G. FARBENIND. A.-G. (F.P. 631,082, 17.3.27).—In the usual tin phosphatesilicate process, phosphate solutions produced from pure phosphoric acid are used. B. P. RIDGE.

Manufacture of softener for silk. R. E. Rose, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,691,994, 20.11.28. Appl., 17.11.25).—Diarylguanidine salts of sulphonated castor oil are used as softeners or lubricants for weighted silks. C. HOLLINS.

Cross-dyeing of cellulose fabric. J. C. WATSON, Assr. to B. B. & R. KNIGHT CORP. (Re-issue 17,295, 14.5.29, of U.S.P. 1,629,769, 24.5.27).—See B., 1927, 553.

Fluid treatment of artificial silk threads or filaments. J. BRANDWOOD (B.P. 311,803, 17.2.28).

Bleaching of fatty acids (G.P. 454,308). Bleaching of fish oils (B.P. 311,665).—See XII.

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

Recent developments in the manufacture of sulphuric acid. S. F. SPANGLER (Ind. Eng. Chem., 1929, 21, 417-421).—The following developments and tendencies are noted. (1) The introduction of a mixing nozzle whereby anhydrous ammonia can be discharged into water, thus allowing ammonia for oxidation units to be transported anhydrous and used as liquor. (2) The development of the two-stage submerged-pipe method of concentrating sludge acid from oil refineries. Cottrell precipitators are used with silicon-iron tubes. (3) The introduction of the vanadium mass in contact plants: eight plants to use the improved "Selden mass" are now under construction in America. They incorporate a new type of sulphur burner burning molten sulphur and having a centrifugal air-blower which provides all the draught required. The sulphur dioxide heats a steam boiler before entering the converter; the sulphur trioxide is cooled and passed through three absorbing towers, through the first two of which acid is circulated, whilst the last is a filter. This plant is considered likely to produce 95% acid at a lower cost than any other type. C. IRWIN.

Determination of the purity of potassium and sodium ferrocyanides by titration with zinc sulphate solution. FARBSALZ-GES.M.B.H. (Chem.-Ztg., 1929, 53, 399).—A solution of 1 g. of the salt in 150 c.c. of water and 10 c.c. of 0.1N-sulphuric acid is titrated at  $15-20^{\circ}$  with 0.2N-zinc sulphate solution, the end-point being determined by means of a spot test on filter paper impregnated with ferric ammonium sulphate solution; no blue colour should develop in 2—3 min. The solution should be standardised against the corresponding pure salt as the titre with potassium ferrocyanide is not the same as that with the sodium salt.

A. R. POWELL

Recovery of bromine from sea water. C. M. A. STINE (Ind. Eng. Chem., 1929, 21, 434-442).—In the recovery of bromine from sea water (which contains 60-70 p.p.m. of bromine) as tribromoaniline the reaction is  $3Br^{-} + 3Cl_2 + PhNH_2 = C_6H_2Br_3.NH_2 +$  $3H^{+} + 6Cl^{-}$ . Chlorine in sea water at this concentration

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is converted into hypochlorous acid to the extent of 73%, but by the addition of 200 p.p.m. of sulphuric acid this hydrolysis is reduced to 15% at 25°. The aniline is used in the form of the sulphate. The mixing of the acidulated chlorine solution with the aniline salt solution must be as rapid as possible in order to avoid oxidation losses. An experimental shore plant treating 25 gals./min. in a continuous pipe-line plant gave yields of 40-50%, the tribromoaniline being largely contaminated with sand. This latter point determined the further development of the process on board ship. The s.s. Ethyl was fitted up to treat 7000 gals. of sea water per min. Sulphuric acid and aniline were stored in steel tanks and handled by means of compressed air, liquid chlorine in 1-ton drums. The mixing devices used included large injection rings and turbulence chambers. The chlorinated water was handled by rubber-lined pumps and tanks. Aniline was converted into aniline sulphate solution (15%) in a lead-lined tank daily, and further diluted by the recirculation of one third of the treated water. The total time of passage of the sea water through the three mixing systems was 10 sec. It was then filter-pressed. The process depends on accurate feed control, and certain difficulties were experienced on the first and only voyage through interference with this by corrosion. It is, however, believed that these could be readily remedied. C. IRWIN.

See also A., June, 626, Phosphorescent calcium tungstate (Schleede and TsAo). 650, System potassium perchlorate-sodium nitrate or chloridewater. System sodium nitrate-sodium chloridepotassium perchlorate-water (CORNEC and NEU-MEISTER). 662, Phosphorescent alkaline-earth compounds (VANINO and SCHMID). 666, Ultra-violet colorimetry and fluorescent substances (EISEN-BRAND). 667. Determination of phosphoric acids (SOMEYA; STOLLENWERK and BÄURLE; DWORZAK and REICH-ROHRWIG). 668, Determination of arsenic (VILLECZ; GNESSIN). Test for boric acid and borates (DODD). Detection of alkali metals in salts (TANANAEV). Determination of metals as sulphates (GUNTZ and BARBIER). Detection of potassium (REED and WITHROW). Determination of beryllium in rocks (DIXON). 669, Volumetric determination of thallous salts (JILEK and LUKAS). 672, Containers for caustic solutions (Soule). 713, Determination of halogens in organic substances (ROMAN).

Stainless iron nitric acid equipment. MITCHELL. —See X. Analysis of three-component systems. TAUFEL and RUSCH.—See XII. "Kalkammon." NEHRING. Acidimetric titration of silica. KÜHN.— See XVI. Germicidal washing solutions. MYERS.

#### PATENTS.

Manufacture of sulphur trioxide and sulphuric acid. R. TERN (B.P. 311,862, 8.3.28).—Nitrogen oxides produced from air and formed on non-conductors at the end of current conductors, by the action of high-tension currents or fields, are employed to effect the catalytic oxidation of sulphur dioxide to sulphur trioxide under the action of similar currents or fields. Steam and warm air may be admitted to the oxidation chamber, and the treated gas mixture may be sucked into an electric filter. J. S. G. THOMAS.

Production of nitric acid. C. C. SMITH, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 311,553, 10.5.28). —Nitrogen oxides, preferably under pressure, from the oxidation of ammonia are absorbed in dilute nitric acid, the resulting acid is concentrated by indirect contact with the hot burner gases, and the condensate (2—3% nitric acid) is used for further absorption, which may be brought about by injecting the acid into the hot burner gases before or after their passage through the heatexchanger of the evaporator. W. G. CAREY.

Synthetic production of hydrochloric acid. W. HIRSCHKIND and C. W. SCHEDLER, ASSTS. to GREAT WESTERN ELECTRO CHEM. Co. (U.S.P. 1,695,552, 18.12.28. Appl., 14.9.25).—Chlorine and steam are passed through hot carbon in approximately the proportion required by the equation:  $C + 2H_2O + 2Cl_2 = 4HCl + CO_2$ , a ratio of 7—9 cub. ft. of carbon to 100 lb. of chlorine entering being maintained in the reaction zone.

R. BRIGHTMAN. Calcination of alkaline-earth materials. W. CROW, ASST. to DITTLINGER CROW CO. (U.S.P. 1,710,967, 30.4.29. Appl., 5.3.23).—Alkaline-earth carbonates are treated with superheated steam (100 lb./in.<sup>2</sup>) at 320°, then subjected to the action of air sufficiently heated to decompose the hydroxide formed. H. ROYAL-DAWSON.

Manufacture of beryllium oxide. R. C. PRICE and H. S. COOPER, Assrs, to BERVILIUM CORP. OF AMERICA (U.S.P. 1,710,840, 30.4.29. Appl., 13.12.23).— Beryllium mineral is heated with a flux which includes a calcium compound, sulphuric acid is added, and the product is dehydrated, dissolved in water, evaporated to d 1.31, and the precipitated calcium sulphate separated. W. G. CAREY.

Production of titanic oxide. L. E. BARTON and C. J. KINZIE, ASSIS. to TITANIUM PIGMENT Co., INC. (U.S.P. 1,695,270, 18.12.28. Appl., 16.2.25).—Titaniferous ores are heated with sulphuric acid and nitre cake (or other alkali sulphate) at 200—350°, the titanic oxide and sulphates are dissolved out, and the former is separated in known manner. R. BRIGHTMAN.

Production of lead arsenate. A. F. MEYERHOFER (Swiss P. 122,353, 22.7.24. Ger., 20.12.23).—The compound is formed by the interaction of lead fluosilicate and, *e.g.*, calcium arsenate in accordance with the equation:  $3PbSiF_6+2CaHAsO_4=Pb_3(AsO_4)_2+2CaSiF_6+H_2SiF_6$ . L. A. COLES.

Treatment of phosphorites with a low phosphoric acid content. E. ORLOV (Russ. P. 3464, 31.8.27).—A suspension of the phosphorites in water or dilute ammonia is treated with gases containing sulphur dioxide and, after filtration, the solution obtained is heated to expel excess sulphur dioxide and to cause separation of calcium and ammonium phosphates, sulphates, and sulphites. L. A. COLES.

Base-exchange body. A. O. JAEGER, Assr. to SELDEN RES. & ENG. CORP. (U.S.P. 1,694,620, 11.12.28. Appl., 28.2.27).—Substances, obtained by base-exchange methods, which are free from combined silicon and contain catalytically active elements in a non-exchangeable form and at least one exchangeable base other than alkali metal are used as catalysts, *e.g.*, for organic oxidations, hydrogenations, etc. Examples are given of potassium vanadite, tungstate, chromate, molybdate, or tantalate, treated with, *e.g.*, vanadyl sulphate, in presence or absence of diluent siliceous material, infusorial earth, or of ferric, silver, or copper oxides, base-exchange reaction taking place in neutral, acid, or alkaline solution. Many other examples are given. R. BRIGHTMAN.

Catalytic agent and its manufacture. W. A. PATRICK, ASST, to SILICA GEL CORP. (U.S.P. 1,695,740, 18.12.28. Appl., 5.5.21).—Silica gel which has not been completely dried is impregnated with a suitable solution (e.g., ammonium chloroplatinate, ferric acetate) and dried at high temperature. C. HOLLINS.

Preparation of moisture-absorbing material. W. R. ORMANDY, T. J. I. CRAIG, and P. SPENCE & SONS, LTD. (B.P. 311,578, 15.6.28).—An absorbent mass which can be many times revived at comparatively low temperatures is formed from aluminous silicates in lump form, which are treated with mineral acid to remove most of the alumina, a hard porous mass being left after extraction and washing. This mass is impregnated with a solution of an absorbent (e.g., calcium chloride) and heated till practically all the solvents used are evaporated. Calcination may be employed before or after treatment with mineral acid. B. M. VENABLES.

Production of hydrogen. J. S. BEEKLEY, Assr. to LAZOTE, INC. (U.S.P. 1,711,036, 30.4.29. Appl., 10.9.26). —Oxygen and a reducing gas are added to a mixture of steam and a gaseous hydrocarbon, and the mixture is passed over a heated catalyst. W. G. CAREY.

Production of hydrogen and gases containing hydrogen. K. GORDON, and IMPERIAL CHEM. INDUS-TRIES, LTD. (B.P. 311,299, 31.1.28).—A process is described for removing carbon dioxide from mixtures with hydrogen, carbon monoxide, etc. The gases are scrubbed with water under pressure (e.g., 200 atm.). Partial release of the pressure (to 20 atm.) separates gases richer in hydrogen, leaving in solution gases richer in carbon dioxide. The process may be repeated to effect further separation. J. A. SUGDEN.

Liquefying and separating the constituents of gaseous mixtures at low temperatures. L'AIR LIQUIDE Soc. ANON. POUR l'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 287,558, 15.3.28. Fr., 25.3.27. Addn. to B.P. 281,675; B., 1928, 603).-Oxides of nitrogen are removed from gas mixtures prior to liquefaction by hydrogenating them. The process is carried out at a temperature between 100° and 200° (or at any rate below a moderate upper temperature) in the presence of a catalyst such as reduced copper or reduced iron, and is effected when the proportion of oxides of nitrogen is very small relatively to the hydrogenating constituents present. Acetylene will also become hydrogenated under similar conditions. Another method of removing the oxides of nitrogen is to wash the gas mixture with a hot solution of ammonium sulphate, with the liberation of nitrogen and formation of sulphuric acid.

B. M. VENABLES.

**Production of sulphur.** A. P. THOMPSON, Assr. to GEN. CHEM. Co. (U.S.P. 1,695,068, 11.12.28. Appl., 16.12.26).—Sulphur is recovered from gases containing sulphur dioxide by reducing the latter with carbonaceous fuel in presence of a controlled amount of carbon dioxide at temperatures sufficiently high for the endothermic reduction of carbon dioxide to carbon monoxide by the fuel to take place. The endothermic reduction prevents the temperature rising high enough to fuse the ash and cinder. The exit gas may be used in part as source of carbon dioxide. R. BRIGHTMAN.

Manufacture of aluminium oxide from aluminium sulphide. C. von GIRSEWALD (U.S.P. 1,713,411, 14.5.29. Appl., 28.2.28. Ger., 4.3.26).—See B.P. 294,079; B., 1928, 710.

Brine evaporation (B.P. 309,104-6). Antifreezing composition (U.S.P. 1,711,324). Conveyance etc. of liquid gases (B.P. 280,569).—See I. Spent sulphite liquors (U.S.P. 1,710,272).—See V. Fertilisers (Norw. P. 42,724 and Swiss P. 122,821).—See XVI.

# VIII.—GLASS; CERAMICS.

Clays. XIII. Flocculative action of hydrophilic sols on suspensions of clays and its practical uses. T. OKAZAWA and T. SANO (Bull. Inst. Phys. Chem. Res., Tokyo, 1929, 8, 356-368).-Suspensions of clay are readily flocculated by the addition of small quantities of hydrophilic colloids. The flocculation values for casein, albumin, gum arabic, and starch are small, gelatin has a high value, but the most effective agent is a sol of "Konnyaku-ko" (a powder obtained from the tuberous root of the devil's tongue), which precipitates clay suspensions when present at a concentration of only 2 p.p.m. An acid suspension of clay is more susceptible to flocculation than an alkaline suspension. Suspensions of charcoal in water are difficult to flocculate by hydrophilic colloids, but are readily flocculated thereby after addition of a small amount of clay. Clay particles in non-aqueous media also are flocculated by hydrophilic colloids. The precipitated particles are much coarser than those flocculated by electrolytes, and the method is therefore applicable to the acceleration of filtration in industrial processes. E. S. Hedges.

See also A., June, 637, System silica and its stability regions (VAN NIEUWENBURG). 650, Ceramics of highly refractory substances (RUFF and others). 668, Detection of alkali metals in silicates (TANA-NAEV). 669, Detection of magnesium in silicates (LEITMEIER and FEIGL). 671, Calibration of Lovibond glasses (PRIEST and others).

#### PATENTS.

Continuous-tunnel kiln. T. G. McDougal, Assr. to AC SPARK PLUG Co. (U.S.P. 1,710,995, 30.4.29. Appl., 11.3.25).—The ware passes through a zone of steady graduated temperature directly heated by the counterflow of combustion gases. J. A. SUGDEN.

Continuous kiln. H. R. STRAIGHT, Assr. to E. H. STRAIGHT (U.S.P. 1,711,910, 7.5.29. Appl., 28.9.26).— The upper corners of the stack of ware on the truck fit into longitudinal grooves in the arch of the kiln. Adjustment for the track is provided. J. A. SUGDEN. **Refractory.** J. M. LAMBIE and D. W. Ross (U.S.P. 1,712,005, 7.5.29. Appl., 1.2.24).—Sufficient finelydivided cyanite is added to counteract the firing shrinkage of the other ingredients. J. A. SUGDEN.

Manufacture of dense and solid articles or appliances of zirconia or other highly-refractory oxides. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (B.P. 292,529, 5.4.28. Ger., 21.6.27).—To the refractory oxide is added 5% of magnesia and a solution of a salt of the refractory oxide to the extent of the equivalent of 5% of the magnesia. J. A. SUGDEN.

Manufacture of translucent, hollow glass articles, particularly of milk-glass bulbs, for electric incandescence lamps. M. PIRANI, Assr. to PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN (U.S.P. 1,713,394, 14.5.29. Appl., 8.3.28. Ger., 24.3.27).—See B.P. 287,545; B., 1929, 55.

# IX.—BUILDING MATERIALS.

Effect of temperature on the setting times of cements, and on the strength of cements, mortars, and concretes. W. W. THOMAS and N. DAVEY (Dept. Sci. Ind. Res., Bldg. Res., Spec. Rept. No. 13, 1929, 38 pp.).-The setting of Portland cement is invariably accelerated by rise in temperature, but there is no direct connexion between the actual times and the variations. Rapid-hardening cements were found to behave similarly. One exception may be due to consistency changes in the wet mix rather than to setting. Aluminous cements which evolve heat on setting have given conflicting results. A slight reduction of the strength of concrete with rise of temperature during setting is perhaps due to increased internal stresses. The strength obtained by curing for a given time at  $1.5^{\circ}$  was found to be only half that with the same treatment at 16°. Rapid-hardening cements behave similarly. Laboratory tests with aluminous cements again show a less strength at low temperatures, though thermal effects make comparison with large-scale work difficult. If only the earlier part of the treatment of any cement is at low temperature, full strength may or may not be attained. Different results have been recorded as to whether any hardening occurs below 0°. In most cases a period of freezing during curing slows down the rate of increase of strength when the temperature has again become normal. This applies also to aluminous cement. There is some evidence to show that the effect of freezing is more severe the earlier after mixing it occurs. Experimental data on alternate freezing and thawing are uncertain. Increased cement or water content of mix increases the damage to Portland cement concrete, and probably to aluminous cement concrete. There is some evidence to show that unduly high temperatures may cause failure with aluminous cement. C. IRWIN.

