

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

DECEMBER 9, 1927.

I.—GENERAL; PLANT; MACHINERY.

Producing small bubbles of gas in liquids by submerged orifices. C. G. MAIER (U.S. Bur. Mines, Bull. 260, 1927, 62—120).—Regulation of the dimensions of gas bubbles is necessary for the successful catalytic autoxidation of ferrous sulphate and sulphur dioxide. For a static bubble of low gas velocity ruptured only by buoyancy, in water at 20° with perfect wetting of the tip, $r = 9.05R^3$, where R is the radius of the free bubble and r that of the tip, both in cm. When there is a finite gas velocity R will also be modified by the effect of circulation and viscosity. The size of bubbles was determined in practice by counting, by the bubble burette (a tapered burette in which one bubble was caught), by photography, and by conductivity determinations. The last method depends on the fact that the resistance of a column of liquid up which a number of uniform bubbles are rising depends on the total gas volume and the average bubble size. For accurate results this method requires that the bubble size should not be too small relative to the column. By these methods the size of bubbles produced by various tips was determined. The results showed that the size was greatly influenced by gas pressure or velocity in the tip. Small bubbles produced by simple tips require very small orifices and in consequence uneconomically high pressures. The internal diameter of the tip only is of importance if wetting is perfect, so a perforated plate should give similar results to a single tip. Results with static bubbles were in good agreement with calculation. With increase in gas velocity the size passed through a minimum. The prime factor in selecting a material for the orifice is that it shall be perfectly wet. For bubbles below 1 mm. in diameter the pressure required to overcome surface tension is such that the power required would be excessive. Agitation devices for breaking up bubbles are not susceptible of accurate control. The size of bubbles can, however, be reduced by giving the liquid a motion at right angles to the bubble tip. In this way the grid type "shear bubbler" was designed. It consists of a grid of parallel pipes spaced closely with orifices near the plane of the axes of the pipes. The dimensions of such a bubbler for a given output are calculated, and also the power required for pumping liquid. Either a grid of lead or a fabric diaphragm (e.g., woollen filter cloth) is thought most likely to be suitable for the process under investigation.

C. IRWIN.

Automatic apparatus for the determination of moisture [by distillation with benzol etc.]. H. RÖSSLER (Chem.-Ztg., 1927, 51, 688—689).—A wide-

necked Erlenmeyer flask with ground-glass stopper is connected by the latter to a measuring cylinder holding benzol or the like and graduated in 50 c.c. A side-tube from a bulb above the stopper connects to the base of a reflux condenser. Below this is a tube with run-off tap graduated in 1/10 c.c. in which the distilled water is collected. The flask is intended particularly for the ready weighing of pasty liquids.

C. IRWIN.

Boiler-scale prevention. SAUER and FISCHLER.—See XXIII.

PATENTS.

Pulverisers. F. L. DUFFIELD (E.P. 277,734, 15.6.26).—A disintegrator is combined with a preliminary crusher; the latter has yielding parts to permit the passage of uncrushable material. The first set of beaters of the disintegrator serve also as fan, and an additional fan is placed after the beaters, but mounted on the same shaft, to draw material and air through the apparatus.

B. M. VENABLES.

Ball mills. E. C. R. MARKS. From TRAYLOR ENGINEERING & MANUF. CO. (E.P. 278,214, 6.12.26).—Transverse screens for tube or ball mills are constructed of a number of grate bars, all individually alike and easily renewable, which are held in a frame so that the resulting screen is conical with the base towards the inlet. Beyond the screen is a diaphragm plate with central hole, and between the diaphragm and screen are lifters to pass the undersize into the next grinding compartment or to the final discharge.

B. M. VENABLES.

Crushing and mixing mills of the edge-runner type. H. MARTINEZ and R. H. KIRK (E.P. 278,236, 8.2.27).—The edge-runners have non-radial axes and are provided with vertical adjustment either together or individually and with means for applying spring pressure.

B. M. VENABLES.

Edge-runner mills. R. B. LUCAS (E.P. 278,069, 28.6.26).—In an edge-runner mill a number of discs are grouped on a shaft which is offset to the vertical driving shaft, i.e., the runner shaft is not radial. The discs may themselves be made heavy or pressure may be applied by an additional weight or spring. A runner of normal type may or may not be used in addition.

B. M. VENABLES.

Disintegrating machine. D. C. ADDICKS (U.S.P. 1,643,938, 4.10.27. Appl., 3.4.26).—A crushing chamber with a bottom grating is mounted with a feed hopper in a housing, and a rotor in the chamber co-operates with a breaker plate. The material is fed into the chamber by a gyrating plate located between the breaker plate and a pendant hinged plate forming a movable bottom for the hopper. The gyrating plate is driven

from the rotor, and its lower end reciprocates on the breaker plate and slides over the movable bottom.

H. HOLMES.

Separation of solid materials from meal or powder. A. W. SIZER (E.P. 277,817, 9.10.26).—The material is delivered to a shaking screen or table provided with upstanding pins, preferably in staggered relation, and at its discharge end with a lateral trough, preferably pivoted, of which the discharge lip is adjustable in height. Materials, *e.g.*, string, wood, etc., not engaged by the pins are retained by the trough.

H. HOLMES.

Suction and filter apparatus for removing dust from grinding and like machines. F. H. and H. S. POCHIN (E.P. 278,156, 6.9.26).—A dust-collecting fan with individual inlet ducts from each source of dust and fabric-collecting filters.

B. M. VENABLES.

Mixing apparatus. A. B. and C. R. SMITH (E.P. 278,177, 5.10.26).—In addition to moving blades for throwing the material about, the mixer has fixed blades or inclined pressure surfaces which subject the material to a squeezing or wedging action.

B. M. VENABLES.

Concentrator table. A. H. STEBBINS (U.S.P. 1,644,753, 11.10.27. Appl., 10.11.24).—A deck mounted for reciprocation in a supporting frame is provided with a surface comprising unapertured sections with intervening sections having apertures through which air under pressure is supplied from below. The materials delivered to the deck are thus subjected alternately to the grading action of the deck alone, and to that of the air and moving deck combined. The concentrates on reaching an apertured section fall through against the lift of the issuing air. Lips at the apertures prevent the entry of the tailings.

H. HOLMES.

Method of drying material. J. E. ALEXANDER (U.S.P. 1,645,366, 11.10.27. Appl., 28.11.24).—Variable degrees of heat concentration are applied to the material in the drying chamber, while maintaining a uniform temperature of the superheated steam in the chamber surrounding the material.

H. HOLMES.

Dryer or dehydrating plant. F. F. KNIPSCHILD (U.S.P. 1,645,760, 18.10.27. Appl., 10.5.26).—A heat-circulating system within the drying enclosure is provided with an outlet and with guide means enabling it to draw in fresh air from the space immediately around the enclosure.

H. HOLMES.

Spray drying. INDUSTRIAL ASSOCIATES INC. (E.P. 278,263, 4.4.27. Addn. to E.P. 272,859; B., 1927, 801).—In the apparatus described in the original patent several nebulisers or atomisers are used all at about the same level. Since each central spray is surrounded by its own downwardly-flowing stream of hot air there is no danger of mist from adjoining sprayers coalescing into drops.

B. M. VENABLES.

Apparatus for atomising liquids. S. WRIGHT (E.P. 277,912, 9.6.27).—Liquid is lifted by helical vanes on the interior of an inverted cone, which is in rapid rotation. The liquid passes through openings in the cone over the surface of discs attached to its exterior. A number of spraying points are formed on the edges of the discs by notching and twisting them, and the

discs are surrounded by notched strips preferably arranged helically.

B. M. VENABLES.

Separation of solids from liquids. S. SYMINGTON (U.S.P. 1,641,708, 6.9.27. Appl., 26.6.20).—The mixture of solid and liquid is delivered on to an endless fabric belt which is mechanically folded to enclose the mixture and passed over a suction box to remove the liquid. The belt is next led through a stream of washing liquid, which is removed by a second suction box, and after passage through a dryer the belt is mechanically unfolded and the solid removed from it.

T. S. WHEELER.

Evaporators. H. HILLIER (E.P. [A] 278,075 and [B] 278,299, 29.6.26).—(A) The evaporator comprises a shell containing the liquid to be evaporated and a space for vapour. Projecting from the side of the main shell is another shell for heating medium, within which is a bank of horizontal or inclined tubes opening at one end to the liquor space in the main shell and at the other to a header within the projecting shell. To allow for expansion the header is not attached to the small shell, but only to the tubes. The tubes in the lower row or rows are larger in diameter and serve for the inward flow of the circulation. (B) The upper tubes which serve for the outward flow of liquid plus vapour increase in length downwards so as to obtain approximately equal flow through each; to effect this the tube plate within the main shell is stepped or inclined. Guide plates are placed within the main shell to deflect the issuing liquor and vapour towards the surface of the liquor.

B. M. VENABLES.

Crystallisation of liquids. G. T. WALKER (U.S.P. 1,644,161, 4.10.27. Appl., 24.3.24. Cf. U.S.P. 1,615,151; B., 1927, 176).—The hot solution is progressively cooled as it passes through a container where its level is maintained constant with overflow discharge. Crystallisation is assisted by devices which lift the crystals and drop them through the solution. The crystals are advanced through the container and finally lifted for discharge with the solution.

H. HOLMES.

Fluid filters. SVENSKA ACKUMULATOR AKTIEBOLAGET JUNGNER, Assees. of B. H. LUNDBORG (E.P. 272,841, 9.11.26. Swed., 19.6.26).—The filter is of the type where the filtrate passes between the spaces left between an inner cylinder and a surrounding sleeve, each being formed with grooves, the valleys on one being opposite the hills on the other. The cylinder and sleeve are tapered so that the size of the interstices may be adjusted by relative longitudinal movement.

B. M. VENABLES.

Apparatus for filtering. W. H. FURNESS, Assr. to CELLOCILK Co. (U.S.P. 1,643,299, 27.9.27. Appl., 6.12.24).—The filter is composed of a casing made up of a lower and an upper casting, the former being provided with an inlet and the latter with an outlet tube. The filtering element, which is secured to the upper casting, consists of a number of plates the lower surfaces of which are entirely flat, but with upper surfaces consisting of a flat central portion surrounded by an outer portion which tapers towards the lower surface from a point slightly below the level of the flat central portion. The liquid is enabled to pass through the plates by means of holes adjacent to the central flat portion, communication being established between these holes

by means of a channel or groove, whilst the solid material is held back at various points in the taper between the plates according to the size of the particles.

F. R. ENNOS.

Decolorisation of aqueous liquids. BRIT. DYE-STUFFS CORP., LTD., C. HOLLINS, and E. CHAPMAN (E.P. 278,485, 24.8.26).—Certain wetting-out agents, namely alkylnaphthalenesulphonic acids, ligninsulphonic acid, naphthenic acids, sulphonated isopropylated mineral oil fractions, and sulphonated higher fatty acids, facilitate contact between the adsorbent substance (charcoal, silica, etc.) and the liquid to be decolorised, and thus accelerate decolorisation. The substances are added either to the liquid or to the adsorbent material.