Hydraulic moduli and resistance limits of cement. S. GIERTZ-HEDSTRÖM (Svensk Kem. Tidskr., 1929, 41, 119—124).—A review of the various formulæ which have been employed to obtain a "hydraulic modulus" for Portland cement. In the case of hydr aulic slags a new modulus is proposed, calculated from the amounts of the components of the slag which are attacked and unaffected by water respectively. If the slag is to be hydraulic, the ratio between the two groups must be greater than 1, *i.e.*,  $(CaO+MgO+CaS+SO_3+$ ......)/(SiO<sub>2</sub>+Al<sub>2</sub>O<sub>3</sub>+FeO+MnO+ . . .)>1. It is claimed that the above formula affords a better measure of the hydraulic value of the slag than the one usually employed, *i.e.*,  $(CaO+MgO)/(SiO_2+Al_2O_3)$ .

#### H. F. HARWOOD.

# PATENTS.

Manufacture of light-weight concrete. R. CROSS and W. A. COLLINGS, ASSTS. to SILICA PRODUCTS CO. (U.S.P. 1,710,921, 30.4.29. Appl., 25.3.25).—A hydraulic cement and mineral filler are mixed with gelatinising clay, the last-named serving to hold the aggregate in suspension. J. A. SUGDEN.

Treatment of wood. J. MAXIMOFF and M. S. DE COSTA (F.P. 629,558, 6.5.26).—Gases and liquids for drying, extracting, or impregnating wood are charged into the wood in the direction of the natural channels in it, so that the processes are effected rapidly. The apparatus is so devised that each piece of wood can be treated at a desired temperature and pressure.

L. A. Coles.

Constructon of road and like surfaces. C. W. SHARROCK (B.P. 311,751, 16.2.28).—Suitably-graded aggregate mixed with powdered bitumen, with or without the addition of an oil, is laid cold on the surface of the road, and then hot-rolled at 120—180°.

H. ROYAL-DAWSON.

Product containing wood fibre and asbestos. J. FAHRNI (Swiss P. 122,820, 5.6.26).—Purified asbestos fibres are treated in a bath containing powdered metallic oxide, tannin, water-glass, formaldehyde, and ammonium chloride, and wood fibre in one containing ammonium chloride, magnesium chloride, boric acid, and sulphitecellulose waste liquor. After squeezing or filtration, the fibres are mixed and pressed into sheets. The product is useful as insulating or building material.

B. P. RIDGE.

Production of hydraulic cement composition. L. Forsén (U.S.P. 1,712,818, 14.5.29. Appl., 3.6.26. Finland, 24.10.24).—See B.P. 272,163; B., 1927, 909.

Preservation of timber etc. A. M. KOBIOLKE (U.S.P. 1,713,388, 14.5.29. Appl., 27.7.27. Austral., 10.8.26).—See B.P. 295,126; B., 1928, 713.

Brick kilns. A. HABLA (B.P. 311,884, 30.3.28).

[Fixing of] roof, wall, and other surface coverings of corrosion-proof metals or alloys, especially copper. METALLGES. A.-G. (B.P. 292,621, 23.6.28. Ger., 23.6.27).

Fluid-storing material (U.S.P. 1,705,482–4).— See I. Treatment of fibres (U.S.P. 1,708,586).— See V.

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

Corrosion in cast-iron main pipes. J. R. BRAD-SHAW (Gas J., 1929, 186, 593-596).—Eight samples of cast-iron pipes from water and gas mains, under varying conditions of time and soil, were examined inside and out for characteristics of fracture, deposit, corrosion, pitting, wear, etc. Analyses of inside and outside deposits showed essentially brown hydrated ferric oxide with varying amounts of ferrous oxide, and for gas mains carbonaceous and organic matter. Outside corrosion was typical of electrolytic soil action and the product was ferrous oxide. Microstructure examination indicated corrosion emanating from graphite nuclei, whilst in several cases polishing started an etching attack round the graphite areas. The varying amounts of corrosion in the samples show the necessity of obtaining a standard quality of high-density iron which will give an almost indefinite life when buried underground. For this purpose spun pipe may be found preferable. The influence of cold weather on tensile strength and the stress and strain on cast-iron pipes due to modern traffic are subjects which should not be ignored.

C. B. MARSON.

Corrosion—a problem in protective coatings. F. N. SPELLER (Ind. Eng. Chem., 1929, 21, 506-510).-Observations on failures in large pipe systems, e.g., gas services, indicate that there is little difference in the rate of corrosion of steel and iron in soil, and that the method of manufacture or the quantity of foreign inclusions within wide limits has little influence. Copper in excess of 0.15% in steel appears to exert a protective effect towards exposure to air not evident with any other additional elements. For the protection of underground pipe paints have little value, but linings of Portland cement or bitumen reinforced with sand are used extensively, often being applied by centrifugal action. Where cost is not of primary importance, enamelled or rubber-lined pipes are available, or more resistant metals may be used, particularly an alloy containing 18% Cr and 8% Ni. C. A. KING.

Metallic materials of construction for chemical engineering equipment. E. P. PARTRIDGE (Ind. Eng. Chem., 1929, 21, 471-476).-Alloys of iron with high chromium content have proved resistant at high temperatures to the influence of sulphur gases and to scaling; in the artificial silk industry the action of mixed acids has been uneven. Chromium-nickeliron alloys (e.g., Staybrite steel) are used increasingly in high-pressure oil-cracking stills and dairy and preserved food equipment, as are also aluminium and nickel containers. For resistance to acids a group of alloys which might be classed as bronzes is available, and a high-zinc alloy, Zilloy, has recently been developed for roofing the construction of industrial plants. Coated metals possess advantages in certain fields, but suffer from the fundamental disadvantage that even a minute fracture in the coating may cause the ruin of a C. A. KING. whole equipment.

Stainless iron nitric acid equipment. W. M. MITCHELL (Ind. Eng. Chem., 1929, 21, 442-445).-Stainless irons resistant to nitric acid contain 16-20% Cr and under 0.10% C. They are suitable for all synthetic nitric acid plant, but are attacked by the halide acids present in Chili saltpetre. Riveting presented difficulties at first owing to the brittleness of the alloy if overheated, but the technique is now understood. Riveting is, however, only done in the shop, which limits the size of absorption towers etc. Welding is usually excluded owing to the risk of local corrosion following recrystallisation. Forgings and castings (using metal with 0.3-0.4% C) are quite satisfactory. If welding is unavoidable, 7-10% of nickel should be added. The construction of drums in such alloys for nitric acid transport is still experimental. C. IRWIN.

Welding in the chemical and process industries. W. SPRARAGEN (Ind. Eng. Chem., 1929, 21, 425-431).— Methods of welding using thermite or oxyacetylene and various methods of electrical welding are described. The steel used should be selected with reference to the requirements of the particular case, but that with carbon content below 0.25% is the most easily handled. Greater strength is obtained by welding parts designed for welding than by welding a lap joint intended for riveting. The need for adequate supervision of welding work is referred to, and developments in the welding of stills, pipelines, etc. are mentioned. C. IRWIN.

Hydraulic classification of minerals. G. Bozza (Giorn. Chim. Ind. Appl., 1929, 11, 151—154).—The conditions to be maintained in an ore classifier with an ascending water current so as to obtain perfect classification are deduced, these conditions corresponding with those on which Fahrenwald's controlled-density classifier (B., 1928, 695) is based. Experimental values obtained show that neither the theory advanced by Fahrenwald nor that usually assumed concerning the relation between fall in mass and free fall represents the actual facts of the case. T. H. POPE.

Oxygen in iron and steel. II. P. OBERHOFFER, H. HOCKSTEIN, and W. HESSENBRUCH (Arch. Eisenhüttenw., 1928-9, 2, 725-738; Stahl u. Eisen, 1929, 49, 799-800; cf. B., 1927, 966).-The properties of certain alloy steels prepared in the normal way have been compared with similar steels prepared from iron which has been subjected for 1 min. to an oxidising blast. Oxidised vanadium and chromium steels are very viscous when molten, whereas nickel and tungsten steels are quite mobile. All the oxidised steels have a coarse-grained structure and the nickel steel is distinctly hot-short due to the presence of oxide in solid solution. Addition of chromium offsets slightly the presence of oxygen. Vanadium and aluminium are such strong deoxidising agents that they remove most of the oxygen from the steel, and their oxides, if retained in the metal, are so pliable that they do not affect the hot-working properties. The presence of oxygen in alloy steels hinders greatly the diffusion of carbon during cementation and the resulting structure is fine and hackly; it also narrows the hardening range and intensifies the effect of overheating.

A. R. POWELL.

Use of nickel in grey iron castings. G. BRODSKY (J. Sci. Instr., 1929, 6, 168—169).—Addition of 2.5% of nickel to iron gives castings free from blow-holes and sponginess, especially suitable for hydraulic parts. C. W. GIBBY.

Self-deforming zinc alloys. L. LOSANA (Notiz. chim.-ind., 1927, 2, 616—622; Chem. Zentr., 1928, I, 1092—1093).—The changes that occur in the structure during ageing of zinc alloys containing aluminium and copper or aluminium and tin have been investigated.

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Alloys with more than 3-4% Al undergo appreciable deformation on keeping at the ordinary temperature, especially when the castings are rapidly cooled. Copper accelerates the change and tin retards it. The deformation appears to be due to decomposition of the  $\beta$ zinc-aluminium solid solution and to an allotropic change in the  $\eta$ -zinc-copper constituent. These changes may continue for several years before reaching equilibrium. The completely stabilised alloys exhibit different anomalies on the dilatometric curve from those of the freshly cast alloys. A. R. POWELL.

Risk of poisoning when working with mercury. O. STELLING (Svensk Kem. Tidskr., 1929, 41, 80-85).-The risk of contracting mercurial poisoning when work is carried on for long periods in rooms where this metal is exposed to the air has been pointed out by Stock (A., 1926, 707). In its earlier stages the disease is difficult to diagnose, as the symptoms closely resemble those met with in neurasthenia, but recourse should be had to examination of the urine and faces, which contain small quantities of mercury if poisoning by that metal is in question; the air of the room should also be tested, using the colorimetric method with diphenylcarbazide, which permits of the detection of  $7 \times 10^{-6}$  mg of mercury. The presence of only a few thousandths of a mg. of mercury per m.<sup>2</sup> of air can give rise to chronic mercurial poisoning. The question as to how far the amalgams used in dentistry can give rise to mercurial poisoning is still undecided, but there seems little doubt that the copper amalgam formerly employed for fillings is definitely injurious to health.

H. F. HARWOOD.

Electrolytic recovery of metals. G. EGER (Z. angew. Chem., 1929, 42, 518-522).—A lecture before the Rhenish-Westphalian local section of the German Chemical Society describing the principles and methods used in the recovery of metals by electrolysis.

A. R. POWELL.

Electroplating of cadmium from cyanide baths. L. R. WESTBROOK (Amer. Electrochem. Soc., May, 1929. Advance copy. 14 pp.).-In a commercial type of bath containing the double cyanide, NaCd(CN)<sub>2</sub>, free sodium cyanide, sodium hydroxide and sulphate in the equivalent proportions 1:2:1.25:1.25, increasing the total concentration increases the conductivity and stability of the bath, raises the cathodic current efficiency, throwing power, and maximum current density for satisfactory plates, and gives a smoother and finergrained deposit. Free cyanide keeps anodic polarisation low and anodic current efficiency high by preventing film formation on the anode, and also increases the smoothness and brightness of the deposit. Sodium hydroxide opposes the tendency of free cyanide to increase cathodic polarisation, whilst the tendency of the hydroxide to increase anodic polarisation is balanced by the opposite tendency of the cyanide. Increasing the sodium hydroxide concentration increases the cathodic current efficiency, the conductivity, and the brightness and ductility of the deposit. Sodium sulphate hardly affects the electrical properties of the bath, but seems to render it more stable. The cadmium content of the bath is maintained at a suitably high value

(generally about 40 g./litre) by using cadmium anodes of greater surface area than the cathodes and by maintaining sufficient free cyanide in the bath. Sodium cyanide and hydroxide are added at intervals to make up for losses due to hydrolysis and absorption of carbon dioxide, but the sodium carbonate so formed has practically no effect on the electrical properties of the bath nor on the character of the cathodic deposit if the cyanide and hydroxide concentrations are kept up. Of metals which act as "brighteners," nickel is most effective and 0.2-0.3 g./litre is commonly used. This addition of nickel (as sulphate) increases the brightness, ductility, and hardness of the deposit and greatly increases the maximum cathodic current density at which satisfactory plates can be obtained. Also impurities such as arsenic and antimony, which have a very bad effect on the deposit, are much less harmful in the presence of nickel. The effects of organic addition agents are discussed and the composition and conditions of operations of two typical cadmium-plating baths are H. J. T. Ellingham. given.

Electrical resistance method of measuring corrosion of lead by acid vapours. R. M. BURNS and W. E. CAMPBELL (Amer. Electrochem. Soc., May, 1929. Advance copy. 15 pp.).—A method is developed for measuring the rate of corrosion of a metal in terms of the rate of increase in resistance of a wire as it is reduced in cross-section by the progress of corrosion (cf. Hudson, B., 1928, 488). The accuracy and reproducibility of the method were determined by experiments with lead wires exposed to the vapour in equilibrium with dilute acetic acid of various concentrations. The percentage increase in resistance is a linear function of the time, and duplicate tests are reproducible to an accuracy of 15%. In order to compare the corrosive properties of green Douglas fir with those of other woods which might be used to replace it in the construction of conduits to carry lead-sheathed cables, the variation in the resistance of lead wires exposed to the vapour in equilibrium with damp sawdust from various woods was examined. Raw Douglas fir is found to be more corrosive than yellow pine or hemlock, and acidity determinations on the woods are in agreement with these results. Kiln drying does not reduce the corrosive action of the fir, but creosoting causes a notable reduction. The rate of corrosion of lead in acetic acid vapour falls off rapidly when the specimen is removed into the vapour of distilled water, and still more rapidly when put into the vapour from 1% ammonia solution. The rates of corrosion of two cable-sheath alloys and of pure lead in the vapour of 0.02M-acetic acid were compared, and the method was also applied to the examinations of the corrosion of lead in nitric acid vapours.

H. J. T. ELLINGHAM.

See also A., June, 631, Internal stress in carbon steels (SERITO). 668, Determinations of metals as sulphates (GUNTZ and BARBIER). 669, Electrolytic determination of lead (TÖPELMANN). 671, Determination of tin by electrolysis (Ŝvéda and UZEL). Separation of niobium and tantalum (YNTEMA). Separation of ruthenium and osmium, and titration of osmium (SAITO). Metal-corrosive papers. KALB and VON FALKEN-HAUSEN.—See V.

PATENTS.

Salt-bath furnace. A. G. E. HULTGREN (U.S.P. 1,713,472, 14.5.29. Appl., 17.5.28).—A non-volatile acid oxide, preferably silica, is introduced into a fused salt bath for hardening steel to prevent decarbonisation. C. A. KING.

Smelting furnace. A. JONES, ASST. to U.S. SMELT-ING FURNACE CO. (U.S.P. 1,712,769, 14.5.29. Appl., 29.10.25).—In a rotatable tilting furnace a fuel supply pipe extends along the furnace with a lateral branch carrying the burner, so that when the pipe is rotated the nozzle may be in or out of alinement with the furnace. C. A. KING.

Ore-dust treatment. C. V. McINTIRE, Assr. to CONSOLIDATION COAL PRODUCTS CO. (U.S.P. 1,711,153, 30.4.29. Appl., 6.7.25).—The dust is mixed with coal and the mixture heated and agitated in a retort to form agglomerates of semi-coke suitable for blast-furnace smelting. A. R. POWELL.

Recovery of volatilisable metals [zinc] from iron-containing materials. F. KRUPP GRUSONWERK A.-G. (B.P. 299,300, 20.8.28. Ger., 22.10.27).—Iron ore containing zinc is mixed with at least 45% of coke and the mixture heated in a rotary furnace to reduce the iron to metal sponge and volatilise the zinc; the large excess of fuel prevents sintering of the charge and segregation of the sponge iron. A. R. POWELL.

Direct reduction of iron from its ores. S. L. MADORSKY, ASST. to GATHMYS RES. CORP. (U.S.P. 1,711,738, 7.5.29. Appl., 1.2.26).—Iron oxide ore containing titanium is fractionally reduced by passing hydrogen through the molten ore substantially free from carbon and nitrogen. C. A. KING.

Manufacture of cast iron. INTERNAT. NICKEL CO., P. D. MERICA, J. S. VANICK, and T. H. WICKENDEN (B.P. 311,798, 15.2.28).—A cast iron which does not exhibit the phenomenon of growth contains 3% C, less than 1.5% Si, and up to 5% Ni, 2—4% Ni being added for every 1% Si below the normal amount.

A. R. POWELL.

Carburisation of iron and steel. R. G. GUTHRIE and O. J. WOZASEK, ASSTS. to PEOPLE'S GAS BY-PRODUCTS CORP. (U.S.P. 1,706,356, 19.3.29. Appl., 2.1.29).—Iron or steel is case-hardened in a retort filled with a carburising gas, a minute quantity of oxygen being admitted at the beginning of the heating to form an oxide catalyst which increases the depth of penetration of the carbon.

A. R. POWELL.

Improvement of ferrous metals and products thereof. E. S. FISHER, ASST. to UTAH METALS FLUX Co. (U.S.P. 1,712,879, 14.5.29. Appl., 20.8.26).— Molten iron is brought into contact with material similar to the carbonaceous shale occurring in districts of Emery County, Utah. C. A. KING.

Annealing of carbon-containing iron and steel. VEREIN. STAHLWERKE A.-G., Assees. of E. H. SCHULZ and W. HULSBRUCH (B.P. 283,200, 12.12.27. Ger., 8.1.27).— To prevent decarburisation of the surface layers of iron and steel during annealing, a neutral atmosphere comprising a mixture of a carburising and of a decarburising gas is maintained in the furnace. The most suitable mixtures are 10-40 vol.-% of methane and 90-60 vol.-% of hydrogen. [Stat. ref.]