C. HOLLINS.

Washing apparatus involving the intermixing of gases and liquid. E. L. PEASE (E.P. 278,105,7.7.26).

—Liquid is circulated between upper and lower chambers by a chain pump. In the rising pipe the discs fit closely and lift the liquid, which descends in the other pipe, where there is considerable clearance, and through which the gas passes con- or counter-current to the falling liquid. A number of units may be used in series for counter-current washing.

B. M. VENABLES.

Apparatus for cooling and purifying gases.

RICHARDSONS, WESTGARTH, & CO., LTD., and F. G. INGLIS (E.P. 278,118, 21.7.26).—On one or both sides of a fan are placed alternate fixed and rotating perforated discs, the latter being on the fan shaft. Jets of liquid are admitted to the spaces between the discs through holes in the boss driving the rotating discs. The gas passes axially through the discs, and is exhausted by the fan.

B. M. VENABLES.

Filtration of gases with a high oxygen content.

I. G. FARBERIND. A.-G., Assees. of A. BENISCHEK (G.P. 442,504, 22.9.25).—Liquid triaryl phosphates are used for removing dust from the gases.

L. A. COLES.

Method and apparatus for [heat-]treating materials. DWIGHT & LLOYD METALLURGICAL CO.,

Assees. of R. W. HYDE (E.P. 269,480, 26.11.26. U.S., 16.4.26).—A number of grates or perforated pallets are attached to a chain conveyor which travels beneath a low roof of refractory material. Flame is produced in combustion chambers below the roof alongside the conveyor, and the hot gases are drawn down through the material (e.g., alkaline-earth carbonates, clay products, etc.) on the conveyor to wind (vacuum) boxes below. At the feed end provision is made for preheating the material by passing the gases through twice, and at the outlet end for continuing the reaction and cooling by passing the material under a continuation of the roof which is not provided with combustion chambers or wind box.

B. M. VENABLES.

Muffle furnace. W. G. BRIDGE and W. A. PERVIER, Assrs. to C. E. PARKHURST (U.S.P. 1,644,107, 4.10.27. Appl., 17.4.26).—A refractory lining within a layer of heat-insulating material is provided with means for heating it electrically. The lining comprises a series of plates, the series being removable from the muffle, and each plate readily separable from the series.

H. HOLMES.

Annular rotary-hearth ovens. J. E. POLLAK. From TROCKNUNGS-, VERSCHWELUNGS-, U. VERGASUNGS-GES.M.B.H. (E.P. 278,207, 23.11.26).—An annular hearth is constructed in three stories, connected together by hinge-like members to allow for distortion. The lower story comprises the carriage and driving mechanism below the heating zone, the middle comprises a non-conducting bed of the furnace and gas-tight joint, and the upper is a conducting hearth or platform which supports the goods and is arranged at a considerable height above the bed, so that the platform may be heated from below by radiation from the lower part of the furnace walls. Fixed walls and roof are provided enclosing the two upper hearths.

B. M. VENABLES.

Furnace walls. SIMON-CARVES, LTD., and B. F.

CLARK (E.P. 278,159, 9.9.26).—A furnace wall is provided with heat-insulation containing at least one air space, a mattress of solid heat-insulation is held against the inner wall (e.g., the water wall of a boiler furnace) by means of round iron rods laid longitudinally in V-shaped spaces formed between the mattress and inclined lugs attached to the outer wall across the air space. Under expansion and contraction strains the iron rods can follow up or rise.

B. M. VENABLES.

Heat-exchange apparatus. J. MUCHKA (E.P. 272,152, 16.7.26. Austr., 7.6.26).—A heat exchanger is constructed of flat rectangular plates joined together by U-shaped members at opposite edges, alternately the vertical and horizontal edges. Distance pieces in the form of bars are also inserted.

B. M. VENABLES.

Heat-exchange apparatus. B. F. STURTEVANT CO., Assees. of G. C. DERRY (E.P. 254,702 and 269,560, 23.6.26. U.S., 6.7.25).—A heat exchanger, e.g., for use as an economiser in a boiler flue, is constructed of a number of horizontal tubes, of which the heating surface is increased by flange-like fins, and in which the water zig-zags across and downwards with an upward flow of the flue gases. Only a few tubes are connected to any one header, and they are supported to allow for free expansion and contraction, and are easily cleaned or removed.

B. M. VENABLES.

Sublimation apparatus. C. FIELD, Assr. to NAT. ANILINE & CHEMICAL CO., INC. (U.S.P. 1,644,518, 4.10.27. Appl., 11.2.21).—Pulverulent material which becomes plastic at the sublimation temperature is subjected to continuous sublimation by the use of a receptacle provided with a heating jacket and with stirrers operating adjacent to the heated walls. A spraying device converts the highly heated residue into granules adapted for ready removal through a discharge opening.

H. HOLMES.

Process of refrigeration. W. H. CARRIER, Assr. to CARRIER ENGINEERING CORP. (U.S.P. 1,642,942—3, 20.9.27. Appl., [A] 23.4.23; [B] 22.3.24).—(A) *s*-Dichloroethylene is suitable as a working fluid for refrigerating machines. (B) It is preferable to separate it by fractional distillation into its stereoisomerides, and to use one or other separately.

T. S. WHEELER.

Fire-extinguishing compound. "POLEO" FEUER-LÖSCH APPARATE G.M.B.H. (E.P. 273,713, 27.6.27. Ger., 30.6.26).—The compound consists of 60% of methyl bromide, 35% of carbon tetrachloride, and 5% of ethylene bromide.

H. ROYAL-DAWSON.

Friction surfaces etc. BRIT. DYESTUFFS CORP., LTD., C. J. T. CRONSHAW, J. BADDILEY, and E. CHAPMAN (E.P. 278,465, 21.7. and 3.11.26).—The slipping effect due to water on a braking surface is greatly diminished and the braking power may be increased by the presence at the braking surface of emulsifying, dispersing, or wetting-out agents, *e.g.*, isopropyl-naphthalenesulphonic acids, sulphonated aldehyde-phenol condensation products, ligninsulphonic acid, sulphonated acid resins, etc., and in particular the sulphonated isopropylated petroleum fractions of E.P. 274,611 (B., 1927, 841). These substances similarly prevent the slipping of driving-belts on pulleys. They are applied ordinarily as aqueous solutions of their sodium salts. C. HOLLINS.

Continuously-operating apparatus for gas analysis. O. RODHE, Assr. to SVENSKA AKTIEBOLAGET MONO (U.S.P. 1,644,951, 11.10.27. Appl., 15.12.22. Swed., 15.12.21).—The gas mixture is passed through a first group of cells in which the percentage of carbon dioxide is determined, thence through means for oxidising carbon monoxide and methane to carbon dioxide, and finally through a second group of cells in which the percentage of carbon dioxide in the resultant mixture is determined. H. HOLMES.

Instrument for measuring fluids for turbidity, colour, and other characteristics. W. G. EXTON (U.S.P. 1,644,331, 4.10.27. Appl., 23.10.25).—The fluid is contained in a transparent receptacle supported within a casing provided at one end with an observation opening and open at the other end to admit light. A target member is mounted in this open end, and a measuring wedge is slidable transversely through the casing between the eye-piece and the sample under test. H. HOLMES.

Apparatus for determining the specific gravity [of gases]. G. B. LINDERMAN, JUN., Assr. to AMER. METER Co. (U.S.P. 1,644,684, 11.10.27. Appl., 27.5.24).—Two pressure-developing centrifugal devices, operated at a predetermined speed ratio, are supplied the one with air and the other with the gas to be tested, and means are provided for maintaining equilibrium between the pressures of the air and gas supplies. The outlet from each device is connected to one limb of a corresponding manometer, the other limb of which is subjected to the pressure of the air and gas supplies and is provided with a float. The floats operate means for indicating the relative sp. gr. of the gas and air. H. HOLMES.

Separation of two or more substances from a solution. I. ISAACHSEN, Assr. to A./S. KRYSTAL (U.S.P. 1,646,454, 25.10.27. Appl., 26.2.23. Norw., 29.3.22).—See G.P. 392,193; B., 1924, 584.

Continuous determination of a constituent of a mixture of gases. R. P. MASE (E.P. 271,027, 8.11.26. U.S., 15.5.26).—See U.S.P. 1,634,331; B., 1927, 592.

Refrigerating plant. I. AMUNDSEN (U.S.P. 1,647,208, 1.11.27. Appl., 29.10.26. Norw., 28.1.26).—See E.P. 266,683; B., 1927, 592.

[Blocks for] furnaces having suspended arches. L. MORTON and J. HARGROVE (E.P. 277,768, 7.7.26).

[Slabs for] heat insulation. R. W. ANDERSON (E.P. 277,747, 22.6.26).

Separation of solids from liquids (E.P. 277,500).—See IX.

II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Chemical constituents of lignites. J. MARCUSSEN (Z. angew. Chem., 1927, 40, 1104—1106).—Lignites contain, on an average, about 69% C, 5.5% H, 25% O, and 0.8% N (reckoned on the ash-free, dry substance). The constituents, other than water and mineral matter, are waxes, resins, free humic acids, humic acid anhydrides, humic ketones, water-soluble carbonyl acids, acetic acid, lignin, and cellulose. Waxes and resins are extracted with carbon tetrachloride, and separated with a mixture of alcohol and ether at a reduced temperature, the waxes being insoluble. Free humic acids are removed with ammonia, and humic acid anhydrides are hydrated with hot 1% sodium hydroxide. Lignin and ketones are separated by mixing with molten resorcinol, the ketones remaining unchanged. Cellulose is determined by chlorination. Lignites derived from tree trunks are principally composed of humic acid anhydrides, lignin, cellulose, and water-soluble carbonyl acids. In earthy lignites, lignin and cellulose have been almost completely converted into humic acids and anhydrides. The content of humic ketones increases with the age of the lignite. Alkali-soluble lignites contain a high proportion of free humic acids, and their cellulose is in the form of oxycellulose, so that they are almost completely soluble in cold 1% sodium carbonate. Water-soluble carbonyl acids present in lignites have reducing properties and comprise a mixture of glycuronic acid and an acid derived from it by oxidation. W. T. K. BRAUNHOLTZ.

Mechanical strength of coke. G. DÖRFLINGER (Stahl u. Eisen, 1927, 47, 1867—1871).—The strength is determined by placing 50 kg. of the coke in pieces of about 50 mm. in diameter in a rotating cylinder which is rotated through 100 revolutions in 4 min. The coke is then screened on a series of screens with holes from 40 mm. to 10 mm. At least 72% should remain on the 40 mm. screen and only a very small quantity of material finer than 10 mm. should be obtained.