#### A. R. POWELL.

**Production of protective coatings on ferrous metals.** F. B. RINCK, ASST. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,690,378, 6.11.28. Appl., 17.10.24).— Ferrous metal is subjected to an oxidising atmosphere, *e.g.*, steam, at constant temperature (about 760°), the admission of steam being periodically increased, the hydrogen concentration reduced by having the hydrogen and the excess of uncombined oxygen minimised, *e.g.*, by the introduction of hydrocarbon vapour.

R. BRIGHTMAN.

Production of diffused layers of metals [on iron]. E. LAY (U.S.P. 1,711,603, 7.5.29. Appl., 31.10.27. Ger., 15.6.26).—Iron is heated with a powder containing 8—30% of free aluminium.

#### H. ROYAL-DAWSON.

Hardening of molybdenum irons or steels. J. L. F. VOGEL (B.P. 311,588, 4.7.28).—The case-hardening of molybdenum steels by nitrogenisation is effected by heating them at 500—600° with calcium cyanamide and passing a controlled current of steam through the vessel for several hours. The following nitrogenising compounds may be used in place of the cyanamide: cyanides, urea, sodamide, naphthylenediamine, dicyanodiamide, phenylhydrazine, acetamide, or guanidine.

A. R. POWELL.

Iron-nickel alloy. F. KRUPP A.-G. (B.P. 302,994, 4.8.28. Ger., 24.12.27).—Alloys having high sp. resistance and high initial permeability, and containing 33-48% Ni and 0.3-5% Si, are claimed.

F. G. CROSSE.

Magnetic alloy. O. E. BUCKLEY, ASST. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,695,038, 11.12.28. Appl., 6.3.25).—A nickel-iron alloy, containing 80—83% Ni, with a higher permeability than iron at magnetising forces less than a few tenths of a c.g.s. unit, and independent of tension up to 5000 lb./in.<sup>2</sup>, is used as loading material on signalling conductors for submarine cables. R. BRIGHTMAN.

Stable-surface alloy steel resistant to acids. R. P. and H. A. DE FRIES, ASSTS. to LUDLUM STEEL CO. (U.S.P. 1,712,030, 7.5.29. Appl., 26.11.26).—The alloy consists of 14—30% Ni, 6—20% Cr, 1—3% Si, 1—3% Ta, 0—1% C, the remainder being iron.

#### H. ROYAL-DAWSON.

Rust-, acid-, and heat-resisting ferrous alloy containing chromium and tantalum. P. A. E. ARMSTRONG (U.S.P. 1,711,484, 7.5.29. Appl., 8.8.25).— The alloy contains 7—25% Cr, less than 1.25% C, and less than 10% Ta. H. ROYAL-DAWSON.

[Manufacture of chromium or manganese] iron or steel alloys. T. R. HAGLUND (B.P. 311,256, 7.2.28). —High-grade iron ore with a low content of silica is smelted with 60—70% ferrochromium, or with 80% ferromanganese, and with carbon so that an iron alloy with the desired chromium or manganese content is obtained together with a slag rich in chromic or manganous oxide and containing less than 20% of silica. Before tapping, the slag is heated to such a temperature that the carbon content of the alloy is reduced to the correct amount by interaction with the slag, and subsequently other alloying elements, *e.g.*, nickel, are added as required. A. R. POWELL.

Steel alloy. J. L. Cox (U.S.P. 1,711,519, 7.5.29. Appl., 12.2.27).—A forgeable steel alloy contains 0.35— 1.5% C, 4—20% Cr, 6—18% W, and 1—6% Al. The chromium content is at most four times that of tungsten, and that of tungsten at most six times that of aluminium. C. A. KING.

Alloy steel. G. J. COMSTOCK, ASST. to FIRTH-STERLING STEEL CO. (U.S.P. 1,695,916, 18.12.28. Appl., 30.6.27).—Alloy steel containing  $1\cdot 2$ — $1\cdot 8\%$  C, 10—14%Cr,  $0\cdot 75$ — $1\cdot 25\%$  V, and  $0\cdot 5$ — $1\cdot 25\%$  Mo has a high resistance to abrasion and can be air-hardened or annealed soft. R. BRIGHTMAN.

Reconditioning iron and steel drums. R. C. HEINZMAN (U.S.P. 1,696,036, 18.12.28. Appl., 15.10,27). —Internally-rusted drums are reconditioned by applying a solution of oxalic acid evenly over the interior surface, admitting live steam to the drum, and finally applying a solution of aluminium acetate. A rust-resisting, waterproof coating is produced over the inner surface.

R. BRIGHTMAN.

Agent for cleaning and imparting rust-inhibitive properties to steel. J. D. KLINGER (U.S.P. 1,695,430, 18.12.28. Appl., 27.6.24).—A mixture of alcohol, citric acid, and sulphuric acid is used. C. HOLLINS.

Concentration of copper ores. J. A. HOLLADAY, Assr. to UNION CARBIDE SALES CO. (U.S.P. 1,706,293, 19.3.28. Appl., 11.8.26).—Oxidised copper ores are prepared for flotation by treating the finely-divided ore suspended in water with acetylene in the presence of an inert, non-combustible gas, *e.g.*, nitrogen, whereby the action of the acetylene on the particles of copper mineral is intensified. A. R. POWELL.

Refining of copper. H. H. ALEXANDER (B.P. 311,812, 16.1.28. Cf. B.P. 309,848; B., 1929, 479).— After refining, the surface of the molten copper is protected from oxidation and contamination by a molten layer of a sodium silicate or silicates to which may be added calcium oxide or fluoride; this layer covers the molten metal bath entirely, and is maintained during casting. M. E. NOTTAGE.

Heat treatment of aluminium castings. R. J. ROSHIRT, ASST. to BOHN ALUMINUM & BRASS CORP. (U.S.P. 1,713,093, 14.5.29. Appl., 18.1.26).—Aluminium alloys are quenched from as high a temperature as possible without distortion and reheated at 218—246°.

Surface-treatment [colouring] of aluminium articles. A. PACZ (U.S.P. 1,710,743, 30.4.29. Appl., 16.4.26).—The articles are dipped in a solution containing a soluble fluorine compound and a salt of silver, nickel, tin, cobalt, zinc, cadmium, iron, manganese, lead, antimony, or copper. H. ROYAL-DAWSON.

Solder for aluminium. A. YERSIN (Swiss P. 122,845, 21.1.27).—An alloy of 60% Sn, 35% Zn, and 5% Al is claimed. A. R. POWELL.

Increasing the sp. gr. of molten electrolytes [for refining aluminium]. R. MÜLLER (G.P. 454,719, 13.1.26).—An addition of barium or strontium oxide, peroxide, or carbonate is made. A. R. POWELL.

Aluminium alloy. H. C. HALL and T. F. BRAD-BURY (B.P. 312,555, 11.9.28. Addn. to B.P. 300,078; B., 1929, 24).—In the alloy described in the prior patent, the silicon content is increased up to 1.75% at the expense of the aluminium. F. G. CROSSE.

[Nickel] alloys. INTERNAT. NICKEL Co., and (A) E. J. BOTHWELL, (B) N. B. PILLING (B.P. 311,749 and 311,799, 15.2.28).—(A) An alloy containing 55—80% Ni,  $43 \cdot 5 - 6 \cdot 5\%$  Cu, 5—1% Si,  $0 \cdot 5 - 5\%$  Fe,  $0 - 0 \cdot 5\%$  C, and up to 3% Mn is claimed. The alloy is annealed at 830—1150°, quenched or air-cooled, and tempered at 500—890°. (B) The working properties of alloys with a high nickel content are improved by the addition of calcium just before pouring, preferably in the form of nickel or copper-nickel alloy containing 5—95% Ca and introduced below a layer of silicious material covering the molten metal. A. R. POWELL.

Protection of magnesium and its alloys. G. MICHEL (B.P. 287,046, 16.2.28. Fr., 12.3.27. Addn. to B.P. 249,484; B., 1927, 785).—The metal is coated with a varnish mixture having a base of synthetic resin and containing free phenol. The coating is subsequently baked. F. G. CROSSE.

Treatment of magnesium and its high-percentage alloys in the molten state. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 311,998, 9.8.28).—In the production of sound castings from magnesium and its high-percentage alloys, a coherent film in the shape of a hose of reaction products of the metal is caused to be formed on the surface of the flowing metal; this may be assisted by adding substances such as finely-divided sulphur to the atmosphere surrounding the molten metal. The mouth of the ladle is kept in a fixed position to the gate of the mould, and the rate of flow is regulated by tipping the ladle or crucible, the mouth of which acts as the pivot-point. M. E. NOTTAGE.

Manufacture of tungsten alloys low in tin and arsenic. F. M. BECKET, Assr. to ELECTRO METALLUR-GICAL CO. (U.S.P. 1,706,344, 19.3.29. Appl., 11.12.26).— Tungsten ores containing tin and arsenic are reduced in an electric furnace to produce an alloy containing at least 10% Si. This alloy is then fused with a metal oxide in quantity sufficient to oxidise and slag off the silicon. A. R. POWELL.

Recovery of zinc oxide. C. A. H. DE SAULLES, Assr. to AMER. SMELTING & REFINING CO. (U.S.P. 1,712,553, 14.5.29. Appl., 31.12.21).—Zinc ore is reduced continuously on the slag bath in a furnace having a slightly oxidising atmosphere, and the volatilised zinc products are condensed in a cooler chamber having an essentially oxidising atmosphere. C. A. KING.

Treatment of nickeliferous material [South African copper-nickel-platinum concentrates]. S. C. SMITH (B.P. 311,909, 18.4.28).—The ore is smelted to matte, which is roasted and reduced to a metal sponge by means of coal gas or other reducing agent. The metal sponge so obtained is leached with hot dilute

C. A. KING.

sulphuric acid, then air is drawn through the mass, whereby oxidation ensues with rise of temperature. A second charge of acid is passed through the mass and the air oxidation is repeated. Alternate leaching and oxidation are continued until no further metal is extracted, the residue is again roasted and reduced, and the leaching and oxidation are repeated until a product with a high content of platinum metals is obtained which can be refined by known methods.

#### A. R. POWELL.

Concentration of ores containing platinum and metals of the platinum group. E. C. R. MARKS. From CAYZER TIN SMELTING CO. (PROPRIETARY), LTD. (B.P. 311,991, 24.7.28).—Ores containing sulphides of nickel, copper, and iron, together with the platinum metals, are roasted sweet, mixed with 10% of bituminous coal or semi-coke, and heated at 900—1100° in a reducing atmosphere in a furnace with rotating hearth. The product is cooled out of contact with the air, and the sponge of iron, nickel, and copper containing all the platinum metals is separated by known means and treated for the recovery of the platinum metals.

A. R. POWELL.

Coating for metals. C. CHAFFER and H. HARGREAVES (B.P. 312,478, 12.5.28).—The metals are coated with an aqueous solution of gelatin. Hardening of the coating is effected with alcohol. F. G. CROSSE.

Producing points of adhesion for galvanic deposits on light-metal bodies. LEIPZIGER SCHNELL-PRESSENFABR. A.-G. (B.P. 305,453, 29.5.28. Ger., 4.2.28).—Pieces of metal having a better adhesion for the galvanic deposit than the light metal are placed in the casting mould of the light-metal body to be cast so that they are enclosed and appear on the surface of the finished casting. J. S. G. THOMAS.

Working of blast furnaces. P. HESKAMP, Assr. to VEREIN. STAHLWERKE A.-G. (U.S.P. 1,713,435-6, 14.5.29. Appl., 5.11.26. Ger., [A] 6.4.26, [B] 8.9.26).-See B.P. 269,071 and 272,751; B., 1927, 575, 659.

Production of iron sponge S. E. SIEURIN (U.S.P. 1,714,280, 21.5.29. Appl., 17.12.27. Swed., 26.1.26).— See B.P. 296,235; B., 1928, 820.

Heat treatment of steel. N. D. CHOPRA and F. J. BULLEN (U.S.P. 1,712,253, 7.5.29. Appl., 18.3.27. U.K., 1.4.26).—See B.P. 271,606; B., 1927, 560.

Ascertaining the resistance to rusting shown by iron, steel, and iron alloys. C. MAULER (U.S.P. 1,714,035, 21.5.29. Appl., 11.7.27. Austr., 22.7.26).— See B.P. 292,794; B., 1928, 608.

Extraction of metals [zinc] from their [oxide] compounds. C. J. G. AARTS (U.S.P. 1,712,641, 14.5.29. Appl., 17.7.24. Belg., 28.7.23).—See B.P. 219,667; B., 1925, 676.

Reduction of zinciferous materials. F. G. BREVER and E. H. BUNCE, Assrs. to NEW JERSEY ZINC CO. (U.S.P. 1,712,132 and 1,712,134, 7.5.29. Appl., [A] 27.1.27, [B] 20.4.27).—See B.P. 298,921 and 300,519; B., 1928, 931; 1929, 100.

Production of finished metal articles. E. G. BEK and E. THOMA (U.S.P. 1,712,244, 7.5.29. Appl., 23.9.25. Ger., 11.12.24).—See B.P. 244,487; B., 1927, 633.

Electrolytic separation of metallic chromium for production of chromium coatings on other metals. R. APPEL (U.S.P. 1,713,514, 21.5.29. Appl., 3.8.26).—See B.P. 274,882; B., 1928, 97.

Steel etching plates (U.S.P. 1,705,155).-See XXI.

#### XI.—ELECTROTECHNICS.

Insulating masses for transformers. W. HAM-MERSCHMIDT (Z. angew. Chem., 1929, 42, 523—525).— The value of mixtures of rosin, rosin oil, and American asphalt as insulated sealing masses for transformers has been determined by measuring the voltage at which the insulation breaks down using electrodes 0.5 mm. apart. The results show that addition of rosin oil to rosinasphalt mixtures rapidly decreases the insulating power, the decrease corresponding with the increase in fluidity. A. R. POWELL.

See also A., June, 632, Magnetic properties of iron (von Auwers). 654, Electrolytic polarisation: complex cyanides of silver and of copper (GLAS-STONE). 671, X-Ray tube for spectroscopy (BAND and MADDOCK). 673, Glass electrode (MacInnes and Dole).

Comparison of colours. TOUSSAINT.—See I. Recovery of metals. Eger. Cadmium plating. WESTBROOK. Corrosion of lead. BURNS and CAMP-BELL.—See X. Sterilisation of liquids. LAKHOVSKY. —See XXIII.

#### PATENTS.

Electric discharge tube. F. SKAUPY, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,712,407, 7.5.29. Appl., 30.4.21. Ger., 4.7.19).—Non-alkaline electrodes of appropriate area and consisting of fine wire mesh free from occluded non-inert gases are arranged in a sealed container filled with inert gas under reduced pressure, so that a glow discharge may be continuously produced between the electrodes at operating voltages of about 100 volts.

J. S. G. THOMAS.

[Cathode] filament. F. HOLBORN, ASST. to DE FOREST RADIO TELEPHONE & TELEGRAPH CO. (U.S.P. 1,695,687, 18.12.28. Appl., 9.1.24).—An alloy of iron or nickel and lithium, obtained by mixing iron with lithium amalgam, is claimed. R. BRIGHTMAN.

Electrolysis reduction cell. Apparatus for carrying out electrolytic reduction of sugars to alcohols. H. J. CREIGHTON, Assr. to ATLAS POWDER Co. (U.S.P. 1,712,951-2, 14.5.29. Appl., [A, B] 26.3.26. Cf. U.S.P. 1,612,361; B., 1927, 234).-(A) An agitator projects into a mercury cathode placed within a catholyte container surrounding a porous cylinder containing an anode and anolyte. The porosity of the cylinder wall is such that hydrogen ions can pass from the anodic to the cathodic compartment, and hydroxyl ions in the reverse direction, whilst catholyte is prevented from passing into the anodic compartment. (B) Sugarreducing apparatus comprises an electrolysis cell, from which an amalgam is discharged into a reduction cell provided with an agitator, and a mercury reservoir into which mercury is delivered from the reduction cell and

thence into the electrolysis cell, a given amount of mercury being maintained in each cell.

J. S. G. THOMAS. Electrodeposition. L. C. TURNOCK (U.S.P. 1,712,284, 7.5.29. Appl., 17.12.25).—In an electroplating system electrically-operated means are provided for interrupting the electrical circuit, including the bath, after a predetermined amount of metal has been deposited.

J. S. G. THOMAS.

**Electroplating apparatus.** J. L. MUJICA (U.S.P. 1,712,674, 14.5.29. Appl., 30.7.26).—A rotatable permeable container, having a throat piece at one end, is supported on a carrier suspended on trunnions arranged at one side of a stationary tank, so that the carrier with the container and throat piece may be tipped from a receiving position in the tank to a position outside the tank to discharge its contents clear of the rotating mechanism. J. S. G. THOMAS.

[Mounting for heating elements of] electric resistance furnaces. HEVI DUTY ELECTRIC Co., Assees. of E. L. SMALLEY (B.P. 312,006, 23.8.28. U.S., 22.5.28).

Sulphur trioxide and sulphuric acid (B.P. 311,862).—See VII. Iron-nickel alloys (B.P. 302,994). Magnetic alloy (U.S.P. 1,695,038). Electrolytes for aluminium (G.P. 454,719). Deposits on light metal bodies (B.P. 305,453).—See X.

# XII.—FATS; OILS; WAXES.

Wizöff report: meeting of the German Commission for development of standard analytical methods in the fat industry (Chem. Umschau, 1929, 36, 137—164).—The suggested standard methods of fat analysis applicable to sulphonated oils, waxes, soaps, etc., and glycerin, circulated previously by the Wizöff (Central Association for Scientific Research in the Oil and Fat Industries) were discussed and amended.