A. R. POWELL.

Chemical methods of treating wood: use of waste material and wood preservation. C. SCHWALBE (Z. angew. Chem., 1927, 40, 1172—1176).—A general survey of the application of the products of German forestry. The life of the wood-distillation industry may be extended if the slowness and low thermal efficiency of the usual distillation at 500° can be avoided. The temperature required may be lowered by the use of high pressures, or by the use of a solution of magnesium or calcium chloride with strong sulphuric acid. In the latter case a temperature of 180° and a pressure of 5.6 atm. is sufficient. The hydrochloric acid liberated hydrolyses the cellulose to sugar which is carbonised by the sulphuric acid. The yield of methyl alcohol and acetic acid is greatly increased, no tar being produced. The charcoal is of lower calorific value than ordinary wood charcoal, but may be given a second dry distillation if desired. Both this process and the proposed hydrolysis to sugar have the disadvantage that they usually involve large central plants with carriage charges to them for sawdust etc. that could scarcely be borne. The bulk of waste product discharged from sulphite-pulp mills is very large. Its only likely large-scale application is as

fuel. It can be carbonised at high pressures, but this is too costly. Addition of adsorption carbon, which can be cheaply made by the low-temperature carbonisation of shavings (*vide supra*), to the waste renders it possible to carbonise it completely by heating at 10 atm. A yield of 60–70% of charcoal is obtained, with wood alcohol and acetic acid as by-products. This process also involves certain practical difficulties. Neither does the manufacture of hemicellulose offer an outlet for waste wood products. A chemical method of removing bark in sulphite-pulp manufacture would give an appreciable saving in material. If undried wood could be impregnated, *e.g.*, with sodium fluoride, as some experiments suggest, it might be possible to sterilise and market trees felled in consequence of disease.

C. IRWIN.

Distillation of benzol wash-oil under diminished pressure. F. RASCHIG (*Z. angew. Chem.*, 1927, 40, 1089–1090).—Ordinary benzolised oil is distilled under diminished pressure at about 140°, without the use of free steam, in a still 10 m. high. The vapours pass through a condenser in which they give up their heat to the in-flowing rich oil, so that the latter enters the still at 120–130°. The quantities of steam and cooling water required are, respectively, three times and forty times the quantity of benzol made, as against six times and a hundred times in the usual process. Thickening of the wash oil is much less pronounced, and the benzol vapours are free from water and can be fractionally condensed. To avoid any benzol losses due to incomplete condensation under diminished pressure, the lightest fraction is taken off and condensed under only moderately reduced pressure, the heavier fractions being subsequently distilled under a higher vacuum.

W. T. K. BRAUNHOLTZ.

Esthonian oil shale and its economic importance. I, II. P. W. UHLMANN (*Chem.-Ztg.*, 1927, 51, 745–746, 766–767).—I. The Esthonian oil shale deposit covers an area of about 3000 km.² on the Russian–Esthonian border, and the amount of material available is estimated at 7.2 milliard tons. Analysis of various samples showed the content of organic matter to vary from 35.7 to 51.7%. The air-dried shale consisted of volatile matter 42.7–60.1%, coke 2.9–9.7%, and ash 36–53%. The organic matter contained 71.5–72.4% C, 8.4–8.7% H, 18.7–20.1% O, and 0.2–0.3% N, but no sulphur. Any sulphur present in the shale itself (0.4–1.5%) must therefore be in combination with the inorganic material. Various industrial applications of the shale are indicated, amongst which are the utilisation of its distillation products in the manufacture of asphalt, and of the inorganic material present in it and containing silica, alumina, and lime, for use in the cement industry.

II. The shale is distilled in shaft-like retorts and heated by a current of oxygen-free gas from a gas producer. The gas, produced at 600–1000°, is combined with the cooled gas from the still to reduce the temperature to about 500°, at which temperature the distillation takes place. Six such stills have a throughput of 200 tons of shale per day, producing 30–40 tons of oil. One ton of shale produces about 280 cub. m. of gas, having a calorific value of 4300–4500 kg.-cal./cub. m. and the following composition: 37.6% H₂, 25.8% CH₄, 1.6% C₂H₄, 19.1% CO, 13.3% CO₂, and 2.6% N₂. Tar (3.5%) and

a large proportion of aromatic hydrocarbons as well as phenols are produced. The amount of shale oil produced (16–25%) depends on the method of preparation. From the washing oil in the gas washer is recovered a benzine (*d* 0.76) with a volatility of 29% at 100°. A second fraction, resembling petroleum, has *d* 0.813–0.850, flash point 36°, and contains 0.65–0.77% S. A third fraction also occurs having *d* 0.900, flash point 55–60°, and containing 0.62% S. Phenols occur in the fraction 180–360° in proportions ranging from 20 to 50%, depending on the temperature of distillation of the shale. The use of superheated steam also raises the proportion of phenols. The residue remaining after the distillation of the crude oil above 360° contained pitch and asphalt. Crude shale oil consists of neutral substances (chiefly hydrocarbons) 72.1%, higher phenols 22.4%, carboxylic acids 4%, and basic substances 0.2%. It has *d* 1.004, flash point (P.-M.) 84°, unsaturated compounds 81%, and viscosity at 50° (Engler) 5.38 (cf. Kogerman, B., 1927, 865).

R. C. ODAMS.

Influence of admixture of salts and certain organic compounds on the oxidation of highly refined [transformer] oils. H. VON DER HEYDEN and K. TYPKE (*Petroleum*, 1927, 33, 1253–1254).—150 g. of the oil were mixed with 0.5 g. of each of the various compounds in turn and heated for 7 hrs. at 120° in a flask through which a current of oxygen was passed. After oxidation the acid value, sulphuric acid-soluble material, and tar and saponification values were determined. The solubility of the oil in sulphuric acid was found to be a good indication of the change produced, as in a non-sludging oil this factor did not increase after oxidation. Of the substances used, magnesium oxide and carbonate gave somewhat low tar and acid values. The addition of caustic soda or of sodium or potassium carbonate accelerated the oxidation; ferric oxide had an extraordinarily unfavourable influence. Again, lead monoxide checked the oxidation of the oil, whilst red lead oxide (Pb₃O₄) gave an acid value about the same as that obtained by oxidation of the oil alone, the saponification value, however, being increased considerably, and lead dioxide greatly increased the oxidation. Of organic compounds, phenol, starch, oxalic and oleic acids accelerated oxidation, whilst salicylic acid, hydroxylamine hydrochloride, and especially pyrogallol and aniline had the opposite effect. These results are in contradiction to those of Butkov (B., 1926, 1002), who found that both phenol and aniline were without action on the oil.

R. C. ODAMS.

Shale oil as wood preservative. SOWDER.—See IX.

PATENTS.

Treatment of oil and coal and production of liquid hydrocarbons therefrom. F. LAMPLOUGH and A. E. HODGSON (E.P. 277,419, 15.6. and 11.8.26).—From 10 to 50% by wt. of finely-divided coal is mixed with a heavy mineral oil in such a manner that a stable dispersion of coal in oil is formed. The suspension so formed is subjected to high temperature (500–600°) and pressure (100–600 lb./in.²), and the liquid fractionally distilled. The oil is apparently hydrogenated by the coal, and the process may be furthered to such an extent that the coal, with the exception of the ash, goes almost

completely into the liquid state. The heaviest distilled fractions may be used as a binder in the manufacture of briquettes. Fractional condensation of the oil vapours may be effected in apparatus as described in E.P. 216,922 (B., 1924, 665). The distillation vapours may be hydrogenated and redistilled for spirit. R. C. ODAMS.

Motor fuels. E. V. BERESLAVSKY (E.P. 258,550, 30.9.26. U.S., 17.9.25).—Low-compression fuels, such as gasoline or kerosene, are adapted for use in high-compression engines without "knocking" by the addition of mesitylene, its amino-compounds, or their derivatives. The mesitylene may be dissolved in alcohol or some other blending agent and so admixed with the fuel, or it may be atomised in the engine intake, or fed into the combustion chamber before ignition. R. C. ODAMS.

Utilisation of sludge fuel. GES. FÜR MASCHINELLE DRUCKENTWÄSSERUNG, M.B.H. (G.P. 441,669, 5.5.25).—The fuel is briquetted without binding medium, the briquetting plant being placed near the furnace and the briquettes being delivered directly to the furnace chamber so as to avoid crumbling. C. HOLLINS.

Prevention of [coal] mine explosions. W. E. TRENT, Assr. to TRENT PROCESS CORP. (U.S.P. 1,642,912, 20.9.27. Appl., 20.10.24; cf. U.S.P. 1,479,757; B., 1924, 208).—The walls of the passages of the mine are sprayed with a high-boiling oil with a high flash-point. T. S. WHEELER.

Manufacture of oil gas. R. D. PIKE (U.S.P. 1,644,146, 4.10.27. Appl., 2.5.23. Renewed 28.2.27).—Oil previously treated with steam is passed over heated surfaces in a closed chamber, whereby the oil is converted into a lean gas carrying particles of suspended carbon. The gas is treated in another closed chamber with steam at a predetermined temperature, and the carbon is thus converted into gas. The lean gas is subjected to further treatment and thereby enriched. R. C. ODAMS.

Process and apparatus for cracking hydrocarbons. H. J. JANSEN, JUN. (E.P. 278,235, 7.2.27).—Hydrocarbons are cracked by forcing them in a horizontal direction through a vessel containing a metal (lead) and/or metallic salt (zinc chloride) in a molten condition. The vapour outlet has only a small free surface, so that carbon deposit is removed in suspension by the vapours, which travel at a high velocity. Heavy liquid residue is immediately run off the surface of the molten material. R. C. ODAMS.

Apparatus for cracking oils. O. KAY (E.P. 276,947, 8.1.27. U.S., 4.9.26).—Oil is introduced into an atomiser consisting of a frusto-conical nozzle rotating in a housing open at one end so that the oil impinges radially upon the inner walls of the nozzle. Air is introduced axially into the cone, and cracking takes place by the bombardment of the inner walls of the cone by the hydrocarbon molecules. The various products are separated by causing portions of the spray of varying densities to fall into vessels suitably placed. R. C. ODAMS.

Treatment [cracking] of hydrocarbons. R. DE M. TAVEAU, Assr. to TEXAS CO. (U.S.P. 1,645,553, 18.10.27. Appl., 16.3.23).—The cracking process consists in vaporising the oil in a still so constructed

that it contains a quiescent pool of aluminium chloride which is not in contact with the heated bottom of the still. R. C. ODAMS.

Treatment [cracking] of hydrocarbon oils. R. C. HOLMES, F. T. MANLEY, and O. BEHMER, Assrs. to TEXAS CO. (U.S.P. 1,646,380, 18.10.27. Appl., 10.12.23).—Oil is heated to cracking temperature and passed into a number of converters connected in series and maintained at that temperature. A portion of the generated vapours is condensed and refluxed back to the several converters. R. C. ODAMS.

Continuous distillation of hydrocarbon oils. J. R. CARRINGER, Assr. to STANDARD DEVELOPMENT CO. (U.S.P. 1,644,324, 4.10.27. Appl., 6.5.21).—In a battery of continuous stills, each having a fractional condenser, a common pressure-equalising chamber is fitted, together with means for connecting the vapour spaces of the condensers to the equalising chamber. R. C. ODAMS.