# E. LEWKOWITSCH.

Wizöff communication. Colorimetry of oils and fats. G. GREITEMANN (Chem. Umschau, 1929, 36, 167— 168).—Iodine solutions are recommended as standards for the colorimetry of oils and fats where measurement of depth of colour is the primary requirement: the solutions keep well if protected from oxidation, and the apparatus required is cheap and simple. The " colour number" is defined as the number of mg. of free iodine contained in 10 c.c. of an aqueous iodine-potassium iodide solution which appears of an equal depth of colour in a layer 25 mm. thick examined by transmitted light (daylight lamp). (Cf. preceding abstract.)

#### E. LEWKOWITSCH.

Unification of methods for determination of iodine value. J. GIRAL (Anal. Fis. Quim. [Tecn.], 1929, 2, 259—264).—A review. Suggestions are made for the definition of iodine value and for statements of the conditions used when the results of the determination of iodine values are recorded. The definitions and methods of determination of moisture and cellulose in foodstuffs are also discussed. R. K. CALLOW.

Turbidity and solidification times of solid fats, with especial reference to cacao butter. W. SPRINGER (Chem. Umschau, 1929, 36, 165-167).—The times taken by a melted fat cooling from  $60^{\circ}$  to reach (a) a definite degree of turbidity, and (b) the solidification point are found to be characteristic for different fats, and may be used for discrimination, particularly in the examination of cacao butter. Thus, the turbidity and solidification times, respectively, are : for cacao butter (of the order of) 500—1000 sec., 65—100 min.; for hardened coconut oil and other vegetable fats 300 sec., and 6—9 min.; and for tallow 75 sec., and  $1 \cdot 2$  min. It is preferable to deacidify the fats before testing. E. LEWKOWITSCH.

Effect of clay on rancid fats. G. W. FIERO (J. Amer. Pharm. Assoc., 1929, 18, 491-493).—Colloidal clay removed rancidity from coconut oil either by simple agitation, by boiling with water and clay, or by treatment with clay, superheated steam, and reduced pressure. The first method gave the best product, and colour, odour, and taste were all improved. Under the conditions employed lard could not be completely renovated by any of the methods. E. H. SHARPLES.

Indirect analysis of three-component systems, especially of mixtures of fatty acids. K. TÄUFEL and M. RUSCH (Z. Unters. Lebensm., 1929, 57, 216—224). —The use of triangular co-ordinates for the indirect analysis of three-component systems is illustrated, using as examples : (a) a mixture of oleic, linoleic, and erucic acids, of known iodine and neutralisation values, and (b) a mixture of ammonium carbonate, bicarbonate, and carbamate, of which the contents of ammonia and carbon dioxide are known. The method is inapplicable to a mixture of oleic, linoleic, and linolenic acids, since the lines for the iso-iodine and iso-neutralisation values are parallel. W. J. BOVD.

Glycerides of fats and oils. XIII. Glycerides of chaulmoogra oil. A. BÖMER and H. ENGEL (Z. Unters. Lebensm., 1929, 57, 113-147).-Chaulmoogra oil is rapidly oxidised in the air, but can be protected by hydrogenation. By repeated fractional crystallisation of the deacidified hardened oil from acetoneether and acetone, a dihydrochaulmoogra-didihydrohydnocarpin, m.p. 30.7° (corr.), was isolated in yield amounting to 79% of the deacidified hardened oil. A dihydrohydnocarpo-didihydrochaulmoogrin, m.p. 42.2° (corr.), in 13% yield was also isolated, and a small quantity of difficultly soluble glyceride, probably tripalmitin or a stearodipalmitin. It is deduced that in the unhardened oil the corresponding unsaturated glycerides occur in similar proportions and that the fatty acids of the hardened oil before deacidification consisted approximately of 40% of dihydrochaulmoogric acid and 59% of dihydrohydnocarpic acid. The unusually low m.p., high refraction, and high solubility of hardened chaulmoogra oil as compared with other fats is due to the cyclic nature of the fatty acids. Tridihydrochaulmoogrin (m.p.  $51.0^{\circ}$ , corr.), tridihydro-hydnocarpin (m.p.  $39.2^{\circ}$ , corr.), and didihydrochaul-moogrin (m.p.  $60.7^{\circ}$  corr.) were prepared synthetically from the lead soaps and appropriate halogenohydrins.

W. J. BOYD.

Drying of linseed oil and boiled oils. H. WOLFF and W. TOELDTE (Farben-Ztg., 1929, 34, 2069—2072).— The rate of increase in weight of linseed and manganeseboiled oil films corresponds approximately with the rate of drying as measured by the authors' dye-spreading test (B., 1929, 255); with cobalt-boiled oil change in weight begins after drying is well advanced. Increasing the thickness of the film scarcely affects the course of drying (physical change), but influences considerably the shape of the weight/time curves (chemical change): with thicker films a decrease in weight of the film was observed at the beginning of the drying process.

E. LEWROWITSCH.

Economic utilisation of residues from oil manufacture : production of comestible flours rich in aleurone. E. ANDRÉ (Bull. Soc. d'Encour., 1929, 128, 347—359).—Oil seeds, e.g., sunflower, sesamé, linseed, are rich in aleurone grains : by extracting the oil with light petroleum and applying to the residual meal grading methods similar to those used in milling, a nutritive nitrogenous product valuable for human consumption is obtained. Especially suitable is sunflower-seed meal, which is rich in aleurone grains containing 8.85% N and 3.2% P<sub>2</sub>O<sub>5</sub>, equivalent to a 55% content of proteins. E. LEWKOWITSCH.

See also A., June, 641, Adsorption of soap solutions (NONAKA; MIKUMO). 646, Formation of soap gels (HOLMES and MAXSON). 649, Alkalinity of soap solutions (McBAIN and HAY). 671, Calibration of Lovibond glasses (PRIEST and others). 712, Determination of carbon and carbon dioxide (FRIEDEMANN and KEN-DALL). 714, Fats of Japanese birds (KOYAMA). Silkworm chrysalis oil (DE'CONNO and QUARTO). Action of bromine on insect oils (DAVID). 725, Germicidal and hæmolytic acition of a-bromosoaps (Eggerth). 726, Detection of vitamin-A (STEUDEL). Colour tests of vitamin-A in cod-liver oil (Towle and MERRILL). Fat-soluble vitamins in cod-liver oil (BACHARACH and SMITH). Vitamin-A in the liver (LAQUEUR and others). Vitamin colour reactions (BEZSSONOFF). 727, Irradiation of ergosterol (DELAPLACE and REBIÈRE). 729, Oil from Datura stramonium (VERKADE and COOPS). Seeds of Monarda punctata (HARWOOD).

Analysis of butter. CHARLIERS. Oil content of paprika. Dömötör.—See XIX.

#### PATENTS.

Manufacture of soft soap. L. G. LEFFER, Assr. to H. BACHSTEIN (U.S.P. 1,710,799, 30.4.29. Appl., 25.3.26, Ger., 4.4.25).—Coconut oil, palm-kernel oil, and rape-seed oil are saponified with potash lye under pressure, hydrocarbons being added during the process to regulate the setting point. E. LEWKOWITSCH.

Apparatus for separating fat or other material from gases. J. LYONS & CO., LTD., and G. W. CATLIN (B.P. 311,204, 3.2.28).—The apparatus comprises the chamber in which the mixture is formed, from which the mixture is drawn off laterally on opposite sides and conducted to an auxiliary chamber provided with baffles to retain all or a desired quantity of solid material.

E. LEWKOWITSCH. Detergent. J. TENGLER (F.P. 629,691, 23.2.27. Spain, 18.3.26).—Benzine and carbon tetrachloride are added to soap liquefied by heating, and the product, when cold, is coated with wax, fat, varnish, etc. to prevent loss of the solvent by evaporation. L. A. COLES. Production of a detergent powder. A. LINDAHL (Swed. P. 58,788, 5.4.21).—A mixture of oil or fatty acids, water, and anhydrous sodium carbonate is treated with a blast of compressed air, or, alternatively, the water is replaced by a current of steam. The product settles out as a powder. L. A. COLES.

Bleaching of fatty acids. A. WELTER (G.P. 454,308, 3.9.25).—The fatty acids (or oils) are saponified with soda (or caustic soda); the soaps are bleached with chlorine or sodium hypochlorite, treated with sulphuric acid, and vigorously agitated with air.

E. LEWKOWITSCH.

**Refining and bleaching of fish oils.** J. C. KERNOT (B.P. 311,665, 10.12.27).—The oily emulsion formed by subjecting fish oils to the action of aqueous caustic alkalis below 100° is separated, washed and agitated with a concentrated aqueous solution of sulphur dioxide or like sulphurous bleaching agent, and the separated oil is washed, dried, and filtered. Treatment with sulphurous acid may be repeated or the bleaching carried further by selective hydrogenation in the presence of mixed catalysts (e.g., hydrated 'oxides of nickel and aluminium). E. LEWKOWITSCH.

Prevention of rancidity in vegetable oils used in plastic compositions for coating fabric, paper, and the like. G. E. SCHARFF, and NOBEL'S EXPLOSIVES Co., LTD. (B.P. 311,786, 17.11.27. Addn. to B.P. 256,654; B., 1926, 887).—About 8% of zine (or magnesium) oxide may be advantageously added to the antimony sulphide (or other metallic sulphide of the second group in wet analysis) to be incorporated in the plastic coating, a total of 2% (calc. on the vegetable oil content) of the mixture being suitable.

E. LEWKOWITSCH.

Apparatus for distillation of fatty acids, glycerin, etc. W. GENSECKE (U.S.P. 1,713,431, 14.5.29, Appl., 12.11.24. Ger., 28.11.23).—See B.P. 225,552; B., 1925, 556.

Removal of boiler-scale (B.P. 311,550).-See XXIII.

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

Action of the substratum on coloured paints. H. WAGNER (Farben-Ztg., 1929, 34, 2011-2014).--The unsatisfactory behaviour of chalk as a white base in coloured paints has been investigated by comparing the properties of paints made from it with those of paints made from barium or calcium sulphate. The inferiority of the chalk paints is not due to their impermeability to ultra-violet light, which is about equal to that of paints with barytes and other white bases, but appears to be due to the flocculated condition of the chalk particles in the prepared paint, to the fineness of the particles, to their soft texture, to the high oil content of the paint, and to its great tendency to swell. A. R. POWELL.

Sipalin: a new softening reagent for cellulose lacquers. W. SCHRAUTH (Farben-Ztg., 1929, 34, 2018—2019).—The eyclohexyl and methylcyclohexyl esters of adipic and methyladipic acids are manufactured under the general name of "sipalin" for use in the preparation of cellulose lacquers. They are all viscous liquids with very low m.p. and high b.p., are practically odourless, non-hygroscopic, and non-volatile, have a flash point above 180°, and are readily miscible with cellulose acetate and nitrate, resins, fatty oils, and rubber-producing plastic masses to give tenuous, pliable, and non-cracking films. Solutions of cellulose esters in sipalin may be coloured with inorganic pigments or with organic dyes. A. R. POWELL.

#### See also A., June, 696, Resin acids (ROUIN).

Comparison of colours. TOUSSAINT.—See I. Drying of linseed oil and boiled oils. WOLFF and TOELDTE.—See XII.

#### PATENTS.

Production of oiled pigments. H. GROSSMAN, Assr. to W. S. PRITCHARD (U.S.P. 1,696,034, 18.12.28. Appl., 25.2.27).—An emulsion of oil in water is added to a suspension of the pigment, the protective colloid (casein) is coagulated, and the oil in separating carries the pigment with it, giving an oiled pigment which retains its fine state of division. C. HOLLINS.

Brushing lacquers. S. D. SHIPLEY, Assr. to ATLAS POWDER Co. (U.S.P. 1,692,584, 20.11.28. Appl., 28.9.25). —A nitrocellulose lacquer is employed which contains the minimum of glycol monoethyl ether as solvent, diluted with petrol; *e.g.*, glycol monoethyl ether (40 pts.), gum solution (25 pts.), petrol (35 pts.), nitrocellulose (12 oz. per gal.), and pigment (3.25 lb./gal.).

C. HOLLINS.

Lacquer solvent. J. P. TRICKEY, ASST. to QUAKER OATS CO. (U.S.P. 1,703,697, 26.2.29. Appl., 15.1.27).— Tetrahydrofurfuryl alcohol, alone or mixed with, *e.g.*, butyl alcohol (5—15%), butyl acetate (5—15%), ethyl acetate (20—30%), or toluene (30—45%), is used as solvent for cellulose nitrate, gums, etc. for lacquers.

R. BRIGHTMAN.

Production of synthetic resins. LA FIBRE DIA-MOND (B.P. 282,435, 16.12.27. U.S., 16.12.26).—A mixture of a phenol, e.g., cresol, with a larger quantity of an amine, e.g., aniline, is heated with an unsubstituted aldehyde, e.g., formaldehyde, and the product is distilled under reduced pressure to remove uncondensed constituents etc., leaving as residue a soluble, readily fusible condensation product which may be used direct for the manufacture of varnishes etc., or may be heated under pressure and in the presence of hardening agents, e.g., hexamethylenetetramine, for the production of hard, infusible resins. L. A. COLES.

Resinous material and its manufacture. C. ELLIS (U.S.P. 1,691,271, 13.11.28. Appl., 28.11.24).— A low-grade phenolic oil is condensed at 15° with a mixture of magnesia and formaldehyde; plaster of Paris is added (to take up the 4—5% of water in the separated resin) together with other fillers etc. to give a moulding-powder. C. HOLLINS.

Production of plastic composition materials. C. A. NASH, ASST. to CUTLER-HAMMER MANUF. Co. (U.S.P. 1,692,856, 27.11.28. Appl., 12.3.21).—Phenol and formaldehyde (or hexamethylenetetramine) are caused to react below 80° in presence of a filler, *e.g.*, asbestos, reaction being stopped when about half the materials have combined. C. HOLLINS.

Sulphur product and its manufacture. C. ELLIS (U.S.P. 1,690,335, 6.11.28. Appl., 7.4.28).—Crude cresylic acid, e.g., a tar-acid fraction 50% of which distils below 212°, is treated with an equal weight of sulphur monochloride in presence or absence of a diluent, e.g., toluene, and the product is deodorised by steaming at 140—150°. The sulphurised product, m.p.  $91.5^\circ$ , is incorporated with sulphur, a mixture of 25% of resin and 75% of sulphur having m.p. 105°. Sulphuryl chloride or sulphur dichloride may also be used.

R. BRIGHTMAN.

Mill for fine grinding [for paints etc.]. J. R. TORRANCE (U.S.P. 1,713,487, 14.5.29. Appl., 1.10.23. U.K., 17.10.22).—See B.P. 209,829; B., 1924, 238.

Production of titanium-containing pigments. F. G. C. STEPHENS, L. J. ANDERSON, and W. A. CASE, Assrs. to NAT. METAL & CHEM. BANK, LTD. (U.S.P. 1,714,408, 21.5.29. Appl., 24.3.27. U.K., 26.3.26).— See B.P. 273,017; B., 1927, 661.

Protection of magnesium etc. (B.P. 287,046).—See X.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

See A., June, 644, Viscosity of caoutchouc solutions (KROEPELIN). 674 and 702, Natural and synthetic rubber (MIDGLEY and HENNE).

#### PATENTS.

Manufacture of rubber articles. H. REITZ (G.P. 453,899, 2.11.23).—Latex, to which colloidal sulphur or soluble sulphides may have been added, is treated with aldehydes in the form of solution or vapour, which renders it viscous. The latex is then shaped or spread as desired and vulcanised. D. F. Twiss.

Vulcanisation of rubber. I. OSTROMISLENSKY, Assr. to REVERE RUBBER CO. (U.S.P. 1,696,409, 25.12.28. Appl., 24.3.25. Cf. U.S.P. 1,249,180; B., 1918, 66 A).— Rubber is vulcanised with a nitro-compound (trinitrobenzene) in presence of amines (aniline). As accelerators elements of the carbon group (lamp black, graphite, silicon) are used, and as activators metal oxides, *e.g.*, litharge. C. HOLLINS.

Vulcanisation [of rubber]. GIBBONS BROS., LTD. From C. DRESSLER (B.P. 311,507, 21.3.28).—Rubber articles, in moulds or otherwise, are passed slowly and continuously through an elongated chamber so that they are first gradually heated, then subjected to vulcanising heat, and finally cooled, the atmosphere, if desired, being of carbon dioxide. Arrangements may be provided for any moulds to be closed after the warming operation and before vulcanisation. D. F. TWISS.

# XV.-LEATHER; GLUE.

Moisture in leather. II. H. BRADLEY, A. T. MCKAY, and B. WORSWICK (J. Soc. Leather Trades' Chem., 1929, 13, 87—106; cf. B., 1929, 446).—The permeabilities of various leathers to water vapour have been determined. "Waterproofed" chrome upper leathers were most highly resistant to the passage of water vapour through them. Vegetable-tanned sole and insole leathers were least resistant. Diffusion constants have been determined for water into various leathers by immersing a rectangular piece of the leather in distilled water and noting the increase in weight at frequent intervals. The diffusion constant is a characteristic of the leather. The greater it is, the more absorbent or less waterproof is the leather. The smallest diffusion constant was shown by waterproofed chrome-tanned upper leathers. Curried vegetable-tanned splits had a diffusion constant three times, and waxed chrome-tanned sole leather five times, that of the above leathers. Sole leathers are only one fifth as waterproof as the corresponding uppers. D. WOODROFFE.

Double refraction of tanned collagen fibres. A. KÜNTZEL (Collegium, 1929, 207-214).—Collagen fibres were treated for 24 hrs. with solutions of gallotannic acid, sumac, pine bark, sulphite-cellulose, and chromium chloride, respectively, dried, immersed in different liquids, and examined. The double refraction of fibres treated with sumac and gallotannic acid was found to be reversed when they were examined in aniline, eugenol, and nitrobenzene, but the other treated fibres showed positive double refraction. No reversal occurred with tanned fibres when similarly treated. The tannins are not taken up between the micelles but by them, with consequent change of structure and optical properties. Tannage is a "permutoid reaction" and not a micellar surface reaction. D. WOODROFFE.

Action of water on vegetable-tanned leather. I. Rate of removal of uncombined soluble matter. H. B. MERRILL (J. Amer. Leather Chem. Assoc., 1929, 24, 235—242).—Leathers loaded with salt, glucose, and gallic acid were washed in Wilson-Kern extractors with distilled water and samples of the leather analysed at intervals. The whole of the loading material was removed after 2 hrs.' treatment. The removal of all the soluble, natural non-tannin took 4 days, but 5% of the water-soluble organic matter was removed in 2 hrs. Natural and added non-tannins cannot be differentiated by selective extraction.

D. WOODROFFE.

Tanning properties of dyewood extracts. H. A. BRECHT (Collegium, 1929, 186-191).-Dyewood extracts contain flavonols or closely allied products, which precipitate gelatin solutions and salts of heavy metals, reduce Fehling's solution, and give all the reactions of the pyrocatechol tannins. They resemble tannins in the insolubility of the pure flavonols and the solubility of the dyewood extract. Analyses of extracts of the following by the filter-bell method gave (% tannin): fiset wood,  $d \ 1.180, \ 22.2$ ; liquid fustic,  $d \ 1.241, \ 45.6$ ; and dried logwood, 34.1. The non-tan filtrates were colourless. By the shake method of analysis lower results were obtained, and logwood and hæmatin extracts gave pale yellow non-tan filtrates. The hæmatein was absorbed by the hide powder in the tannin analysis, but the hæmatoxylin remained in the liquor. Dyewood extracts are too expensive to use for tanning purposes, but they are often used unknowingly for dyeing on account of their tanning properties, giving weight and a superior feel to the leather, which cannot be obtained by coal-tar dyes, hence their non-replacement by the latter for certain purposes. The close relationship between the structure of the pyrocatechol tannins and the natural dyes is described.

D. WOODROFFE.

Evaluation of tannin solutions by interferometry. F. ENGLISH (Collegium, 1929, 109—119).—It is shown that the measurement of interference effects of tannin solutions can be used for their evaluation and for distinguishing tans and non-tans. An exact determination of the interference factor has hitherto been impossible owing to the effect of concentration of the solution, temperature, and the method of detannisation. It is suggested that interference measurements could replace gravimetric analysis on the same tannins in extract factories or special laboratories. D. WOODROFFE.

Determination of insoluble matter in tannin solutions. D. McCANDLISH and W. R. ATKIN (J. Soc. Leather Trades' Chem., 1929, 13, 69-72, J. Amer. Leather Chem. Assoc., 1929, 24, 231-235, and Collegium, 1929, 98-101).-The official international method of filtration requires too long for myrobalans and crude quebracho extracts, with which it is practically impossible to comply with the regulations. Filtration under reduced pressure gives clearer filtrates. A 500-c.c. filter flask is fitted with a rubber stopper and cylindrical separating funnel (120 c.c. capacity) with stopcock and side tube. A glazed Buchner funnel (2.75 in. diam.), fitted with a stopcock on the stem, is connected to the separating funnel. The side tubes of the filter flask and separating funnel are connected through a T-tube to a vacuum pump. A Whatman No. 5 paper is placed in the Buchner funnel and drawn down by running 25 c.c. of tan liquor over it. The two stopcocks being left open, the vacuum is increased, a mixture of 1 g. of kaolin in 30 c.c. of tan liquor is filtered to form a kaolin pad, the vacuum is allowed to fall, and tan liquor is passed through continuously for 1 hr., by which time sufficient filtrate for evaporation is collected in the separating funnel. Good concordance is claimed for the method. D. WOODROFFE.

Determination of insoluble matter in tannin extracts. C. RIESS (J. Amer. Leather Chem. Assoc., 1929, 24, 229—231, and Collegium, 1929, 103—4).—The procedure agrees with that of McCandlish and Atkin (cf. preceding abstract) with the difference that suction is applied by a suction tube. D. WOODROFFE.

Modified Wilson-Kern extractor [for hide powder]. H. B. MERRILL (J. Amer. Leather Chem. Assoc., 1929, 24, 244—245; cf. A., 1921, 819 A).—The upper part of the Wilson-Kern extractor has been eliminated, and the middle portion, finished off at the top as a bottle neck, is fitted with a 1-hole rubber stopper and tube provided with a stopcock to regulate the flow of ash water. D. WOODROFFE.

Determination of water content of glue and gelatin. E. SAUER and H. DELLENIUS (Z. angew. Chem., 1929, 42, 552—555).—An aqueous solution of 50—80 g. of material is made up to 1 litre at 40°, and 10 c.c. are evaporated to dryness, preferably in an aluminium dish, and dried to constant weight at 110°. J. S. CARTER.

Determination of insoluble matter in tanning extracts. A. TURNBULL (J. Soc. Leather Trades' Chem., 1929, **13**, 65–68, and Collegium, 1929, 101–103). —See B., 1929, 405.

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See also A., June, 646, Setting of gelatin sols (LOTTERMOSER and MATTHAES). Swelling of gelatin (NORTHROP and KUNITZ). Hydrolysis of gelatin (YAITSCHNIKOV).

Water supply for tanneries. AUERBACH.—See XXIII.

#### PATENTS.

Material for tanning and its manufacture. C. J. HERRLY and A. M. MATHESON, ASSTS. to NIACET CHEMICALS CORP. (U.S.P. 1,695,655, 18.12.28. Appl., 7.3.28).—Rapid tanning agents are made by condensing 2 mols. of a phenol with paraldehyde at 45—55° in presence of a little sulphuric acid and sulphonating the product. C. HOLLINS.

Tanning of hides. G. POVARNIN (Russ. P. 3364, 15.9.24).—The hides, with or without the usual pretreatment, are treated with furfuraldehyde or diacetyl or with solutions containing them. L. A. COLES.

**Treatment of hides and skins.** H. Dodge (U.S.P. 1,690,969, 6.11.28. Appl., 28.11.27).—Dried hides and skins are immersed in an aqueous solution of nitre, sodium bicarbonate, and formaldehyde. R. BRIGHTMAN.

Manufacture of [waterproof] glue. A. HOCHE, Assr. to LITMO ADHESIVE & PRODUCTS CO. (U.S.P. 1,710,687, 30.4.29. Appl., 11.10.22).—Chrome-tanned leather (2-5%  $\text{Cr}_2\text{O}_3)$  is treated with successive baths of sulphuric acid of decreasing acidities, e.g., d 1.162, 1.075, and 1.037, for 30 min. in each, and the extracted leather is dissolved by heating with water.

D. WOODROFFE.

Water-resistant animal glue. C. E. HRUBESKY and F. L. BROWNE (U.S.P. 1,712,077, 7.5.29. Appl., 14.3.28).—The glue consists of animal glue, water, paraformaldehyde, and a suitable amount of acid. H. ROYAL-DAWSON.

Adhesive material and its manufacture. L. BRADSHAW and H. V. DUNHAM (U.S.P. 1,703,134, 26.2.29. Appl. 17 12 26) Storeb assain and a soad mad rich

Appl., 17.12.26).—Starch, casein, and a seed meal rich in protein are incorporated with sufficient alkali for liquefaction in water. R. BRIGHTMAN.

Fertiliser (U.S.P. 1,695,081).-See XVI.

# XVI.—AGRICULTURE.

Influence of substituted cations on the properties of soil colloids. M. S. ANDERSON (J. Agric. Res., 1929, 38, 565—584).—In an investigation of the change in properties that different soil colloids may undergo when the exchangeable bases are substituted by different cations, determinations have been made of the heat of wetting, adsorption of water vapour, swelling, moisture equivalent, cataphoresis, and hydrogen-ion concentration of widely different colloidal materials when saturated with calcium, magnesium, potassium, sodium, hydrogen, and the cation of methylene-blue. The order of cation effects on heat of wetting and moisture adsorption is Ca>Mg>Na>H>K> methylene-blue, and on the remaining properties the order is Na>K>Ca>Mg>H. The varying effects are ascribed to differences in solu-

bility and dissociation tendency of the colloid-cation combinations. The influence of a cation appears to be in direct proportion to its percentage of complete replacement. The amount of change suffered through replacement varies widely in different colloidal soil materials. The ranges of alteration of different soil colloids correlate fairly well with magnitudes of the properties of the untreated or calcium-saturated materials, with the total content of exchangeable bases, and with the  $SiO_2/(Al_2O_3 + Fe_2O_3)$  ratio of the colloid. This is explained on the ground that many properties of the soil colloids are directly influenced by the total content of exchangeable bases, which is to some extent dependent on the  $SiO_2/(Al_2O_3 + Fe_2O_3)$  ratio. The range of alteration of the colloidal soil material should be considered in judging what soils would show appreciable change in properties following heavy applications of a single fertilising salt and also in indicating those alkaline soils which will become impenetrable on leaching. In most soil colloids except those of saline soils the general properties will be indicated by the total base-exchange capacity, or one of the determinations associated with it. E. S. HEDGES.

Soil structure. I. Mutual interaction of the sols of ferric hydroxide, silicic acid, and permanganate. V. N. SYMAKOV and S. P. KRAVKOV (State Inst. Exp. Agron., Bur. Soils Bull. No. 3, 1928; Proc. Internat. Soc. Soil Sci., 1929, 4, 54—55).—The proportions in which the sols mutually coagulate each other are determined, and the conditions of complete mutual coagulation of mixtures of 3 sols are examined and discussed. A. G. POLLARD.

Soil structure. I. Dependence of stability of soil structure on adsorptive complex and silt. II. Aggregate analysis as a method for determining real soil structure. A. T. TIULIN (Agric. Exp. Stat. Perm, No. 2, 1928; Proc. Internat. Soc. Soil Sci., 1929, 4, 49—51, 51—54).—I. The stability of soil structure as determined by washing out the sample with water at a definite pressure depends on the saturation capacity of the soil for calcium when the absorbing complex is saturated with calcium and magnesium, and is inversely proportional to the silt content. It can be expressed in the relationship percentage of silt/ adsorptive capacity for calcium. This relationship is designated a " colloid-mechanical factor of soil-structure stability."

II. Structural aggregates in soil are classified as (1) real aggregates, bound together by gels of biand ter-valent cations, and (2) false aggregates bound together by gels, or even suspensions, containing univalent cations. The real aggregates are stable in water, whereas false aggregates disperse. Soil structure depends on the first class. Real aggregates can be separated in water by Robinson's or the sieve method without pretreatment. False aggregates are separated in the air-dry condition with sieves, and those of smaller dimensions by Robinson's method using benzene in place of water. From the values obtained the amounts of real aggregates in the corresponding fractions are deducted. In comparative experiments more exact values for the real aggregates are obtained by deducting from the experimental values the amounts of elementary particles in the various fractures as obtained by normal mechanical analysis. There is a general parallelism between the "colloid-mechanical factor" and the results of aggregate analysis, although the latter method gives a more exact valuation of the actual soil structure. Comparative aggregate analyses of a number of soils classified these in the same general order as their cropproducing powers. Structural differences between the surface and subsoil due to tillage are reflected in the differences in aggregate analyses. A. G. POLLARD.

Composition of absorbing soil complex. A. T. TIULIN (Agr. Exp. Stat. Perm, No. 1, 1927, 1-28; Proc. Internat. Soc. Soil Sci., 1929, 4, 38-40) .--Parallel determinations of the saturation capacity (calcium) and humic content of the adsorbing complex of soils of various types are recorded. Increased humic content is coincident with increased adsorptive capacity, although the relationship is not uniform in all soils. In chernozem soils the ratio of carbon in the adsorbing complex (Gedroiz) to saturation capacity for calcium is constant. The value of the constant differs for different soil types. If the separated adsorbing complex after treatment with sodium chloride is coagulated with tervalent aluminium salts, no further exchange reactions in the complex can occur. This condition of the complex is designated "passive." Pretreatment with barium or calcium hydroxide renders the complex active, and the barium and calcium ions can take part in exchange reactions. A. G. POLLARD.

Alterations in the degree of acidity of soils by drying the samples. B. AARNIO (Bull. Agrogeol. Inst. Finland, 1928, No. 26; Proc. Internat. Soc. Soil Sci., 1929, 4, 27-28.)-Changes in the acidity of soils during drying are more rapid at higher temperatures, all soils examined becoming more acid. The reaction of moist soils at 25° increases rapidly at first but falls again later. Drying at 100° increases the acidity rapidly, the exchange acidity as well as the reaction of the water extract being affected. At higher temperatures (400°) a neutral clay became more acid and later more alkaline, approximating to the unheated soil. An acid clay, the reaction of which was unchanged at 400°, became less acid as the temperature was raised and was neutral at 1000°. Such reaction changes are most marked in heavy soils, are small in loams, and scarcely appreciable in sands. In soils rich in humus the changes are relatively smaller. The alteration of reaction probably results from the diminished total surface area brought about by drying, with the accompanying liberation of ions (acid salt-forming bases—Steenkamp), whilst at temperatures of 400° and upwards chemical reactions occur whereby the ions are again combined and the acidity decreases. A. G. POLLARD.

Physical properties of sugar cane soils and their value in practical agriculture. O. ARRHENIUS (Med. Proefstat. Java-Suikerind., 1928, 195-254; Proc. Internat. Soc. Soil Sci., 1929, 4, 59-60).—Comparison of reports of cane growers with mechanical analyses of numerous soils by the methods of Mohr and of Goldschmidt showed no relationship between the size of particles and the permeability, ease of working, or crop yields. Better correlation was possible when Atterberg's method was adopted for mechanical analysis. The calculation of hygroscopicity from mechanical analyses by Briggs' method proved valueless. Hygroscopicity is an important factor in soil examination. Using this value and the moisture content the total water supply available for the plant can be calculated. The water requirement for cane is 300 litres/kg. of plant (confirming Leather's values). A. G. POLLARD.

Relations between different properties of sugar cane soils. O. ARRHENIUS (Arch. Suikerind. Ned.-Indië, 1927, No. 48, 1197—1213; Proc. Internat. Soc. Soil Sci., 1929, 4, 89—90).—A relation exists between the reaction of a soil and its phosphate content, the latter being low in acid soils and high in alkaline ones, with a fairly constant value over the range  $p_{\rm H}$  6.5—7.5. This is ascribed to varying solubility, and therefore ease of removal by leaching, of the three simple phosphates of calcium. Nitrification in soils is not dependent on soil reaction, but varies directly with the phosphate content. The activity of the nitrifying organisms depends on the supply of phosphate.

A. G. POLLARD.

Soil acidity and sugar cane. O. ARRHENIUS (Arch. Suikerind. Ned.-Indië, 1927, No. 41, 1027–1043; Proc. Internat. Soc. Soil Sci., 1929, 4, 83).—Examination of numerous field soils together with culture experiments in which the soil reaction was artificially adjusted showed the maximum growth of sugar cane occurs at  $p_{\rm H}$  7. A. G. POLLARD.

Reaction of sugar cane soils in Java. O. ARR-HENIUS (Med. Proefstat. Java-Suikerind., 1927. No. 6207-6228; Proc. Internat. Soc. Soil Sci., 1929, 4, 87).—Liming of acid soils increases the yield of cane. Sugar yields were higher in cane grown on neutral or alkaline soils than on acid soils. Soil reaction and phosphate deficiency were related, and the lower yields from acid soils may be indirectly the result of this.

A. G. POLLARD.

Changes in some physical properties of soils brought about by lime. A. GEHRING (Z. Pflanz. Düng., 1929, 8B, 239—254).—The application of marl, quicklime, and slaked lime to soils increased their pore space without markedly affecting their absolute moisture contents. Such treatment of field soils led to increased soil temperature, variations in which showed a general relationship to the corresponding changes in pore space. The maximum and most definite effects of these calcareous materials were observed with quantities corresponding to lime required to produce a 70% saturation of the soils with bases. A. G. POLLARD.

"Kalkammon" [as fertiliser]. K. NEHRING (Z. Pflanz. Düng., 1929, 8B, 233–239).—No appreciable losses of nitrogen occurred during the storage under ordinary conditions of "Kalkammon" (a mixture of ammonium chloride with 30% of chalk). In pot experiments the material proved physiologically slightly acid, but in field trials the soil reaction was not affected. In normal soils the fertiliser value of "Kalkammon" was similar to that of ammonium sulphate or sodium nitrate, but in acid soils with sensitive plants (e.g., barley) it was not fully effective. A. G. POLLARD.

Iodine question in agriculture. W. GAUS and R. GRIESSBACH (Z. Pflanz. Düng., 1929, 13A, 321-425). -An extensive and critical review of existing knowledge is given, and numerous research reports are recorded. In field trials no definite increases in crop yields from iodine manuring were observed. The higher iodine contents of plants following iodine manuring were irregular and generally confined to the stems and leaves. In some cases actual decreases in the iodine contents of roots resulted from the iodine treatment. Sodium iodate was more effective than potassium iodide in increasing the iodine content of sugar beet. Among the plants examined spinach and lettuce had the highest iodine contents. Of the iodine manure applied only a very small proportion was absorbed by the plant (seldom more than 0.1%). Continuous heavy dosing of tomato soils with iodine during the growing period resulted in large increases in the leaf iodine content and smaller ones in the fruit. Under these conditions the greater part of the iodine enters the plants by the leaves. The absorption of iodine by leaves from iodine vapour is approximately 100 times as great as from potassium iodide solution. The accumulation of iodine by plants is in no way connected with any vital processes occurring in the plant tissues. The iodine in soil is mainly concentrated in the finer fractions. Considerable variations were observed in the iodine contents of sea coast soils. On light, permeable soils no permanent increase in iodine content resulted from manuring with seaweed. In recently dyked soils the iodine content was greatest in the upper (A-) horizons, but in older soils the tendency was for the greatest iodine accumulation to appear in the B-horizons. The iodine liberated during the burning of coal in furnaces etc. does not become evenly distributed throughout the atmosphere, but remains concentrated locally. It is precipitated only with rain, there being no appreciable absorption of iodine by alkali solutions exposed to the air. A. G. POLLARD.