Decomposition of gaseous or vaporous hydrocarbons by means of water vapour. I. G. FARBER-IND. A.-G. (E.P. 265,989, 11.2.27. Ger., 11.2.26).—A mixture of hydrocarbons and water vapour is decomposed by the action of heat into hydrogen and carbon monoxide. The highly heated gases obtained from the decomposition are cooled by contact with cold water. The water thus heated is brought into direct contact with fresh gases containing hydrocarbons to be subjected to the reaction, thus heating the gases and charging them with water vapour. The resulting gas mixture is then introduced into the decomposition chamber, where carbon monoxide and hydrogen are produced. If pure hydrogen is desired, the gas issuing from the decomposition chamber is passed, with further additions of steam or water, over a suitable catalyst, preferably at a lower temperature, and the carbon monoxide and carbon dioxide are eliminated. R. C. ODAMS.

Preparation of alcohols from olefine-bearing gases. PETROLEUM CHEMICAL CORP., Asses. of H. S. DAVIS (E.P. 249,834, 1.3.26. U.S., 30.3.25).—The mixed gases containing olefines, especially gases from cracked oils, are freed from hydrogen sulphide by means of iron oxide and scrubbed with 80% sulphuric acid to remove water and olefines more reactive than propylene and ethylene. The propylene is then absorbed in 100% sulphuric acid at 1–2 atm. pressure in towers cooled below 30°. The product is diluted with water and the constant-boiling mixture of isopropyl alcohol and water is distilled off. A preferred method uses six towers, two for 80% sulphuric acid and four for 100% sulphuric acid; the towers are 24 ft. high and 6 in. diameter, the gas flow being 10 cub. ft. per min., and the 100% acid flow 2–3 gals. per min. Dilution of the acid by the absorption products may be compensated by addition of oleum. The use of 100% acid enables 1 mol. of acid to absorb as much as 2 mols. of propylene, and these concentrated absorption products are much more stable than those obtained with 90% acid. C. HOLLINS.

Manufacture of a clarifying and decolorising agent [for oils]. M. L. CHAPPELL, R. F. DAVIS, and M. L. MOORE, Assrs. to CONTACT FILTRATION CO. (U.S.P. 1,642,871, 20.9.27. Appl., 1.8.21).—Bentonite clay mixed with water is heated with sulphuric acid at 100°

for 14 hrs., the mixture being thoroughly agitated by air. The solid residue is washed with water and dried.

T. S. WHEELER.

Separation of oily emulsions. G. W. COGGESHALL and A. REILLY, Assrs. to JEFFERSON CONSTRUCTION & OIL TREATING Co. (U.S.P. 1,643,698—9, 27.9.27. Appl., 10.11.24. Renewed 1.3.27).—(A) Petroleum emulsion is treated with the sulphonic acid mixture obtained in the purification of oil (0.2%), and with an alkali or alkaline-earth chloride (1.5%) in solution. (B) The sulphonic acid mixture is replaced by a soap solution.

T. S. WHEELER.

Breaking of petroleum emulsions. M. DE GROOTE and W. C. ADAMS, Assr. to W. S. BARNICKEL & Co. (U.S.P. 1,641,804, 6.9.27. Appl., 11.8.26).—The ammonium salt of the product obtained by heating a Twitchell reagent with oleic or hydroxystearic acid (1 pt.) is employed.

T. S. WHEELER.

Treatment of organic liquids [hydrocarbons] with alternating electric discharge. SIEMENS & HALSKE A.-G. (F.P. 615,581, 4.5.26).—The hydrogen generated by the discharge through liquid hydrocarbons is used for boiling the liquid, thus causing frothing. L. A. COLES.

Briquetting of fuel. P. E. WELTON and G. H. WADSWORTH, Assrs. to P. E. WELTON ENGINEERING Co. (U.S.P. 1,642,055, 13.9.27. Appl., 25.11.22).—See E.P. 244,624; B., 1926, 181.

Treatment of oil. G. C. KELLEY (Re-issue 16,778, 25.10.27, of U.S.P. 1,465,398, 21.8.23).—See B., 1923, 1012 A.

Manufacture of bituminous products etc. D. B. W. ALEXANDER (E.P. 270,649, 7.3.27. U.S., 4.5.26).—See U.S.P. 1,603,502; B., 1927, 39.

Annular rotary-hearth ovens (E.P. 278,207).—See I.

Hydrogenation process (G.P. 441,164).—See XX.

III.—TAR AND TAR PRODUCTS.

Analysis of phenol (carbolic acid). S. DEL MUNDO (Philippine J. Sci., 1927, 33, 363—373).—A comprehensive and somewhat polemical discussion of the numerous modifications of Koppeschaar's original bromination method of analysing aqueous solutions of nearly pure phenol. Syrupy phosphoric acid is suggested as a better reagent than concentrated hydrochloric acid to liberate bromine from potassium bromide-bromate in countries such as the Philippines where normal laboratory temperature is 30°. The gravimetric method is inaccurate owing to uncertainty as to whether tribromophenol or tribromophenol bromide is precipitated.

E. HOLMES.

PATENT.

Extraction of neutral oils from tar, tar-oil, or pitch. H. WITTEK (E.P. 256,923, 15.6.26. Ger., 12.8.25).—The acid constituents of crude coal tar are separated by dissolving the latter in an organic solvent (e.g., alcohol, acetone) not soluble in paraffin oil, and agitating the solution with an oil composed mainly of hydrocarbons of the paraffin series. The neutral oils may be separated from the paraffin extract by subsequent distillation. The process may be carried out in the vapour phase or under pressure.

R. C. ODAMS.

IV.—DYESTUFFS AND INTERMEDIATES.

Coal-tar colours in food. NICHOLLS.—See XIX.

PATENTS.

Manufacture of vat dyes of the anthraquinone series. I. G. FARBENIND. A.-G., Assees. of F. KADEF (G.P. 439,614, 29.3.25. Addn. to G.P. 436,535; B., 1927, 387).—The processes of the prior patent are extended to anthraquinoneoxazoles. 2-Phenylanthraquinone- α -oxazole [6 : 7-phthaloyl-2-phenylbenzoxazole], obtained by heating 1-chloro-2-benzamidoanthraquinone with sodium carbonate in nitrobenzene, gives by nitration and reduction a red vat dye; the β -oxazole, from 2-bromo-1-benzamidoanthraquinone, yields a similar dye. Nitration and reduction also convert into vat dyes the oxazoles from 1 : 5-dichloro-2 : 6-dibenzamidoanthraquinone (brown), from 1-chloro-2-aminoanthraquinone (2 mols.) and teraphthalyl chloride (1 mol.; brown-red), and from 2-aldehydoanthraquinone and *o*-aminophenol (brown-red). Yellow vat dyes with good fastness properties are obtained by acylation (benzoylation) of the amino-oxazoles.

C. HOLLINS.

Manufacture of new vat dyes and intermediates for vat dyes. BRIT. DYESTUFFS CORP., LTD., A. SHEPHERDSON, and A. J. HAILWOOD (E.P. 278,102, 7.7.26).—Pyranthrone is oxidised with manganese dioxide in sulphuric acid. The product may be alkylated directly, or after reduction with sodium hydrogen sulphite, with or without removal of unchanged pyranthrone by extraction with nitrobenzene. Methylation gives a brown vat dye, fast to acids, alkalis, and chlorine.

C. HOLLINS.

Dyes and dyeing [soluble esters of leuco-vat dyes]. MORTON SUNDOWN FABRICS, LTD., J. MORTON, B. WYLAM, J. E. G. HARRIS, and J. I. MORGAN (E.P. 278,399, 3.5.26. Addn. to E.P. 251,491; B., 1926, 625).—The processes of the prior patent are performed in presence of a diluent, such as acetone or nitrobenzene.

C. HOLLINS.

Manufacture of halogenation products of perylene-tetracarboxylic di-imide and its derivatives. KALLE & Co. A.-G., Assees. of M. P. SCHMIDT and W. NEUGEBAUER (G.P. 441,587, 27.10.22).—The di-imide and its derivatives (excluding those of G.P. 276,956) are halogenated in chlorosulphonic acid. The tetrachloro-di-imide, obtained by the action of chlorine in chlorosulphonic acid in presence of iodine, dyes cotton from the vat a yellow-red shade, becoming lilac on soaping.

C. HOLLINS.

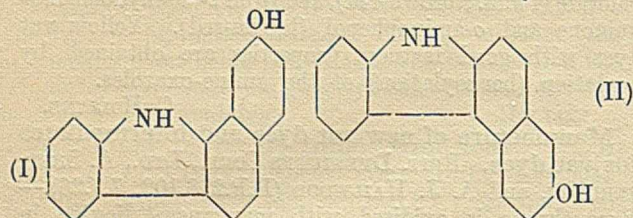
Preparation of ice-colours. E. GEBAUER-FÜLNEGG and W. SPECHT (Austr. P. 105,341, 14.11.24).—Diazo or tetrazo compounds are coupled with β -naphthols containing in the 1-position halogen, nitro-, alkyl, aryl, or acid groups. The substituents are not expelled, coupling occurring in the hydroxyl group. *p*-Nitrodiazobenzene is thus coupled with 1-chloro- or 1-bromo- β -naphthol (brown), 1-bromo-2-hydroxy-3-naphthoic acid (brown-orange), 1 : 3 : 6-tribromo- β -naphthol (orange), 1-nitro- β -naphthol (light brown), 1-methyl- β -naphthol (yellow-brown), 1-sulphomethyl- β -naphthol (yellow-red).

C. HOLLINS.

Manufacture of azo dyes. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING

(E.P. 255,900, 26.7.26. Ger., 25.7.25. Addn. to E.P. 231,529; B., 1925, 909).—4-Arylsulphonylalkyl- (or aralkyl-)amino-2-aminophenyl alkyl or aralkyl ethers, obtained by the action of an arylsulphonyl chloride on a 2-nitro-4-aminophenyl ether with subsequent alkylation or aralkylation and reduction, are diazotised and coupled in substance or on the fibre with a 2:3-hydroxynaphthoic arylamide. The dyes give redder shades than those of the prior patent. 4-Benzenesulphonylmethylamino-*o*-anisidine, diazotised and coupled with 2:3-hydroxynaphthoic 5-chloro-*o*-toluidide, gives a bordeaux-red. C. HOLLINS.

Manufacture of insoluble azo dyes. I. G. FARBENIND. A.-G. (F.P. 617,211, 8.6.26. Ger., 26.6.25).—Hydroxynaphthacarbazoles are coupled in substance or on the fibre with unsulphonated diazo compounds. 2-Hydroxy- α -naphthacarbazole (I), obtained by alkaline



fusion of the Bucherer reaction product from phenylhydrazine and α -naphthylamine-7-sulphonic acid, is coupled with diazotised *m*-nitroaniline (red-brown) or 4-nitro-*o*-anisidine (black-brown); 4-hydroxy- α -naphthacarbazole (II, from Brönnner acid) with 4-nitro-*o*-anisidine (black-violet); 2-hydroxy-9-methyl- α -naphthacarbazole (as I) with 4-nitro-*o*-toluidine (bordeaux).