Influences of the development of higher plants on the micro-organisms of the soil. II. Influence of the stage of plant growth on the abundance of organisms. R. L. STARKEY (Soil Sci., 1929, 27, 355—378; cf. B., 1929, 447).—Examination of numerous soils under field and greenhouse conditions show that growing plants tend to increase the number of soil organisms. This effect increases with the age of the plants, and differs with the nature of the plant and the species of soil organism. The greatest proportional increases were observed in the *B. radiobacter* group. Nitrogen-fixing bacteria, actinomyces, and filamentous fungi were but little affected. The influence on the soil organisms decreases after the death of the plants, but is not associated with the size of the plant or the extent of its root system. A. G. POLLARD.

Determination of the surface area of soils by adsorption of liquids. A. NEUGEBORN (Diss., Breslau, 1927; Proc. Internat. Soc. Soil Sci., 1929, 4, 55-56).—The Mitscherlich method for determining the surface area exposed by a soil is not only imperfect in technique but theoretically unsound. The process for measuring total surface area in soils by hygroscopicity is ad rsely criticised. The quantity of water

absorbed by soil is not proportional to the total surface exposed since capillarity, swelling phenomena, and hydration are also concerned, and there is no uniformity in the thickness of the aqueous film on the soil surface. A. G. POLLARD.

Determination of the degree of humification of forest litter. L. KRÓLIKOWSKI (Z. Inst. Gleboznawezego Univ. Poznavskiego, Wplyneto 21, XI, 1927; Proc. Internat. Soc. Soil Sci., 1929, 4, 37–38).— Robinson and Jones' method for examining the humification of soil constituents etc. was successfully applied to various forest litters. The transition from the upper horizon  $A_0$  to the sedimentary humus layer  $A_2$  is gradual, and the difficulty of accurate sampling of individual horizons prevents close correlation of the degree of humification with the age and genus of the forest trees or the natural conditions. A. G. POLLARD.

Errors in the determination of humus in soil. M. COUTURE (Giorn. Chim. Ind. Appl., 1929, 11, 149-150).-When soil contains dolomite or magnesite the combustion method of determining humus should be discarded, since it becomes difficult to expel all the carbonic acid by treatment with phosphoric acid, and excess of the latter renders troublesome the subsequent evaporation to dryness. Moreover, commercial phosphoric acid often contains sufficient proportions of organic matter to vitiate the results. In determining humus by the loss of weight on heating, the loss of water of crystallisation and of constitution undergone by normal mineral components of the soil may constitute a source of error; if the soil is slightly moistened with ammonium nitrate solution, oxidation of the humous material may be effected at a comparatively low temperature (in an oven at 250-300°), and subsequent treatment of the residue with ammonium carbonate solution not only reconverts the oxides formed into carbonates, but also replaces the water lost. Tests on kaolin confirm these views. Oxidation by means of permanganate and sulphuric acid is the most suitable method for determining humus if a number of determinations are made, but chlorides and other extraneous substances reacting with permanganate must be present in only minimum quantity. Calculation of the organic matter from the proportion of nitrogen in soil is not to be recommended, as the percentage of nitrogen in humus is variable and humus readily absorbs appreciable amounts of nitrogenous compounds. Any method is subject to the sampling error, since at most 2 g. are taken for analysis. The presence in the soil of vegetable and animal residues may introduce a considerable error, which may be minimised by the following procedure: 10 g. or more of the soil are added gradually to a 50- or 100-c.c. cylinder containing saturated sodium sulphate solution and standing in a flat dish. When most of the soil has settled, more of the solution is added to make the liquid overflow and carry the lighter matter with it. The liquid is stirred occasionally with a rod and the process is repeated. The matter thus removed is collected on either an ordinary or a Gooch filter, washed, and dried at 100°. The carbon of the residue, calculated as humus, may amount to 5-10% of the total present T. H. POPE. in the soil.

Permanganate method for the determination of the oxidisability of organic matter in water and aqueous soil extracts containing chlorides. J. N. ANTIPOV-KARATAËV (State Inst. Exp. Agr., Leningrad, Bull. Bur. Soils No. 3, 1928; Proc. Internat. Soc. Soil Sci., 1929, 4, 40-42).-To the sample (50 c.c.) are added 3 c.c. of sulphuric acid (1:3) and a weighed excess of silver sulphate to precipitate the chlorides (determined in a separate sample). After at least 1 hr. (the liquid being occasionally stirred) 10 c.c. of 0.01N-potassium permanganate are added, while stirring, and the liquid is heated gently for 10 min. If more permanganate is required further heating is necessary. 10 c.c. of 0.01Noxalic acid are then added, and when the liquid has cleared completely the excess is titrated back with permanganate. Blank tests are necessary.

A. G. POLLARD.

Application of the strychnomolybdic process to the determination of phosphoric acid in soil. C. ANTONIANI and S. BONETTI (Giorn. Chim. Ind. Appl., 1929, 11, 154-155; cf. A., 1928, 979, 1265).-20 g. of the soil are boiled for 1 hr. with 30 c.c. of hydrochloric acid (d 1.18), 20 c.c. of nitric acid (d 1.40), and 50 c.c. of water. The cooled solution is made up to 500 c.c. with water, mixed, and filtered through a pleated paper, 50 c.c. of the filtrate being neutralised to phenolphthalein with 10% sodium hydroxide solution and the precipitated basic salts redissolved by addition of a few drops of 10% nitric acid. The clear liquid is mixed with 45 c.c. of the strychnomolybdic reagent and the precipitate, after 1 hr., is collected, washed with 100 c.c. of nitric acid (10 c.c. of acid  $[d \ 1 \cdot 40]$  made up to 100 c.c.), and dried in a steam-bath to constant weight. Multiplication of the weight of precipitate by 0.0257 gives P<sub>2</sub>O<sub>5</sub>, the volume occupied by the soil being allowed for by multiplying by 0.983 (experimental figure). To determine  $P_2O_5$  soluble in 1% citric acid solution, 100 g. of the soil are treated in a Stohmann flask with the citric acid solution until all carbonate-carbon dioxide is expelled, a further quantity of 750 c.c. of the solution being then added. The flask is shaken for 5 hrs. in a revolving shaking apparatus and then kept for 12 hrs., after which 250 c.c. of the liquid (filtered if necessary) are evaporated to dryness on a water-bath. The residue is gently calcined with a few crystals of ammonium nitrate until all organic matter is oxidised, the ash being then heated with a little water and a few drops of nitric acid for some minutes on a water-bath, and the liquid is filtered. The neutralised filtrate is treated as in the determination of the total phosphoric acid. Test T. H. POPE. analyses yielded satisfactory results.

Gravimetric determination of carbonates in soils. N. A. CLARK and E. R. COLLINS (Soil Sci., 1929, 27, 407-414).—The customary method involving treatment of the soil with acid and the removal of the carbon dioxide by aspiration is modified. Ascarite is used as an absorbent and the air current is previously dried with phosphorus pentoxide. Dilute hydrochloric acid (1:10) is used and the soil suspension stirred instead of shaken during the aspiration. The operation is carried out at room temperature. At 50° there is a slight increase in the amount of carbon dioxide obtained. A. G. POLLARD. Acidimetric titration of silica and aluminium with an extension of the theory of acidimetric titrations. S. KÜHN (Sonderh. Ung. Kön. Geol. Anstalt Budapest, 1928, 34; Proc. Internat. Soc. Soil Sci., 1929, 4, 34).—A rapid method (10—15 min.) is described for determining silica in general analysis and aluminium in soil analysis in the presence of other soil constituents. A. G. POLLARD.

Detection and significance of manganese dioxide in soil. W. O. ROBINSON (Soil Sci., 1929, 27, 335— 350).—Manganese dioxide in soils decomposes hydrogen peroxide so much more rapidly than do other normal soil constituents that its presence in soil may be established by this means. Determinations are not satisfactory, since the rapidity of evolution of oxygen is dependent on the fineness of division of the manganese dioxide. The greater part of the manganese dioxide in soil appears in the silt fraction, a smaller amount in the sand, and none in the clay or colloidal fraction. In the soils examined manganese dioxide appears to be formed in concretory deposits as a result of the precipitating effect of chalk. A. G. POLLARD.

The seedling method [for determining soil nutrient values]. H. NEUBAUER (Z. Pflanz. Düng., 1929, 8B, 219-232).—The technique and practical application of the Neubauer and Schneider's original method (*ibid.*, 1923, 2A, 329) are discussed.

A. G. POLLARD.

Apparatus for mechanical analysis of soils. J. PADEREWSKI (Rocz. Nauk Koln. i Lesnych, 1928, 19; Proc. Internal. Soc. Soil Sci., 1929, 4, 48).—A modified elutriator apparatus is described in which the three vessels are arranged directly above one another, thus affording simplicity of manipulation and ease of repetition of determinations with the same soil suspension. A. G. POLLARD.

Efficient soil tube jack. C. A. TAYLOR and H. F. BLANEY (Soil Sci., 1929, 27, 351-353).—The essential point of the jack is a lever operating a hinged conical grip closing on the soil tube at each lift and releasing and sliding down the tube with the return motion of the lever. A. G. POLLARD.

Differential apparatus for measuring the volume of soils. J. PADEREWSKI (Stat. agric. d'exp. Kutno, 1925; Proc. Internat. Soc. Soil Sci., 1929, 4, 56—57).— Apparatus is described for the measurement of soil volumes by determining differences in air pressure resulting from the introduction of the soil sample into a closed space. A. G. POLLARD.

See also A., June, 667, Determination of phosphoric acids (SOMEYA; STOLLENWERK and BÄURLE; DWORZAK and REICH-ROHRWIG). 719, Aluminium toxicity (MCLEAN and GILBERT). 728, Absorption of carbon dioxide by roots (BERGAMASCHI). Growth of the tomato (NIGHTINGALE and others). Behaviour of the olive under uranium radiations and ionised air (PETRI). Influence of nitrate ion on growth of summer wheat (GOEDEWAAGEN). Nutrition of higher plants with ammonia (PRIANISCHNIKOV). Nitrogen metabolism of forest soil (FEHÉB).

#### PATENTS.

Dry disinfectant for seeds. L. and F. MEYER (CHEM. FABR. L. MEYER) (B.P. 286,220, 21.2.28. Denm., 28.2.27).—Iodine compounds such as mercuric iodide are mixed with known fungicidal materials. *E.g.*, a mixture of talc with mercuric chloride and iodide is used against bunt in wheat, stripe disease in barley, and smut in oats. Alternatively, mixtures of potassium iodide and mercuric chloride may be used. A preparation containing talc, mercuric chloride, potassium iodide, and tricalcium arsenate is used against crown gall in roots. A. G. POLLARD.

Disinfection of seeds and composition therefor. E. W. SCHMIDT, Assr. to J. D. RIEDEL-E. DE HAEN A.-G. (U.S.P. 1,711,545, 7.5.29. Appl., 2.7.26. Ger., 20.11.25). —A mixture of powdered calcium silicofluoride and copper carbonate is claimed. H. ROYAL-DAWSON.

Fertiliser. G. BARSKY and F. W. FREISE, ASSTS. to AMER. CYANAMID Co. (U.S.P. 1,695,081, 11.12.28. Appl., 25.6.23).—Leather scrap, or other nitrogenous organic waste material, is dissolved in sulphuric acid, and calcium cyanamide is stirred in. A porous solid fertiliser is obtained. C. HOLLINS.

**Production of fertilisers.** NORSK HYDRO-ELEK-TRISK KVAELSTOF A./S. (Norw. P. 42,724, 9.9.24. Addn. to Norw. P. 41,452).—The mixture obtained by treating with nitric acid mineral silicates containing potassium, *e.g.*, leucite, is evaporated to dryness and then fused to expel nitrous gases; the gases are worked up to nitric acid and the residue is used as a fertiliser.

L. A. Coles.

[Production of phosphatic] fertilisers. COMP. DE L'AZOTE ET DES FERTILISANTS SOC. ANON. (Swiss P. 122,821, 23.7.26).—Calcium phosphate etc. is treated at 20—100° with an acid, e.g., sulphuric acid, in the presence of the ammonium salt of an acid which forms an insoluble calcium salt, e.g., ammonium sulphate, and, if necessary, potassium salts, e.g., potassium sulphate. L. A. COLES.

Emulsifiable oil (U.S.P. 1,695,197).—See II. Spent sulphite liquors (U.S.P. 1,710,272).—See V. Treatment of phosphorites (Russ. P. 3464).—See VII.

# XVII.—SUGARS; STARCHES; GUMS.

Sugar factory filter cloths. I—III. K. ŠANDERA (Z. Zuckerind. Czechoslov., 1929, 53, 321—329, 457— 463, 483—492).—I. On a basis of 1000 dzn. of roots, the total filtering area used in beet-sugar factories is  $27 \cdot 2 - 68 \cdot 6 \text{ m.}^2$ ; the frequency of re-dressing is 4—40 days of the campaign, and the life of the cloths is 10—80 days. Jute cloths last on the average 10—14 days, and cotton up to 80 days. About 54% of the material used is cotton, 31% a mixture of cotton and jute, and 15% a mixture of linen and jute. Most factories now prefer cloths having the strengthened edge.

II. Filter-cloths of cotton, linen, jute, and of mixtures of these were submitted to various physical tests, as tearing strain, extension, rate of flow of water and molasses, and were also examined for hygroscopicity, density, regularity, etc., but these observations failed to lead to any definite standards by which their filtering and wearing qualities could be judged.

III. Cotton and linen cloths during use lost 63% of their original strength, as determined by the tearing strain, and jute about 46%, though these values are subject to wide variations, even in the same factory. Extension tests showed use to diminish this value, so that a cotton cloth which originally had a value of about 4% showed after use one about 1% lower. Figures for the rate of flow showed an immense retardation for a mixture of cotton and linen, but for pure cotton and jute fabrics the differences were considerably less, showing, in fact, sometimes a greater rate of flow. In general, these tests are complicated by other factors bearing on the problem of filtration. J. P. OGILVIE.

Combined milling and diffusion. W. R. McALLEP (Internat. Sugar J., 1929, 31, 279-280).—It seems improbable to the author that more economical means will ever be developed for extracting the first 80-90% of the sugar from cane than pressure in a couple of mills with ordinary maceration. Subsequent to this, however, the diffusion principle should be applied by removing the bagasse from the mill, and extracting the greater part of the remaining sugar by circulating the maceration water systematically. In this way extraction could be improved at moderate cost, whilst grinding at a greater rate than at present without the installation of costly additional mills is possible. J. P. OGILVIE.

Elimination of colloidal matter [from beet juices]. R. J. BROWN (Sugar Press, 1929, 13, 14—15). —As a positive colloid for the removal from beet juice of the negatively-charged colloidal matter, chromium oxide hydrosol proved to be the most effective, the iron oxide preparation being the least so. Alumina hydrosol also gave good results, and when equal volumes of it and beet juice were mixed a good coagulation resulted, the filtrate containing only a negligible quantity of colloidal matter. J. P. OGILVIE.

De Vecchis' process for extracting sugar from beet. I. GIORDANO (Giorn. Chim. Ind. Appl., 1929, 11, 155-163).—Descriptions are given of earlier attempts to dry beet, and of the plant and detailed working, including the economics, of the De Vecchis process.

T. H. POPE.

Evaporation of [beet] juices in the presence of active carbons. V. STANÊK and P. PAVLAS (Z. Zuckerind. Czechoslov., 1929, 53, 493—499).—A small amount (0.2%) of decolorising carbon present in the thin juice during its evaporation acted very favourably in diminishing its final colour. "Carboraffin" gave better results than "Standard Norit." When the active carbon was used in combination with sodium sulphite (0.01%), a juice resulted which was distinctly lighter in colour than the original.

J. P. OGILVIE.

Effect of carbon monoxide and other impurities of saturation gas on carbonatation [of sugar solutions]. R. SIGMUND (Z. Zuckerind. Czechoslov., 1929, 53, 473–479).—Using saturation gas containing 40% CO<sub>2</sub> throughout, it was observed that the most rapid saturation was achieved with the pure gas. It was diminished with increasing amounts of carbon monoxide, so that with 10% of this impurity the rate fell to less than half. Even 0.5% appreciably retarded the operation. Hydrogen sulphite and tarry matters exerted no marked effect. J. P. OGILVIE.

Determination of sugar in bagasse by cold extraction : new figure for milling control. H. EGETER (Archief, 1928, 36, II, 691—729).—In the coldextraction method of Khainovsky and Egeter (Med. Java-Proefstat., 1927, No. 4) for the determination of sucrose in bagasse, a lower polarisation is obtained than in the ordinary method, this having been proved to be due in the new procedure to the presence of intact cells incapable of extraction without heat. Based on this observation, the author elaborates a method of estimating the quantity of juice left in the unruptured cells after milling, and it is proposed that this figure should be used as a standard for the control of milling in order to indicate the efficiency of crushing.

#### J. P. OGILVIE.

Analysis of cane sugar. P. HONIG (Archief, 1928, 36, II, 639-647).—Reducing sugars are determined by Schoorl's method (cf. Internat. Sugar J., 1919, 21, 578, 621) after clarification with normal lead acetate, followed by sodium phosphate. The size of grain is found by a series of standard Tyler sieves giving fractions from above 1.65 mm. down to less than 0.3 mm. (dust). Colour determination is expressed by values obtained by means of the König-Martens spectrophotometer after carefully filtering the solution and adjusting its  $p_{\rm H}$  to 7.0, measurements being made at six different wavelengths. J. P. OGILVIE.

See also A., June, 682, Oxidation of dextrose by alkaline copper solutions (LUNDIN). 683, Synthesis of sucrose (ZEMPLÉN and GERECES). Condensation of dextrose and lævulose ; *isosucrose* (IRVINE and others). 702, Beet-saponin (REHORST). 722, Sugarhydrolysing enzymes (WEIDENHAGEN).

Sugar cane soils. ARRHENIUS.-See XVI.

#### PATENTS.

Dehydration of vegetable substances [e.g., sugarbeet slices] of organic character. B. J. OWEN, Assr. to SUGAR BEET & CROP DRIERS, LTD. (U.S.P. 1,713,619, 21.5.29. Appl., 5.11.27. U.K., 5.11.26).—See B.P. 285,115; B., 1928, 311.

Reduction of sugars to alcohols (U.S.P. 1,712,952).—See XI.

#### XVIII.—FERMENTATION INDUSTRIES.

Modern malting methods. G. CHABOT (Woch. Brau., 1929, 46, 208—211).—The alkaline steep advocated by Moufang is criticised, and experimental results are given which indicate that a steep-water adjusted to  $p_{\rm H}$  6—7 is best as regards uptake of water by the barley. With slightly more alkaline or acid steepwaters, the  $p_{\rm H}$  tends to adjust itself to the above value, but with too acid waters ( $p_{\rm H}$  about 4) this may be slight or the water may become still more acid. This effect is more marked when sulphurous acid is used for neutralising a calcium carbonate water than when sulphuric acid is used. The use of alkaline steep-water leads to darkening of the grain. F. E. Day.

Storing and redrying malt. G. D. CLARKSON (J.

Inst. Brew., 1929, 35, 282-287).-The moisture content of malt which is to be crushed for brewing should not much exceed 3%. For the construction of bins for the storage of malt, wood alone or brick or concrete with wood lining is the most satisfactory material, for it does not damage the grain and is impervious to moisture. Probably the best method for redrying malt which has taken up moisture during storage is in a "drum." Heat must be applied slowly during the first 12 hrs., and the temperature raised to 38°. In 18 hrs. the temperature should reach 49° and in 24 hrs. 64°, at which stage a strong draught is necessary. For amber or thickskinned malt 77-82° may be reached in 48 hrs., but for pale malt those temperatures should not be reached until 72 hrs. have elapsed. By redrying, the colour increases 1-3 points, the matters soluble in cold water rise slightly, and the diastatic power remains almost stationary. C. RANKEN.

Use of Irish moss as copper finings [in brewing]. C. RANKEN (J. Inst. Brew., 1929, 35, 287-291).-The flocculation of peptone in boiling wort by means of Irish moss remains practically uninfluenced by the concentration of the peptone and other wort constituents. It is sensitive to the reaction of the wort and to the concentration of the moss per barrel of wort. To obtain optimum flocculation, a greater amount of free acidity in the wort must be met with the addition of a greater amount of Irish moss per barrel. Should a greater amount of moss be added than that required for the  $p_{\rm H}$  value of the wort, the resulting wort will become hazy on the production of acid during fermentation, with possible prejudicial effects on the yeast. The insoluble cellulosic skeleton of the moss is also an important factor in the clarification of the wort. C. RANKEN.

Biology of sarcina. J. FUCHS (Woch. Brau., 1929, 46, 203-208, 213-215).-The development of sarcina in beer is not directly due to incomplete saccharification in the mash tun, but may be so caused indirectly owing to weakening of the yeast. For the cultivation of the organisms slightly alkaline  $(p_{\rm H} 8)$  media are most favourable especially in presence of ammonia, and a test for slight sarcina infection is based on this. In beers the presence of complex proteins is favourable to the growth of sarcina, hence badly modified or highnitrogen malts and conditions which favour poor proteolysis in the mash tun should be avoided. Especially favourable are the products of the autolysis of yeast. Experiments are described illustrating these points and indicating a direct antagonism between yeast and sarcina, the former being apparently capable of excreting a lysin destructive to sarcina. There is no evidence of parasitism of sarcina on, or symbiosis with, yeast.

F. E. DAY.

Wine distillates and wine brandies. E. RUPPIN (Z. Unters. Lebensm., 1929, 57, 243—244).—It is shown on theoretical considerations based on the law of Guldberg and Waage that, contrary to the statement of Büttner and Miermeister (B., 1929, 373), unadulterated wine brandy may contain much less than 100 mg. of ester in 100 c.c. of pure alcohol. The necessity for tasting tests in evaluating these products is emphasised.

W. J. BOYD.

Examination and evaluation of wine distillates and wine brandies. H. WÜSTENFELD and C. LUCKOW (Z. Unters. Lebensm., 1929, 57, 242—243).—Contrary to the views of Büttner and Miermeister (B., 1929, 373), the necessity of tasting tests in evaluation of still wines and their distillation products is emphasised and the fixing of minimum values is considered undesirable. The value of analytical data is admitted in conjunction with tasting tests on the individual fractions obtained by rectification. W. J. BOYD.

Detection of fruit wine in wine by identification of dibenzylidenesorbitol. C. VON DER HEIDE and K. HENNIG (Z. Unters. Lebensm., 1929, 57, 240-241).— The observations of Werder (Mitt. Gebiete Lebensm. u. Hyg., 1928, 19, 294) that fruit wine always contains sorbitol whilst wine never does were confirmed. It is necessary, however, in using Werder's method to examine the precipitate by washing it with alcohol and ether to remove water and various organic substances and then extracting the dibenzylidenesorbitol with benzene, recrystallising it from benzene, and determining its m.p., which should be 162° (uncorr.). The presence of tribenzylidenemannitol, m.p. 213-217° (uncorr.), may otherwise lead to wrong conclusions. W. J. Boyd.

Detection of *iso*propyl alcohol in presence of acetone [in spirit]. W. MEYER (Chem.-Ztg., 1929, 53, 410-411).-50 g. of spirit are distilled and the first 5 c.c. of distillate are tested for methyl alcohol and acetone. The second fraction of 20 c.c. is divided, half being oxidised with dichromate and sulphuric acid. An increase in acetone content after oxidation indicates the presence of *iso*propyl alcohol in the original spirit.

J. S. CARTER.

See also A., June, 721, Malt amylase (SABALITSCHKA and WEIDLICH). Takadiastase (OHLSSON and SWAE-TICHIN). 722, Enzymes and light (OYA). Sugarhydrolysing enzymes (WEIDENHAGEN). Action of invertase (COLIN and CHAUDUN). Barley-malt catalase (CHARMANDARJAN). 724, Alcoholic fermentation with dried yeast (KISCH and LEIBOWITZ). Fermentative bacteria in maceration juice (KOSTYTSCHEV and SCHULGINA). Conversion of acetic acid by *Mucor stolonifer* (BUTKEVITSCH and FEDOROV).

Starch value of frozen potatoes. LAMPE and KILP. —See XIX.

# PATENTS.

Yeast food [for bread-making]. O. R. BROWN (U.S.P. 1,712,025, 7.5.29. Appl., 22.8.27).—The food is composed of potassium nitrate, calcium phosphate, and magnesium sulphate. H. ROYAL-DAWSON.

Manufacture of sparkling wine. C. M. CHARTIER (F.P. 628,964, 8.2.27).—Wine with added sugar is fermented with yeast at 25—28° in an enamelled steel vessel in which the pressure of carbon dioxide is allowed to reach and is retained at 5 atm. The fermented liquid is filtered under air pressure into a collection vessel where it is chilled to 2—5° and saturated with carbon dioxide. After repeated filtration under pressure of carbon dioxide the wine is bottled. The process requires 8—10 days and yields a wine ready for immediate consumption. C. RANKEN.

Manufacture of fruit wine. P. K. ERBEN (Swiss P. 122,802, 2.8.26).—The wine, which retains its brilliancy and has a low alcohol content, is prepared by partly fermenting pressed juice and, after removal of the yeast, adding a sulphite to destroy bacteria. After clarification with a liquid containing tannin, the juice is kept cold to retard fermentation, and as soon as the yeast has separated is filtered through an ultra-filter so that a secondary fermentation may be avoided. C. RANKEN.

# XIX.—FOODS.

Action of chlorine on wheaten flour in treatment with gologas. M. VUK and A. GÖMÖRY (Z. Unters. Lebensm., 1929, 57, 177-191; cf. Vuk and Spanyár, B., 1929, 299).-Chlorine forms with the starch neither a chemical nor an adsorption compound which has any influence on the properties of the flour. It alters the balance between the gliadin and glutelin, so that it becomes difficult to obtain the gluten by washing the flour. The proportion of the various amino-acids in the gliadin and glutelin of untreated, chlorinated, and overchlorinated flours, respectively, were determined as far as possible. The greatest decreases were in the quantities of amide-nitrogen and glutamic acid, and to a less extent of aspartic acid and arginine. The water-soluble nitrogen was found to increase on chlorination. It is concluded that the glutamic and aspartic acids, in the form of glutamine and asparagine, are so united to other amides, mono- or di-amino-substances, that they are set free on treatment of the flour with gologas, and become soluble as free amino-acids or their hydrochlorides. The chlorine forms addition products with the unsaturated glycerides of the flour fat, and the carotin is bleached by conversion into the water-soluble dicarotin. W. J. BOYD.

Electrical conductivity of milk. I. Detection of abnormal milk from diseased cows. J. KRENN (Z. Unters. Lebensm., 1929, 57, 148—177).—Determination of the electrical conductivity of milk is shown to be of great utility in the detection of milk from cows with diseased udders. Such milk has invariably a higher electrical conductivity than normal milk, owing to its higher sodium chloride content. W. J. BOYD.

Rapid analysis of butter. N. CHARLIERS (Chim. et Ind., 1929, 21, 931-936).-In the saponification n-propyl alcohol is used, and under the specified conditions this is salted out along with the higher fatty acids. The small quantity remaining in the aqueous phase has no disturbing effect. The butyric acid removed in the separated alcohol is made constant in quantity by adding potassium stearate, equivalent to 1 g. of stearic acid, to the solution after saponification. Butter (5 g.) is heated under reflux for 20 min. with 25 c.c. of 6% caustic potash solution in n-propyl alcohol, 0.2 c.c. of a 2% solution of phenolphthalein in n-propyl alcohol is added, and the excess of alkali is titrated with N-sulphuric acid. The saponification value is calculated in the usual way. If n c.c. of N-sulphuric acid have been used, there are added further: (a)  $(31 \cdot 8 - n)$  c.c. of water; (b) 100 c.c. of a solution of 10 g. of stearic acid in 4 c.c. of 75% caustic potash solution, mixed with 10 c.c. of n-propyl alcohol and made up to 1 litre; (c) 10 c.c. of a solution of 100 g.

of coconut oil in 40 c.c. of 75% caustic potash solution, mixed with 100 c.c. of n-propyl alcohol and made up to 1 litre; (d) 5 c.c. of 33% sulphuric acid; (e) 15 g. of anhydrous sodium sulphate; and (f) a pinch of kieselguhr. The mixture is shaken, allowed to rest for 20 min., and 125 c.c. are filtered off. Then 50 c.c. of distilled water are added with some pieces of pumice, and 110 c.c. of distillate are collected and titrated with 0.1N-caustic soda. The butyric acid number is  $(k-t) \times 1.75$ , where k and t are the numbers of c.c. of 0.1N-caustic soda used in the determination and in a blank test, respectively. Cacao butter should be used for the blank experiment. The values obtained for the butter content by this method are quite as concordant with those calculated from the Leffmann-Beam value, as are those obtained by the methods of Kuhlmann and Grossfeld (B., 1926, 447) and Van Raalte (B., 1926, 563). W. J. BOYD.

Chemical detection of vitamin-C. B. GLASSMANN and A. POSDEEV (Z. Unters. Lebensm., 1929, 57, 191— 200).—The Bezssonov reaction (B., 1921, 747) supposed to be due to vitamin-C is given by tannins at ordinary temperatures, and by carbohydrates and other plant substances at the temperature of the boiling waterbath. Until the vitamins have been isolated and their reactions studied, their detection is only possible biologically. W. J. BOYD.

Effect of fumigation by hydrogen cyanide on fresh fruit and vegetables. P. BUTTENBERG (Z. Unters. Lebensm., 1929, 57, 204-211).-No effect on plums, grapes, shallots, and onions has been established, and the effect on parsley and tomatoes is doubtful. Early potatoes do not withstand the treatment, but winter potatoes are resistant. In sprouted, wintered potatoes the germ and surface layer of the tuber may be destroyed. On aeration of potatoes after fumigation, an effect like that of frost may be produced. Freshlyharvested Swedish turnips are less resistant than those kept through the winter. Apples, bananas, pears, oranges, lemons, quinces, cucumbers, different kinds of cabbage, carrots, radishes, lettuces, rhubarb stalks, beets, turnips, celery, spinach, and comfrey are damaged. The damage is not always so great as to exclude an early consumption of the produce, and may not appear for W. J. BOYD. some weeks.

Loss of iodine and other inorganic substances from spinach in the usual cooking process. A. MIERMEISTER (Z. Unters. Lebensm., 1929, 57, 235— 239).—A loss of 44% of the total mineral matter was observed in cooking 2 kg. of spinach in 3 litres of boiling tap water to which 15.8 g. of salt had been added, the water being finally poured off and the vegetable washed with distilled water. Of this loss 59% was due to K<sub>2</sub>O, amounting to 57% of the quantity of that base originally present, but the cooked and washed spinach still contained a notable excess of inorganic bases over acids. 100 g. of fresh spinach contained 0.044 mg. of iodine, of which 0.0035 mg. was lost in cooking. The iodine appears to be organically combined, and consequently insoluble. Methods of analysis are described.

W. J. BOYD. Determination of starch value of sound and frozen potatoes, with reference to their use for distilling and drying. B. LAMPE and W. KILP (Z. Spiritusind., 1929, 52, 177–178).—Two samples, partly frozen in the clamp, were obtained, one apparently sound, the other softened and attacked by bacteria. The starch contents by (1) calculation from sp. gr., (2) acid hydrolysis and Fehling's solution, (3) hydrolysis, fermentation, and determination of the alcohol, and (4) subtracting  $5\cdot8\%$  from the dry matter, were, respectively,  $16\cdot7$ ,  $16\cdot97$ ,  $16\cdot82$ , and  $16\cdot77\%$  on the sound and  $12\cdot0$ ,  $23\cdot45$ ,  $24\cdot26$ , and  $24\cdot75\%$  on the unsound sample. The applicability of the determination from the sp. gr. and the effect of the damage resulting from freezing on the value for distilling and drying are discussed. (Cf. B., 1929, 338.) F. E. DAY.

Rapid determination of the oil content of paprika. J. DÖMÖTÖR (Z. Unters. Lebensm., 1929, 57, 239—240). —The material  $(2 \cdot 5 \text{ g.})$  is shaken for 1 hr. with 40—60 c.c. of ether, or the quantities may be modified according to the quality of the product, the relation of material to solvent being kept the same. The solvent is decanted off through three filter papers previously wetted with ether. The filtrate is passed through the filters a second time, and filters and residue are well washed with ether. The clear filtrate is transferred to a tared flask, the ether evaporated off, the oil dried at 105°, and weighed. The whole procedure is completed in 4 hrs. W. J. Boyp.

Rapid method of detecting lemonade containing saccharin. J. VONDRÁK (Z. Zuckerind. Czechoslov., 1929, 53, 501—503).—Lemonade sweetened with saccharin can be detected by its low refraction or low density. The former may be determined by the immersion or sugar refractometer, and the latter by the saccharometer or by means of floats made of approximately equal parts of carnauba wax and asphalt, adjusted so that they neither float nor sink in 6% sugar solution. This concentration may be taken as the minimum allowable in lemonade. W. J. BOYD.

Goose-liver preparations. J. PRITZKER and R. JUNGKUNZ (Z. Unters. Lebensm., 1929, 57, 212–216).— Definitions of "pâté de foie gras" by various authorities are quoted, and analyses of various products of this nature from different sources are given. W. J. BOYD.

Examination and evaluation of cray-fish soups. E. BATER and H. BARSCH (Z. Unters. Lebensm., 1929, 57, 224—234).—Methods for the detection of artificial colouring matter in cray-fish soups are described involving extraction of the pigment with alcohol and testing of its affinity for wool, its reaction with concentrated sulphuric acid, and its behaviour in ultra-violet light, in which solutions of the natural colouring matter show fluorescence and those of synthetic dyes do not. Microscopical examination of the foodstuff itself and observation of its taste and odour when cooked are useful in the examination of these products. W. J. BOYD.

Iodine values. GIRAL. Comestible flours. ANDRÉ.—See XII. Germicidal washing solutions. MYERS.—See XXIII.

# PATENTS.

Maturation of wheat, maize, pulse, and other carbohydrate-containing cereals and seeds, and darkness.

the flour or other similar products derived therefrom. R. W. DUNHAM (B.P. 311,121, 30.5. and 5.6.28).-Apparatus is described for artificially continuing the ripening process of the endosperm of grain, comprising several compartments which the product is made to

traverse, and in which it may be repeatedly subjected in one to heat and light, and in the next to cold and W. J. BOYD.

Making of cheese. H. P. KERNEN (U.S.P. 1,708,099 and 1,708,100, 9.4.29. Appl., [A] 18.2.27, [B] 21.11.27).-Sterile cheese is made directly in one continuous process by coagulating the milk, forming the curds, and then rapidly ripening by (A) developing the requisite acidity in the curds by artificial means in a relatively short space of time, viz., by heating for 12 hrs. at 30-40°, (B) adding  $\frac{30}{8}$  by wt. of each of calcium phosphate, calcium lactate, and ammonium citrate. The whole is then sterilised by heat in the presence of an emulsifying agent while stirring, and allowed to cool.

F. R. ENNOS.

Manufacture of cheese. W. D. RICHARDSON, Assr. to Swift & Co. (U.S.P. 1,711,032, 30.4.29. Appl., 26.7.27).-Dried milk powder is mixed with whole milk to form a mixture having approximately the moisture content desired for the finished cheese, and the product is coagulated. W. J. BOYD.