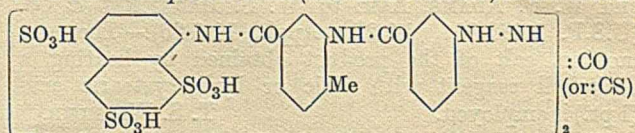
C. HOLLINS.

Manufacture of disazo dyes. I. G. FARBENIND. A.-G., Assees. of R. KIRCHHOFF, R. HAUGWITZ, and M. CANTOR (G.P. 439,518, 16.5.25. Fr., 29.4.26).—Dyes suitable especially for weighted silk are obtained by condensing certain aminodisazo dyes with nitrobenzene derivatives containing a reactive substituent (*e.g.*, 1-chloro-2:4-dinitrobenzene, 1-chloro-4-nitrobenzene-2-sulphonic acid). The disazo dyes are those formed by coupling a 1:8-aminonaphtholsulphonic acid with 1 mol. of a suitable diazo compound and 1 mol. of a *m*- or *p*-nitrodiazo compound or of a nitrophenyl-*m*- or *p*-aminodiazocompound, the nitro-group being subsequently reduced.

C. HOLLINS.

Manufacture of carbazides and thiocarbazides of the naphthalene series. BRIT. DYESTUFFS CORP., LTD., G. M. DYSON, F. A. MASON, and A. RENSHAW (E.P. 278,037, 25.5.26).—Carbazides or thiocarbazides obtained by the action of phosgene or of thiophosgene on hydrazines of the type, $P[NH \cdot X \cdot Q]_{n-2} \cdot NH \cdot Y \cdot R \cdot NH \cdot NH_2$, in which *n* is greater than 1, P is a sulphonated naphthalene or acenaphthene residue, Q and R are aryl, aralkyl, or aralkylene residues, and X and Y are carbonyl or sulphonyl groups, are found to be valuable agents for destruction of blood parasites whilst comparatively non-toxic to man. They are also intermediates for dyes. $P \cdot NH_2$ may be, *e.g.*, di- or tri-sulphonic acids of naphthylamines, aminonaphthols, 5-aminoacenaphthene, etc. $NH_2 \cdot Q \cdot X \cdot OH$ and $NH_2 \cdot R \cdot Y \cdot OH$ may

be aminobenzoic, aminotoluic, aminocinnamic, aminophenylacetic, aminonaphthoic, anilinesulphonic, or naphthylaminesulphonic acids or their substitution products. The amine $P \cdot NH_2$ is first treated with a nitroaryl chloride or a nitroarylsulphonyl chloride, and the nitro-group is reduced. A further nitroaryl or nitroarylsulphonyl group may be introduced, followed by reduction to an amine, and this process repeated. Any of the amines so formed may be diazotised, reduced to a hydrazine, and finally treated with phosgene or thiophosgene. The preparation of carbazido- and thiocarbazido-*bis*-2-(*m*-benzamido)-4-toluoyl-1'-naphthylamine-4':6':8'-trisulphonic acids (annexed formula) is described.



C. HOLLINS.

Manufacture of dinaphthyldicarboxylic acids.

W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 278,100, 6.7.26).—Aminonaphthoic acids or esters are diazotised and decomposed by means of a suitable reducing agent, particularly ammoniacal cuprous oxide solution prepared by passing sulphur dioxide into a solution of copper sulphate and ammonia. 1:8-Aminonaphthoic acid is thus converted into 1:1'-dinaphthyl-8:8'-dicarboxylic acid, m.p. above 300° (ethyl ester, m.p. 183°). 2:2'-Dinaphthyl-3:3'-dicarboxylic acid, m.p. 290–292° (ethyl ester, m.p. 171°), 1:1'-dinaphthyl-2:2'-dicarboxylic acid, m.p. 266°, 1:1'-dichloro-2:2'-dinaphthyl-3:3'-dicarboxylic acid, m.p. 198°, 4:4'-dibromo-1:1'-dinaphthyl-8:8'-dicarboxylic acid, m.p. above 300°, the corresponding 4:4'-dichloro-compound, m.p. above 300° (ethyl ester, m.p. 194°), the 5:5'-dichloro-compound, the 5:5'-dimethoxy- and diethoxy-compounds, m.p. above 300°, and the 4:4'-disulpho-compound, are similarly prepared. 1-Chloro-2-amino-3-naphthoic acid, m.p. 254°, is obtained from 9-chloro- $\beta\beta$ -naphthisatin; 4-bromo-, 4-chloro-, and 5-chloro-1-amino-8-naphthoic acids from the corresponding naphthastyrils.

C. HOLLINS.

Preparation of colouring matters [of the anthraquinone series]. L. J. HOOLEY, J. THOMAS, and SCOTTISH DYES, LTD. (E.P. 278,417, 2.7.26).—3-Chloro-1-bromo-2-aminoanthraquinone or 1:3-dichloro-2-aminoanthraquinone is condensed with primary amines (aniline, *p*-toluidine, methylamine) to give acetate silk dyes, which are converted by treatment with sulphuric acid or oleum into acid dyes for wool or silk.

C. HOLLINS.

[Manufacture of] anthraquinone derivatives [for use as dyes and intermediates]. W. SMITH, J. THOMAS, and SCOTTISH DYES, LTD. (E.P. 287,039, 26.5. and 10.6.26).—1-Halogeno-2-anthraquinonylurethanes are heated with a metal (copper) in presence or absence of a solvent (nitrobenzene) to give flavantrones directly, or at lower temperatures to give 2:2'-diurethano-1:1'-dianthraquinonyls, which may be converted into flavantrones by heating alone, or with concentrated sulphuric acid or alcoholic alkali, or in an inert solvent (nitrobenzene or naphthalene). 2:2'-Diurethano-1:1'-dianthraquinonyl melts above 250°.

C. HOLLINS.

Manufacture of anthraquinone derivatives. I. G. FARBENIND. A.-G., Assees. of P. NAWIASKY (G.P. 440,891, 23.1.25).—Aromatic *p*-quinones and their derivatives, including halogen compounds, are condensed with monoacylated diaminoanthraquinones, or with diaminoanthraquinones and subsequently acylated. The product from 1:5-diaminoanthraquinone and α -naphthaquinone gives with benzoyl chloride an orange vat dye (yellow-brown on wool), with oxalyl chloride an orange, with adipyl chloride an orange (red-brown on wool), with nitroanthraquinone-2-carboxyl chloride a red vat dye; the 1:4-isomeride dyes bordeaux. Benzoylation of the product from 1:5-diaminoanthraquinone and *p*-benzoquinone gives a brown vat dye, becoming orange by treatment with hypochlorite. C. HOLLINS.

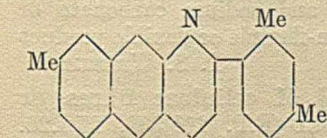
Manufacture of dyes and intermediates. R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (E.P. 278,112, 13.7.26; cf. E.P. 251,313; B., 1926, 576).—The processes of the prior patent are improved by oxidising the benzanthrone at low temperatures. *E.g.*, a solution of manganese dioxide in 10 pts. of concentrated sulphuric acid at 100–110° is cooled to 0° and added to a solution of benzanthrone in sulphuric acid at 0°.

C. HOLLINS.

Manufacture of dibenzanthronyls. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 278,047, 31.5.26. Addn. to E.P. 203,533; B., 1923, 1064 A).—The 4:4'-dibenzanthronyls of the prior patent are obtained by condensation of 4-halogenobenzanthrones with benzanthrone having free 4-position. The reaction proceeds in presence of alkaline condensing agents at 0–15°, or at 90–95°, and may be used for preparing unsymmetrical dibenzanthronyls. 4-Chlorobenzanthrone, m.p. 204°, and benzanthrone with sodioaniline in aniline give 4:4'-dibenzanthronyl. 9-Methyl- (m.p. 330–331°), 9-chloro- (m.p. 312–314°), and 10-chloro- (m.p. 305°) -dibenzanthronyls are obtained similarly from 4-chlorobenzanthrone and 9-methyl-, 9-chloro-, and 10-chlorobenzanthrones, respectively. 4:9- and 4:10-Dichlorobenzanthrones (the former prepared from 2:6-, the latter, m.p. 278°, from 2:7-dichloroanthraquinone) condense with 6- and 7-chlorobenzanthrones to give, respectively, 9:9'- and 10:10'-dichlorodibenzanthronyls.

C. HOLLINS.

Manufacture of heterocyclic compounds. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 258,553, 14.7.26. Ger., 15.9.26. Addn. to E.P. 251,270; B., 1927, 742).—The process of the prior patent is extended to the manufacture of polynuclear heterocyclic systems. A heterocyclic ketone, carbinol, or methane, in which there is an *ortho*-methyl or -methylene group, is cyclised by passing its vapour, in presence or absence of an oxidant, over a hot porous catalyst (active carbon). The hydroxy-methylene derivative of *m*-4-xylyl methyl ketone, when heated with ammonium acetate (cf. G.P. 418,218; B., 1926, 216), gives 2-



over active carbon at 400–420° to form 2-(4'-*m*-xylyl)-8-

methyl-1-anthrapyridine [2-(4'-*m*-xylyl)-8-methyl- $\beta\beta$ -naphthaquinoline], m.p. 172°. C. HOLLINS.

Manufacture of chlorides of *o*-hydroxycarboxylic acids of polynuclear hydrocarbons. BRIT. SYNTHETICS, LTD., and E. B. HIGGINS (E.P. 278,463, 20.7.26).—The dry monosodium salt of a polynuclear aromatic *o*-hydroxycarboxylic acid (2:3-hydroxynaphthoic acid) is ground with one equivalent of thionyl chloride below 60°, *e.g.*, in a ball-mill for 10 min. On removal of sulphur dioxide in a current of air, 2-hydroxy-3-naphthoyl chloride is obtained pure and stable in quantitative yield. C. HOLLINS.

Catalytic oxidation of organic compounds. I. G. FARBENIND. A.-G. (G.P. 441,163, 15.12.22).—A succession of catalysts is employed. The first catalyst should be active at moderate temperatures, and the heat of reaction heats the gases to a temperature suitable for the next catalyst. The rate of oxidation is thus moderated by the use of less active catalysts as the temperature rises. *E.g.*, naphthalene vapour and air are led first over vanadic acid on metal granules at 320°, then over vanadic acid on powdered kieselguhr at 350–410°, phthalic anhydride being obtained in good yield. In some cases it is desirable to conduct the oxidation in stages in order to prevent overheating; *e.g.*, acenaphthene is oxidised first to acenaphthylene, and then to naphthalic acid.