Preservation of eggs. A. V. STEWART, and FARMA CREAM PRODUCT CO., LTD. (B.P. 311,877, 24.3.28).--Eggs are coated with an 8% solution of celluloid in glycol monoethyl ether. The solution may be coloured if desired. Various methods of application are described. It is claimed that eggs so preserved remain fresh for 12 months. E. B. HUGHES.

Food product and its production. CALIFORNIA FRUIT GROWERS' EXCHANGE (B.P. 283,580, 3.1.28. U.S., 15.1.27) .- Prior to dehydration of fruit juice, 4% or less of a gum-like hydrophilic colloid such as pectin, agar, or gelatin is dissolved in it, so as to suppress hygroscopicity in the fruit juice solids. W. J. BOYD.

Percentage composition of diluted substances (B.P. 285,383).—See I. Yeast food (U.S.P. 1,712,025). -See XVIII.

# XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Buffer capacities of acacia and tragacanth. J. C. KRANTZ, JUN. (J. Amer. Pharm. Assoc., 1929, 18, 469-473) .- With emulsions prepared with acacia the buffering action of acacia on acid or alkali added to the emulsion influences the stability of the emulsion. The buffer capacity is more effective in the neutralisation of acids, the instability of the emulsion beginning on the alkaline side at a lower concentration of alkali than it does on the acid side with a corresponding concentration of acid. Dilution with water has little influence on the  $p_{\rm H}$  of acacia solutions. With tragacanth the buffering effect does not appear to have as great an influence on the stability of the emulsion as does acacia. The Van Slyke " 3" (cf. A., 1922, i, 893) for acacia in 0.1M-solution at  $p_{\rm H}$  3 is 0.034. E. H. SHARPLES.

Emulsifying properties of certain salts of arabic acid. J. C. KRANTZ, JUN., and N. E. GORDON (J. Amer.

Pharm. Assoc., 1929, 18, 463-468).-Metallic salts of arabic (from acacia), oleic, valeric, gluconic, i-galactonic, dihydroxystearic, salicylic, and gallic acids have been prepared and their emulsifying properties compared. An explanation for the production of oil-in-water emulsions by acacia and tragacanth is proposed.

E. H. SHARPLES.

Comparative study of methods of determination of citral in lemon oil ; constants of the Spanish oil. O. FERNÁNDEZ and A. MOSCARDO (Anal, Fís. Quím. [Tecn.], 1929, 2, 265-278).-The methods of Walther (B., 1900, 78; 1901, 289), and of Bennett for the determination of citral, using hydroxylamine hydrochloride, yield high results, whilst that of Bennett and Salamon (B., 1928, 68) gives concordant, but slightly high results. Tiemann's method, based on the use of sodium sulphite and titration of the liberated alkali, yields high results. The method of the U.S. Pharmacopœia, using phenylhydrazine, is satisfactory, but that of Ardagh and Williams (A., 1926, 189) vields low results. The following data are recorded for Valencian lemon oil:  $d_{15}^{15}$ 0.8587 - 0.8673,  $n_{\rm D}^{20}$  1.473,  $[\alpha]_{\rm D}^{20}$  -68.5° to -69.17° citral content  $2 \cdot 4 = 2 \cdot 65\%$ . Oil from unripe fruit had  $d_{\frac{15}{9}}^{15} = 0.8577$ ,  $n_{\mathrm{D}}^{20} = 1.474$ ,  $[\alpha]_{\mathrm{D}}^{20} = -67 \cdot 3\%$ , and a noticeably higher citral content  $(3 \cdot 2\%)$ . R. K. CALLOW.

See also A., June, 666, Ultra-violet colorimetry and fluorescent substances (EISENBRAND). 668, Determination of arsenic (VILLECZ; GNESSIN). 676, Decoic acids as bactericides (STANLEY and others). 707, Caffeino-salicylic acid (SCHOORL). Alkaloids of Angostura bark (TRÖGER). Microchemical reactions of physostigmine (WAGENAAR). Acids from brucine (WIELAND and MÜNSTER). 708, Determination of brucine and analysis of nux vomica (KLJAT-SCHKINA and STRUGADSKI). New strychnos alkaloid (WIELAND and OERTEL). 709, Apomorphine alkaloids (CALLOW and others). Sulphur derivatives of aromatic arsenicals (EVERETT). 710, 10-Chloro-5:10dihydrophenarsazine and its derivatives (GIBSON and JOHNSON). 711, Organic compounds of mercury (NESMEJANOV). 725, Mercuric oxycyanide compounds and Gonococcus (TAKAORA). 729, Methyl alcohol from tobacco smoke (NEUBERG and KOBEL). Determination of nicotine in fresh green tobacco (BODNÁR and NAGY). 730, Phloroglucinol drugs (PEYER and LIEBISCH).

# PATENTS.

Manufacture of lipoids, particularly phosphatides. "PHARMAGANS" PHARM. INST. L. W. GANS A.-G., W. KOLLATH, and H. MAGISTRIS (B.P. 311,436, 10.1.28. Addn. to B.P. 285,417; B., 1929, 110).—The dialysis solutions described in the prior patent are subjected to a rapid preliminary concentration, e.g., in compression evaporators, and are then evaporated rapidly to dryness at a low temperature, e.g., by the Krause or Hatmaker process, exposure to the air being reduced to a minimum during the processes. The dry products, the solutions, or the original material may be exposed to the action of ultra-violet rays. L. A. COLES.

Extraction of natural animal and vegetable material. H. SCHMIDT (G.P. 451,000, 17.11.25).-The material, e.g., gum benzoin, tolu balsam, storax, cloves, sandal wood, etc., is extracted with a light, readily volatile solvent which, in the same operation, is displaced by a high-boiling liquid, e.g., benzyl alcohol or its esters, ethyl phthalate, glyceryl triacetate, etc., which absorbs completely the constituents extracted by the volatile solvent. L. A. COLES.

Manufacture of pinacol. H. BERLIN, Assr. to INTERNAT. PATENTS DEVELOPMENT Co. (U.S.P. 1,703,220, 26.2.29. Appl., 20.12.26).-Acetone is agitated and boiled with aluminium (sheet or shavings) and carbon tetrachloride. Mercuric chloride is then added and, after 10-15 min., further carbon tetrachloride is gradually added as the reaction slows down. The product is diluted at 40-50° with water, the filtrate and washings are evaporated in presence of sodium carbonate, and the residue is crystallised from water. Anhydrous pinacol is obtained by treating the molten hydrate with 50% sodium hydroxide, or, alternatively, 50% sodium hydroxide may be added direct to the reaction mixture when addition of carbon tetrachloride 18 complete. R. BRIGHTMAN.

Manufacture of therapeutically active basic nitro-derivatives of 9[10]-aminoacridine. I. G. FARBENIND. A.-G. (B.P. 283,510, 11.1.28. Ger., 11.1.27).-Powerfully active bactericides are obtained by condensing a 10(ms)-chloro-3-nitroacridine with a diamine containing a primary and a tertiary amino-group, or by condensing with ammonia or a primary amine a 10-chloro-3nitroacridine containing already a strongly basic tertiary amino-group in a suitable substituent. An ether group may replace the 10-chloro-atom in the starting-material. The following are described: 3-nitro-10-β-diethylaminoethylamino-8-ethoxyacridine [dihydrochloride, m.p. 245-246° (decomp.)] from 10-chloro-3-nitro-8-ethoxy-acridine by way of the 10-phenoxy-compound and  $\beta$ -diethylaminoethylamine; 3-nitro-10-y-diethylaminoβ-hydroxypropylamino-8-ethoxyacridine, m.p.  $108^{\circ}$ [dihydrochloride, m.p. 226-227° (+ 5H20)]; 3-nitro- $10 - p - \beta$  - diethylaminoethylaminoanilino - 8 - ethoxyacridine, m.p. 120° (decomp.); the corresponding 10-p-ydiethylamino - B - hydroxypropylaminoanilino - compound, m.p. 131-132°, from γ-diethylaminoethyl - β - hydroxypropyl-p-phenylenediamine, b.p. 185°/2.5 mm., which is obtained from *p*-aminoacetanilide and epichlorohydrin with subsequent treatment with diethylamine and hydrolysis; the corresponding 10-p-y-( \beta'-diethylaminoethylamino) - B - hydroxypropylaminoanilino - compound, m.p. 86°, from  $\gamma$ -( $\beta'$ -diethylaminoethylamino)-  $\beta$  hydroxypropyl-p-phenylenediamine, b.p. 230°/3 mm.; 3: 7-dinitro - 10 - p -  $\beta$  - diethylaminoethoxyanilino - 8 - ethoxyacridine, m.p. 155°, by condensing 2-nitro-pphenetidine with 2-chloro-4-nitrobenzoic acid to a diphenylamine, m.p. 254-255°, which is cyclised to the acridone and converted into 10-chloro-3: 7-dinitro-8ethoxyacridine, m.p. 194-196°, and finally condensed with p-aminophenyl B-diethylaminoethyl ether, b.p. 175°/ 13 mm.; 3-nitro-10-amino-8-β-diethylaminoethoxyacridine, m.p. 237-238° [dihydrochloride, m.p. 265° (+3H20)], by the action of ammonia on the 10-chloro-compound, m.p. 159-160°, obtained by condensing 2-chloro-4-nitrobenzoic acid with p-aminophenyl B-diethylaminoethyl

ether to a *diphenylamine*, decomp. 226°, cyclising, and treating the *acridone* with phosphorus pentachloride. C. HOLLINS.

Organic compound of mercury and its manufacture. E. C. WHITE (U.S.P. 1,692,237, 20.11.28. Appl., 27.1.27).—Dibromosulphonfluorescein, from o-sulphobenzoic anhydride and resorcinol, dibrominated, is boiled with mercuric acetate, the precipitate is dissolved in just sufficient alkali, and the solution is evaporated to yield a disinfectant, germicidal mercury derivative suitable for local application or injection.

C. HOLLINS.

Manufacture of stable medicinally active salts of *p*-aminophenylstibinic acid. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 311,448, 11.2.28).—The acid is converted into a salt of a secondary amine, *e.g.*, diethylamine or piperazine. L. A. COLES.

Esters of cholesterol with unsaturated acids. W. MINNICH, ASST. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,690,647, 6.11.28. Appl., 10.11.24. Switz., 19.11.23). —See B.P. 243,510; B., 1926, 141.

[Manufacture of] 8-amino-6-alkoxyquinolines. W. Schulemann, F. Schönnöfer, and F. Mietzsch, Assrs. to Winthrop Chem. Co., Inc. (U.S.P. 1,703,365, 26.2.29. Appl., 30.1.26. Ger., 29.4.25).—See B.P. 275,277; B., 1927, 797.

# XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

See A., June, 660, Phototropic mercury compounds (RAO and WATSON). Formation of latent images (Toy and HARRISON).

#### PATENTS.

Manufacture of photographic silver salt emulsions. I. G. FARBENIND. A.-G. (B.P. 283,223, 7.1.28. Ger., 7.1.27) .- Sensitiveness of photographic silver salt emulsions is increased 30-50-fold by the addition of organic compounds, containing 1 or 2 sulphur atoms, which give silver derivatives gradually transformed into silver sulphide. Such are organic disulphides containing carboxyl groups (disulphidoacetic acid, aa-disulphidopropionic acid), certain thiol derivatives [thiolmalonic ("thiomalic") acid, potassium dithiocyanate, phenyl  $\beta\beta$ -dithiolvinyl ketone], and some cyclic sulphur compounds (cyclic disulphide of β-thiolthiocinnamic acid). In the example 0.015 g. of potassium dithiocyanate is added to an emulsion made from 1 kg. of silver nitrate. C. HOLLINS.

Colour photograph or film and its production. F. E. IVES, ASST. to C. W. FENNINGER (U.S.P. 1,695,284, 18.12.28. Appl., 20.10.26).—Undesirable effects of blue toning on the silver bromide in a colloid layer containing a coloured image produced in multicolour processes are corrected by treatment with a solution of 45 g. of sodium sulphate and 3 g. of potassium bromide in 2 litres of water. R. BRIGHTMAN.

[Recording and projection apparatus for] photography and kinematography in colours. Soc. CIVILE POUR L'ETUDE DE LA PHOTOGRAPHIE ET DE LA CINÉMATOGRAPHIE EN COULEURS (B.P. 293,047, 29.6.28. Fr., 30.6.27). 578

# XXII.—EXPLOSIVES; MATCHES.

Comparative tests of the initiating powers of lead azide and mercury fulminate detonators. B. CSERNECZKY (Z. ges. Schiess- u. Sprengstoffw., 1929, 24, 169-171).—The method employed was in principle that of Wöhler, but phlegmatised picric acid was substituted for phlegmatised T.N.T. as the colour of the smoke from the explosion could be used to indicate whether combustion was complete, being black when complete and yellow when incomplete. The arrangement of the apparatus was that of Hess. The results showed that a mixture of lead azide and lead styphnate is much more powerful than fulminate as a primary charge, and that tetryl is a better secondary charge than trotyl. A recess in the base of the detonator increases the initiating power of copper-tube detonators, but diminishes that of aluminium-tube detonators. Prolonged storage in a moist atmosphere diminishes neither the ease of ignition nor the initiating power of lead azide-lead styphnate detonators. S. BINNING.

See also A., June, 664, Fluorine and chlorine as explosive gas mixtures (FREDENHAGEN and KREFFT). 730, Purification of picric acid (BENEDICT).

# XXIII.—SANITATION; WATER PURIFICATION.

Germicidal properties of alkaline washing solutions, with special reference to the influence of hydroxyl-ion concentration, buffer index, and osmotic pressure. R. P. MYERS (J. Agric. Res., 1929, 38, 521-563).-The investigation was undertaken to determine some of the factors influencing the germicidal efficiency of various alkalis and basic salts, particularly in regard to the cleaning of milk bottles. Tests were made with several commercial washing powders and attention was paid to the relation between hydroxyl-ion concentration and germicidal efficiency. In general, the powders giving high  $p_{\rm H}$  values were more effective as germicides than those giving low  $p_{\rm H}$  values, but a powder containing chlorine was an exception to this. The effectiveness of the poorer powders was increased by the addition of sodium hydroxide. On dilution the efficiency of the powders decreased, but at various rates, and the decrease in efficiency seemed to be closely correlated with the decrease in hydroxyl-ion concentration. A combination of high hydroxyl-ion concentration and high temperature is effective in destroying spores of Bacillus cereus within a few minutes, whilst either factor alone is ineffective. A study was made of the influence of the hydroxyl-ion concentration, buffer index, and osmotic pressure of alkaline solutions on their germicidal action, using spores of a spore-forming aerobe to measure germicidal action. The death rate of spores in the alkaline solutions did not follow the logarithmic rate, but increased as disinfection proceeded. The death rate was increased by (1) an increase in the hydroxyl-ion concentration when the buffer index and osmotic pressure were kept constant, (2) an increase in the buffer index, keeping the hydroxyl-ion concentration and the osmotic pressure constant, (3) an increase in the osmotic pressure when the hydroxyl-ion concentration and buffer index were kept constant. Of these three factors, the osmotic pressure has the least

influence. Neutral sodium hypochlorite is a more effective germicide than alkaline sodium hypochlorite having the same amount of available chlorine. The alkaline hypochlorite solution with available chlorine present in 0.01% concentration was similar in germicidal action to 0.25N-sodium hydroxide. Measurements were conducted on the rate of hydrolysis of gelatin in strongly alkaline solution. The effect of osmotic pressure was not appreciable, but the higher the buffer index and the hydroxyl-ion concentration, the greater was the rate of hydrolysis. A titration method, which consists of determining the amount of alkali that is capable of maintaining a  $p_{\rm H}$  above 12.0, has been proposed as a means of measuring quickly the germicidal power of an alkaline washing solution. E. S. HEDGES.

Sterilisation of water and other liquids by the application of electrical potentials. G. LAKHOVSKY (Compt. rend., 1929, 188, 1069–1071).—Data are given for the bactericidal action produced by the application of electric potentials to silver electrodes immersed in liquid media in the case of *B. coli* and *B. typhosus*.

E. A. LUNT.

Chlorination of the water supply, particularly for tanneries. M. AUERBACH (Collegium, 1929, 104– 109).—Two methods are employed : (a) direct, in which chlorine gas is passed into the water supply, and (b) indirect, in which some water highly charged with chlorine is mixed with the water supply. The amount of chlorine necessary depends on the individual water, but is usually 0.1-0.3 g./m.<sup>3</sup> for a drinking water. Less than this suffices for a tannery water. There is no deleterious action on the pelt. Chlorinated water hinders putrefaction even if it contains no free chlorine. Chlorine is useful in deodorising and disinfecting tannery effluents. A large amount of chlorine is required to destroy anthrax spores. D. WOODROFFE.

See also A., June, 672, Continuous still for conductivity water (DE WITT and BROWN). 725,  $\alpha$ -Bromosoaps as germicides (Eggerth).

Phenol recovery from coal-tar effluents. HATCH. —See II. Cast-iron pipes. BRADSHAW. Mercurial poisoning. STELLING.—See X. Determination of organic matter in water. ANTIPOV-KARATAËV.— See XVI.

#### PATENTS.

Removal of scale or incrustations from boilers or preventing the formation of same. E. D. FELDMAN (B.P. 311,550, 7.5.28).—A composition is claimed consisting of an alkali soap of a fat or fatty oil which, after sulphonation, has been heated for 1 hr. at 150° and 3 atm. pressure, with or without the addition, while hot, of resin, resin soap, or ordinary soap containing fatty acids, so as to form an emulsion. The addition of 0.16 p.p.m. thereof to the contents of a boiler will remove pre-existent scale, and a little added to the feed water from time to time will then keep the boiler clean. C. JEPSON.

Brine evaporation (B.P. 309,104—6). Air for ventilation (B.P. 311,304).—See I. cycloHexyl compounds (U.S.P. 1,703,186).—See III.