C. HOLLINS.

Reduction of organic compounds. I. G. FARBENIND. A.-G., Assees. of M. MÜLLER-CUNRADI, M. LUTHER, and K. PIEROH (G.P. 441,179, 18.1.25).—The reducing agent may be prepared by heating iron pentacarbonyl with methyl-alcoholic potassium hydroxide under reflux. On adding nitrobenzene and distilling off the methyl alcohol, aniline is obtained by steam distillation. If iron pentacarbonyl is added slowly to a warmed mixture of nitrobenzene and dilute potassium hydroxide solution, carbanilide and aniline are obtained. Vat dyes of the indigo, thioindigo, anthraquinone, and quinone groups, sulphide dyes, and azines are reduced to leuco-compounds by adding iron pentacarbonyl to a stirred aqueous alkaline suspension of the vat dye etc. at 60–70°. Azoxybenzene and hydrazobenzene may be obtained from nitrobenzene, *o*-aminophenol quantitatively from *o*-nitrophenol, and benzoin from benzil. C. HOLLINS.

Manufacture of aryl esters of nitroaminobenzene-sulphonic acids. W. DUISBERG, W. HENTRICH, and L. ZEH, ASSIS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,646,785, 25.10.27. Appl., 9.6.25. Ger., 14.6.24).—See E.P. 235,598; B., 1925, 704.

Manufacture of dinitroperylenequinone. A. ZINKE, ASSR. to F. BENSA (U.S.P. 1,642,263, 13.9.27. Appl., 15.6.23. Austr., 20.6.22).—See E.P. 199,720; B., 1923, 1170 A.

Decolorisation of aqueous liquids (E.P. 278,485). Friction surfaces (E.P. 278,465).—See I.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Action of atmospheric influences on fibrous material. H. SOMMER (Chem.-Ztg., 1927, 51, 777–778).—Samples of different fabrics (wool, cotton, silk, hemp, etc.) were exposed to the weather in both clean and

polluted atmospheres for periods of 3, 6, 9, and 12 months, the total number of hours' sunshine being recorded for each period of exposure. With woollen fabrics, dyed and undyed, atrophy of the hairs on the side exposed to the light was observed after three months, the other side being unaffected. Deterioration of the wool was indicated by loss in weight and strength of the material, and was approximately proportional to the number of hours of sunshine; it is attributed in all cases to the action of rays of short wave-length. The presence of sulphuric acid due to the oxidation of contained sulphur was shown. Moisture also accelerates the process and increases the loss in weight by causing the decomposition products to go into solution. The extent of the damage depends also on the thickness and porosity of the material. Pre-treatment of the wool with sulphuric acid retards the destruction, whilst treatment with alkalis hastens it. Dyeing, according to the dyestuff used, exerts a more or less protective action which is approximately proportional to the depth of the dye. Chroming and treatment with copper sulphate result in increased protection. The strength of raw, degummed, and weighted silk was reduced to one tenth of its original value by exposure for three months, a marked loss in weight being also observed; fabrics from vegetable fibres behaved similarly. Besides the destruction of the cuticle of cotton and of the incrusting substances of bast fibres, the cellulose itself undergoes a change which is indicated by the solubility of the modified material in boiling sodium hydroxide, a behaviour analogous to that of oxycellulose. The change is ascribed not to an oxidation process, but to the formation of "photocellulose." If the sensitiveness to weathering be judged by the number of hours of sunshine necessary to reduce the original strength of the fabric to half its value, the materials (not allowing for their unequal thicknesses) may be arranged in the following decreasing order: silk, jute, artificial silk, cotton, flax, hemp, raw wool, and chromed wool.

B. P. RIDGE.

Influence of the preliminary alkaline treatment and of the time of chlorination in obtaining cellulose by the chlorine process. I. MUTTI and M. VENTURI (*Annali Chim. Appl.*, 1927, 17, 391—401).—When, in the manufacture of cellulose by the chlorine process, a high yield is desired, 1% sodium hydroxide solution should be used in the preliminary treatment of the material, whereas when a purer product in lower yield is required, 4% sodium hydroxide solution should be employed. In the former case the action of the chlorine should not be too prolonged, in order that the resulting product may not suffer injury; in the latter case, however, no such injury results, and the products obtained contain up to 86% of α -cellulose and are well suited to the manufacture of artificial silk. Increase of the concentration of the sodium hydroxide solution used in the preliminary treatment from 5 to 10% diminishes the final yield without improving the quality of the cellulose obtained.

T. H. POPE.

Reactions relating to carbohydrates and polysaccharides. XII. Action of chromic acid on cotton cellulose. H. HIBBERT and S. M. HASSAN (*J.S.C.I.*, 1927, 46, 407—411 T; cf. Hibbert and Parsons, *ibid.*,

1925, 44, 473 T).—Dextrose and α -methylglucoside on oxidation with chromic acid in presence of 3—6% sulphuric acid, using a ratio of 2 atoms of "active oxygen" per dextrose mol., yield formic acid and a furfuraldehyde-yielding constituent in the ratio 2:1, but the amount of glycuronic acid formed in both cases is small, and α -methylglucoside probably undergoes hydrolysis under these conditions. Acetic acid was not detected among the oxidation products. Lactose with chromic acid over a wide range (up to about 8 atoms of "active oxygen" per mol.) gives a constant amount of furfuraldehyde-forming constituent, indicating the presence of an intermediate which is stable towards the oxidising agent. Cotton cellulose when treated with chromic acid (2 atoms of "active oxygen" per C_6 complex) in presence of sulphuric acid of varying concentration (10—50%) gives a more degraded product in decreasing yield with increase in acid concentration, but the copper number of the product remains practically constant whilst the furfuraldehyde value decreases, showing that such values are not suitable for the determination of the character of an oxidised cellulose. Cotton cellulose on oxidation with varying amounts of chromic acid (0.25—3 atoms of "active oxygen") in 10% sulphuric acid solution gives a high yield of an oxidised cellulose (94.6—82%), and the product has approximately the same copper number and furfuraldehyde and Lefèvre carbon dioxide values, indicating a gradual progressive oxidation of the fibre surface. Gluconic and saccharic acids do not evolve carbon dioxide when boiled with 12% hydrochloric acid nor reduce Fehling solution.

W. J. POWELL.

Use of waste wood products. SCHWALBE.—See II.

Sulphite-cellulose waste liquor as manure. GÖR-BING.—See XVI.

PATENTS.

Manufacture of artificial silk [from cotton rags]. J. UMBACH (E.P. 278,131, 5.8.26).—Viscose silk having 30% higher elasticity than silk prepared from fir wood sulphite-pulp or cotton linters is manufactured from waste coloured cotton rags, previously purified by digestion with caustic alkalis under pressure at a high temperature. A satisfactory treatment consists in heating the rags for 6—8 hrs. under 4—5 atm. pressure (240—250°) in a 1% solution of caustic soda; subsequent bleaching is unnecessary.

A. J. HALL.

Conditioning [oiling of] textile fibres. R. B. SMITH, Assr. to H. L. SIEVER (U.S.P. 1,642,092, 13.9.27. Appl., 28.6.26; cf. U.S.P. 1,550,396; B., 1925, 842).—Raw cotton or other textile stock is treated with oil atomised with steam.

T. S. WHEELER.

Manufacture of a composition for treating [fire-proofing] fibre products. C. R. FELIX (U.S.P. 1,643,116, 20.9.27. Appl., 6.3.25).—Cotton, paper, etc. is immersed at 85° in a solution containing sodium silicate, sodium tungstate, and sodium carbonate or boric acid, according as a non-flexible or flexible product is required.

T. S. WHEELER.

Treatment [delustring] of cellulose fibres or filaments. B. BORZYKOWSKI (E.P. 261,333, 27.4.26. Ger., 11.11.25).—Viscose silk having a natural silk-like sheen is obtained by bleaching crude viscose before

desulphurisation, and afterwards treating it with a 1–2% solution of an aluminium, magnesium, or zinc salt.

A. J. HALL.

Application of cellulose ethers or esters [dioxan as a solvent]. I. G. FARBENIND. A.-G. (E.P. 275,653, 15.12.25. Ger., 23.12.24).—1:4-Dioxan (diethylene dioxide) dissolves cellulose ethers or esters with or without addition of resins, dyes, and/or other solvents; the solutions are useful for spinning artificial silk or as varnishes.

C. HOLLINS.

Treatment of nitrocellulose [for lacquers] to render it safe during storage or transport. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 252,382, 20.5.26. Ger., 20.5.25).—Lacquer nitrocellulose (of a comparatively low degree of nitration) is moistened with butyl alcohol or a higher alcohol, *e.g.*, amyl alcohol or glycerol, to render it safe during transport or storage and facilitate subsequent treatment in the manufacture of lacquers.

S. S. WOOLF.

Process for bleaching [wood pulp]. C. B. THORNE (U.S.P. 1,643,566, 27.9.27. Appl., 16.10.25).—Wood pulp, concentrated to 15–20%, is introduced at a constant rate at the top of a tower, in which it is treated with bleaching material, being meanwhile agitated mechanically and aerated. The bleached material is withdrawn at the bottom of the tower and washed.

T. S. WHEELER.

Batting process [for fibres]. B. WESCOTT, ASSR. to RUBBER LATEX RES. CORP. (U.S.P. 1,646,605, 25.10.27. Appl., 18.11.22).—See E.P. 232,763; B., 1925, 515.

Manufacture of artificial silk. M. HÖLKEN, JUN. (U.S.P. 1,641,588, 6.9.27. Appl., 25.1.23. Ger., 12.7.20).—See E.P. 211,691; B., 1924, 374.

Manufacture of paper pulp. B. S. SUMMERS (U.S.P. 1,643,826, 27.9.27. Appl., 22.12.23).—See Can. P. 246,537; B., 1926, 153.

Separation of the fibre from the pulp of sisal or like leaves. HYDRAULIC ENGINEERING CO., LTD., and E. D. RUTHERFOORD (E.P. 278,457, 14.7.26).

Manufacture of twisted artificial silk from cuprammonium cellulose solutions by the stretch-spinning process. J. P. BEMBERG A.-G. (E.P. 260,564 and Addn. E.P. 268,393, [A] 1.10.26, [B] 28.3.27. Ger., [A] 2.11.25, [B] 27.3.26).

Manufacture of finely-perforated ceramic plates for use in the manufacture of artificial silk etc. J. F. SCHEID, V. TOUNDORF, and C. ZEISS (E.P. 278,097, 3.7.26).

Yeast (F.P. 614,037).—See XVIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Process for "brightening" [dyed] silk. K. WOLFGANG (Kunstseide, 1927, 9, 517–518).—Much more permanent results are obtained in the usual process for brightening dyed silk by treatment with a soap and an organic acid if oleic acid is added to the potash-olive oil soap usually employed. This improved process has no deleterious effect on the elasticity and durability of the silk.

A. J. HALL.

Light sources for testing the fastness of dyes. I. H. HADFIELD (J. Text. Inst., 1927, 18, 527–558 T).—A summary comprising information published prior to December, 1925, bearing on the suitability of various artificial sources as standard lamps for dye-fading tests.

B. P. RIDGE.

PATENTS.

Dyeing of vegetable fibre. N. N. VOROSHTZOV, and STAATLICHE VEREINIGTE ANILINFABR. (G.P. 441,053, 24.11.22. Russia, 23.3.21).—Cotton is dyed from an alkaline bath with mono-, di-, or poly-azo dyes containing 6-nitro- β -naphthylamine-8-sulphonic acid as first component. With α -naphthol-4-sulphonic acid this gives a violet-red; with α -naphthylamine-6(or 7)-sulphonic acid a grey-violet, which may be diazotised on the fibre and developed to blue to black shades.

C. HOLLINS.

Improvement of washing- and milling-fastness of acid dyeings on wool. I. G. FARBENIND. A.-G., Assees. of F. JUST (G.P. 440,606, 31.1.24).—Acid wool dyeings are after-treated with nitrous acid.

C. HOLLINS.

Fixation of basic dyes on cotton. I. G. FARBENIND. A.-G., Assees. of A. GÜNTHER, J. HALLER, and E. KÖSTER (G.P. 441,326, 20.2.25).—Arylamides of aromatic hydroxy-acids (*e.g.*, 2:3-hydroxynaphthoic β -naphthylamide, 1:2-hydroxynaphthoic β -naphthylamide, m.p. 180°, disalicylbenzidine) are used as mordants for basic colours, and give fast dyeings without after-treatment with tartar emetic. The colour bath is exhausted better than with tannin, and deeper shades are obtainable.

C. HOLLINS.

[Dyeing of "mixed" cellulose acetate] threads, cords, and fabrics. BRIT. CELANESE, LTD., C. W. PALMER, and S. M. FULTON (E.P. 278,116, 21.7.26).—Yarns and fabrics manufactured so that they contain threads of normal and delustred cellulose acetate silk have special advantages in handle and appearance. Since the process of delustring (*e.g.*, treatment of the silk with wet steam at 100°) decreases the affinity of cellulose acetate silk for basic dyes such as Capri Blue, two-colour effects may be obtained by suitably dyeing the manufactured products.

A. J. HALL.

Dyeing of cellulose esters. I. G. FARBENIND. A.-G., Assees. of H. EICHWEDE, E. FISCHER, and C. E. MÜLLER (G.P. 441,325, 28.9.24).—Cellulose esters (*e.g.*, acetate silk) are dyed with monoazo dyes from diphenylamine-monosulphonic acid coupled with unsulphonated diazo compounds of the benzene or naphthalene series. *E.g.*, *m*-nitroaniline \rightarrow diphenylaminesulphonic acid gives a golden yellow.

C. HOLLINS.

Printing and fixing basic dyes on acetate silk. I. G. FARBENIND. A.-G., Assees. of A. SCHNEEVOIGT (G.P. 441,327, 18.4.25).—Clear prints are obtained by the use of phenols (phenol, naphthols, resorcinols, cresolsulphonic acids) in printing or fixing pastes with basis colours. The addition of tannin and after-treatment with tartar emetic are unnecessary.

C. HOLLINS.

Vat dyes and dyeing (E.P. 278,399).—See IV.

Bleaching of wood pulp (U.S.P. 1,643,566).—See V.

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

Blattner process for the preparation of caustic soda from sodium carbonate. HIRCHBERG (Chem.-Ztg., 1927, 51, 765).—Caustic soda can be produced in nearly theoretical yield by heating a mixture of 150 pts. of iron oxide with 100 pts. of sodium carbonate, such process being a modification of that described in E.P. 203,271 (B., 1923, 1070 A). The reaction is hastened by the use of a catalyst. Sodium carbonate of varying grades of purity can be used, from the pure material obtained from the ammonia-soda process to a naturally occurring carbonate containing 12% of sodium chloride and 10% of sodium sulphate. The carbon dioxide evolved during the process is treated with sodium carbonate in absorption chambers or brought into contact in towers with a solution of the same. Sodium bicarbonate is produced from which pure carbon dioxide and sodium carbonate can be regenerated. The use of a rotary furnace filled completely with the reaction material is not considered suitable. An economical type of oven is suggested in which the heating is carried on continuously, the finely-ground and well-mixed material being introduced in a thin layer at one end and carried directly into water at the other. R. C. ODAMS.

[Catalytic] oxidation of ammonia. F. RASCHIG (Z. angew. Chem., 1927, 40, 1183—1185).—Andrussov and Bodenstein's view (B., 1927, 217) that nitroxyl, NHO , is the primary product of the oxidation of ammonia and that it decomposes into nitric oxide and hydrogen is criticised as being without experimental foundation and contrary to the known properties of this compound. The author's view that imide, NH , is the intermediate product is reiterated, and the details of the experiment in which he found hydrazine in the gases from the combustion of ammonia with insufficient oxygen ($\text{NH}_3 + \text{NH} = \text{N}_2\text{H}_4$) are described. The combustion was effected by a coal-gas flame within a flask fed with ammonia and oxygen in regulated amounts and containing ammonia solution. The hydrazine was determined by titration with iodine or as benzylideneazine.

C. IRWIN.

The ferric sulphate-sulphuric acid process. O. C. RALSTON (U.S. Bur. Mines Bull. 260, 1927, 1—61).—Ferric salts are suitable for leaching low-grade ores of copper and other metals, dissolving sulphides as well as oxidised ores. Hitherto, however, no economical method has been known of oxidising the resulting ferrous salts for further use. It has been discovered that ferrous iron and sulphur dioxide catalyse each other's oxidation. When sulphur dioxide diluted with air is passed through a solution of ferrous sulphate the reaction $2\text{FeSO}_4 + \text{SO}_2 + \text{O}_2 = \text{Fe}_2(\text{SO}_4)_3$ occurs. When oxidation of ferrous sulphate has reached its limit for the given conditions, oxidation of sulphur dioxide to sulphuric acid still proceeds up to a certain concentration. These two reactions were studied in detail. The most satisfactory apparatus was found to be a tall glass jar filled with solution with means for passing in bubbles of gas of size not above 1 mm. at the bottom. On a larger scale a high wooden tank was used. It was found that if the ferrous sulphate solution became acid, which

only happened in inefficient apparatus, the rate of oxidation of iron fell off. If the solution was initially acid, a lag in the start of the reaction occurred. After all the iron has been oxidised formation of acid can proceed, at economic rate, up to a concentration of 110 g. of sulphuric acid per litre provided the solution contains 0.5—1.0 g. of ferrous iron per litre. Any concentration of ferrous iron up to saturation can be oxidised. For the oxidation to be complete, however, an optimum ratio of sulphur dioxide to oxygen is necessary. The maximum percentage of sulphur dioxide for 1 mm. bubbles is 7% by volume. For sulphuric acid synthesis after the oxidation of the iron is complete this can be increased. The velocity of the reaction is inversely proportional to the square root of the diameter of the orifice producing the bubbles. With increase of temperature it tends to reach a maximum at about 40°. Increase of pressure gives only a small effect. Copper if present is detrimental to the reaction, and in any application to leaching liquors for copper ore will require removal. If gases from burning sulphur are to be used the desirable concentration will be 5.5% SO_2 , from pyrites 5.0% SO_2 (in each case for iron oxidation). For a 10 ft. column with a diaphragm containing perforations of 0.118 mm. diam. (required to give 1 mm. bubbles) at a space velocity of 500 c.c. of gas per litre per min. the total pressure required is calculated to be 4.93 lb./in.² The total power requirements of the process for blowing are estimated at 3.0—3.5 kw. per 1000 lb. of copper treated per 24 hrs. C. IRWIN.

Production of small bubbles of gas in liquids. MAIER.—See I.

Wood preservatives. CURTIN.—See IX.

PATENTS.

Treatment of crude nitrate of soda (caliche). I. G. FARBERIND. A.-G. (E.P. 266,735, 24.2.27. Ger., 24.2.26).—Caliche is subjected to prolonged treatment at ordinary temperature with a quantity of water such that the sodium nitrate is almost completely extracted but a large portion of the other soluble salts remains undissolved; the resulting solution is then used to leach at 100° a further quantity of caliche, after which it is cooled so that the sodium nitrate separates out. Instead of using fresh caliche for cold-leaching purposes, residues from any warm-leaching process may be employed, and instead of water for the cold-leaching, the solution remaining after the deposition of nitrate crystals may be used. W. G. CAREY.

Separation of native sodium salts, e.g., thénardite and glauberite, from the accompanying rock salt gangue. F. STEIN (G.P. 442,646, 21.10.24).—The crude salt mixture is leached with water or liquors from previous operations in such a way that the rock salt dissolves leaving a residue of sodium sulphate or glauberite. If water is used the crude salt mixture is treated continuously on the counter-current principle with a rapid stream of very cold water. If the mixture contains an excess of rock salt the mother-liquors from the manufacture of Glauber's salt may be used, in which case most of the sodium sulphate in the liquors is deposited together with the insoluble sodium sulphate in the crude salt; the sodium chloride solution so obtained

may be freed from the calcium and sodium sulphate it contains by seeding with artificial glauberite. If the mixture contains an excess of sodium sulphate an aqueous suspension of gypsum dissolves the sodium chloride, whilst the sodium sulphate is converted into glauberite; alternatively, gypsum may be added together with mother-liquors from Glauber's salt.

A. R. POWELL.

Manufacture [purification] of sodium phosphate. H. HOWARD, Assr. to GRASSELLI CHEMICAL CO. (U.S.P. 1,642,244, 13.9.27. Appl., 31.10.22).—Crude sodium phosphate solution obtained in the manufacture of the salt from phosphate rock is decolorised by treatment with chlorine.

T. S. WHEELER.

Production of sodamide. J. F. WAIT, Assr. to NAT. ANILINE & CHEMICAL CO., INC. (U.S.P. 1,646,372, 18.10.27. Appl., 18.12.24).—Gaseous ammonia is cooled below 5° and passed through molten sodium.

H. ROYAL-DAWSON.

Manufacture of ammonium fluorides. J. W. PROCTOR, Assr. to GEN. CHEMICAL CO. (U.S.P. 1,642,788, 20.9.27. Appl., 5.1.23).—Saturated ammonium fluoride solution is treated in a cycle first with hydrogen fluoride and then with ammonia.

T. S. WHEELER.

Precipitation of heavy metals from ammoniacal solutions. I. G. FARBENIND. A.-G. (E.P. 269,164, 31.3.27. Ger., 9.4.26).—The solutions are treated with carbon monoxide, hydrogen, or mixtures containing these gases under pressure and at an elevated temperature. By the application of different temperatures and pressures, several metals may be precipitated separately in succession. Noble metals may be precipitated either under increased pressure and at normal temperatures, or at an elevated temperature and under normal pressure.

M. E. NOTTAGE.

Preparation of cyanogen compounds [calcium cyanide]. R. W. POINDEXTER, JUN. and W. E. OLBERG, Assrs. to CALIFORNIA CYANIDE CO., INC. (U.S.P. 1,642,694, 20.9.27. Appl., 19.8.26; cf. U.S.P. 1,573,732; B., 1926, 486).—Anhydrous hydrocyanic acid is treated with calcium carbide in presence of a suitable catalyst, e.g., ammonium bromide, thiocyanate, or iodide, or calcium bromide.

T. S. WHEELER.

Treatment of lead chloride or basic chloride and application thereof to the winning of lead from ores, residues, etc. S. C. SMITH, and CHEMICAL & METALLURGICAL CORP., LTD. (E.P. 278,093, 2.7.26).—The process of E.P. 237,308 (B., 1925, 758) is modified in that carbon dioxide, which is passed into a suspension of lead chloride in ammonia, is added at such a rate that it forms lead carbonate as soon as it enters the suspension so that dissolved carbon dioxide is avoided.

W. G. CAREY.

Preparation of aluminium sulphate, free from iron, from alum. VEREINIGTE ALUMINIUM WERKE A.-G. (G.P. 442,596, 13.9.23).—The ammonium or potassium alum obtained by treating clay or other aluminous material with ammonium or potassium sulphate is decomposed with 50–60% sulphuric acid solution and the precipitated aluminium sulphate, after washing with more sulphuric acid, is freed from excess acid by agitation with a cold saturated solution of aluminium sulphate obtained in a previous operation.

A. R. POWELL.

Production of silicates. G. HERFELDT (G.P. 442,717, 29.4.24).—Mixtures of silicates with oxides such as lime and magnesia interact with water under the influence of the heat of reaction and in the presence of colloids capable of accelerating the reaction.

L. A. COLES.

Production of normal and other standard solutions. E. DE HAEN CHEM. FABR. "LIST" G.M.B.H. (G.P. 442,706, 23.6.20).—Suitable weights of the desired materials are stored in air-tight bottles, and the contents are dissolved and made up to the correct volume when the solutions are required for use.

L. A. COLES.

Granules for the absorption of [acidic] gases. L. A. LEVY (E.P. 277,540, 25.1.27).—Hydrated barium hydroxide (270 pts.) is mixed with slaked lime (180 pts.), caustic soda or potash (15 pts.), and, if desired, activated charcoal (49 pts.; 100-mesh). Preferably the soda is dissolved in water (25 pts.) and the barium hydroxide added, the mass being then heated until fluid, whereupon the lime and charcoal, intimately mixed, are stirred in and the mass on cooling is granulated.

H. HOLMES.

Catalytic oxidation of carbon monoxide. I. G. FARBENIND. A.-G. (E.P. 265,624, 7.2.27. Ger., 8.2.26).—Active adsorbent substances of gel character, e.g., silica, stannic acid, etc., are used to eliminate vapours and gases injurious to catalysts used in the oxidation of carbon monoxide to the dioxide.

H. ROYAL-DAWSON.

Continuous distillation of crude carbon disulphide to obtain pure carbon disulphide. I. G. FARBENIND. A.-G., Assees. of CHEM. FABR. GRIESHEIM-ELEKTRON (E.P. 260,236, 8.10.26. Ger., 24.10.25).—The rectification is carried out in two similar columns in series fitted with stills and reflux condensers. The first column is irrigated with crude carbon disulphide and the second with partially purified carbon disulphide from the first column, while the still of the first column is kept at a temperature above the m.p. of sulphur and that of the second slightly above the b.p. of pure carbon disulphide. The carbon disulphide vapours are discharged from the lower ends of the columns and are led to the level of the upper ends prior to condensation, while the waste gases (e.g., hydrogen sulphide) issuing from the reflux condensers are further cooled and the carbon disulphide obtained thereby is again used for irrigating the first column. The sulphur separating from the carbon disulphide is withdrawn as a liquid from the still of the first column.

W. G. CAREY.

Treatment of sulphur. J. W. SCHWAB, Assr. to TEXAS GULF SULPHUR CO. (U.S.P. 1,646,838, 25.10.27. Appl., 30.1.24).—The normal colour of discoloured sulphur is restored by passing the molten material through a layer of fuller's earth.

H. ROYAL-DAWSON.

Oxidation of ammonia by means of oxygen. F. G. LILJENROTH (E.P. 276,295, 26.11.26. Swed., 19.8.26).—See F.P. 610,010 and 610,160; B., 1927, 251.

VIII.—GLASS; CERAMICS.

Determination of sulphur present as sulphide in glass. H. HEINRICH (Z. anorg. Chem., 1927, 166, 299–305).—Sulphides in glass may be determined by warming 1 g. of the finely-powdered glass with a mixture of 5 c.c. of water, 25 c.c. of dilute (1:1) hydrochloric acid, and 5 c.c. of 48% hydrofluoric acid in an apparatus

of sulphide-free glass, and passing the hydrogen sulphide evolved into 0.01*N*-iodine solution. Before the acid is added, the air is displaced from the apparatus with a stream of carbon dioxide, which is continued until all the hydrogen sulphide has been expelled. As, in general, the amount of sulphide present is very small, it is desirable to make a separate qualitative test in which the hydrogen sulphide is detected with a lead acetate paper. Analyses made in this way with various glasses show that the effect of a sulphide on the colour of glass is very much greater than that of an equal amount of the corresponding oxide.

R. CUTHILL.

Refractories for the electric steel furnace. E. KOTHNY (Feuerfest, 1927, 3, 157—164).—The characteristics of fourteen different refractories are described and their actual or possible application in electric furnace construction is indicated. The relative costs per cub. m. of brickwork are tabulated for six different types of refractory bricks. Silica bricks may be used in all parts of radiation furnaces and above the slag line in basic furnaces. By reason of their comparatively low price, they constitute one of the chief materials for electric furnace construction. Magnesite bricks are sensitive to abrupt changes of temperature; they are only applied where their slag-resisting property can be utilised. Calcined magnesite and calcined dolomite are suitable for basic furnaces. Chromite bricks may be used to form a neutral layer between acid and basic brickwork. Carborundum bricks are destroyed by the action of slags, and can therefore only be used in the roofs of furnaces. Corundum bricks are very refractory and resistant to temperature changes. Magnesite spinel bricks are very refractory and highly resistant to basic slags and to changes of temperature. Sillimanite bricks are attacked by acid slags. Fused magnesite is suitable for low-frequency induction furnaces. Zirconia bricks have a very high m.p. (3000°), high refractoriness under load (2500°), and are very resistant to basic slag and to temperature changes. The disadvantage of the last-mentioned seven materials (chromite—zirconia) is their high cost as compared with silica. Zirconia, however, may possibly find permanent application in the construction of high-frequency induction furnaces.

F. SALT.

PATENTS.

[Flame]-tinting of glass. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of D. A. DEWEY and C. D. SPENCER (E.P. 271,481, 18.5.27. U.S., 18.5.26).—Glass which, when used for incandescence lamps etc., reproduces the effect of an oil or gas flame is prepared from an iron-manganese amber batch. Thus, *e.g.*, to each 100 lb. of a standard soda-lead batch are added 5 lb. of nitre, 3 lb. of ferric oxide, and 4 lb. of manganese dioxide.

A. COUSEN.

Manufacture of stoneware highly resistant to perforation by the electric spark. F. SINGER (U.S.P. 1,642,754, 20.9.27. Appl., 18.12.26. Ger., 13.11.25).—Titanium compounds, *e.g.*, titanium dioxide, are added to the ingredients usually employed in the manufacture of ceramic insulating materials, and the mixture is fired, first in an oxidising and then in a reducing atmosphere.

T. S. WHEELER.

Unburned refractory [magnesite] brick. G. K. SCHLOTTERER and R. H. YOUNGMAN, Assrs. to HARBISON-WALKER REFRACTORIES CO. (U.S.P. 1,643,181, 20.9.27. Appl., 15.5.26; cf. U.S.P. 1,564,394; B., 1926, 192).—A mixture of dead-burned magnesite and powdered sodium silicate (5—10%) is treated with water, formed, and slowly dried at 55—95° in an atmosphere of progressively decreased humidity.

T. S. WHEELER.

Manufacture of glass. B. LONG, Assr. to SOC. ANON. DES MANUF. DES GLACES & PROD. CHIM. DE SAINT-GOBAIN, CHAUNY & CIREY (U.S.P. 1,633,534, 21.6.27. Appl., 12.1.26).—See E.P. 264,495; B., 1927, 523.

IX.—BUILDING MATERIALS.

Sintered alumina cements from Hungarian bauxite. J. VARGA (Z. angew. Chem., 1927, 40, 1164—1167).—The recently discovered Hungarian bauxite deposits are in two fields, that of Gánt containing 10—15% Fe₂O₃ and 5—12% (SiO₂ + TiO₂) and that of Halimba containing 23—31% Fe₂O₃ with 1.4—4.1% (SiO₂ + TiO₂). Both types on fusion with limestone gave a good sintered cement with m.p. as low as 1250°. A large graphite crucible heated by a blowpipe was used. The time of fusion was without influence on the result. These cements set in a few hours and suffered no loss in strength in a 12 months' test. The Halimba bauxite gave best results when the fusion was in a reducing atmosphere. If the temperature is raised a partial reduction of oxide to metallic iron takes place. The cement then produced has a higher m.p., greater tensile strength, and is slower setting. Less heat is required completely to fuse these aluminous cements than to sinter ordinary Portland cement.

Influence of aluminium and zinc on cement.

C. R. PLATZMANN (Chem.-Ztg., 1927, 51, 833—835).—Porous concrete was made by mixing cement and water with aluminium or zinc powder (U.S.P. 1,087,098) or with zinc powder and calcium chloride (G.P. 327,907; B., 1921, 180 A). The metal formed an aluminate or zincate of calcium with evolution of hydrogen which rendered the concrete porous. Mixtures of 100 pts. of Portland cement, 50 pts. of water, and 0.1—0.25 pt. of aluminium powder showed volume increases for different cements varying from 68 to 127%. The use of more than 0.25% of aluminium powder did not result in a larger increase of volume, as the excess of aluminium remained uncombined. The use of cement-sand mixtures gave smaller volume increases, but an increase of 100% was obtained with a mixture of cement with 75% of coke ash. The density of the concrete increased with the amount of sand used, and addition of more than 40% of sand resulted in a rapid diminution in the crushing strength. The use of zinc powder alone gave volume increases of only 30—50%. Addition of 2½% of zinc powder and 3¾% of calcium chloride to the cement and water yielded a concrete of high porosity and crushing strength.

F. R. ENNOS.

Use of fluorspar in the cement industry. H. BECKER (Zement, 1927, 16, 305—308; Chem. Zentr., 1927, I, 3219).—The liquid phase of clinker ceases at 1270—1300°, but with the addition of calcium fluoride the solidifying point becomes 900°. Rapid cooling is

necessary to prevent running of the clinker; sintering, therefore, is facilitated by the cautious addition of calcium fluoride.

W. G. CAREY.

Wood preservation. II. Arsenites of copper and zinc. III. Preservative properties of basic substances. L. P. CURTIN (Ind. Eng. Chem. 1927, 19, 993—999, 1159—1161; cf. B., 1927, 750).—II. Owing to the production of acid by wood-rotting fungi, salts as wood preservatives were tried which are of low solubility and which are not leached out by rain, but which are soluble in the acid solutions of p_H 5 or less produced by the fungi. Basic cuprous chloride made by reducing copper sulphate with sodium bisulphite in presence of a large amount of sodium chloride is precipitated satisfactorily in yellow pine as oxygen diffuses through the wood. By the substitution of acetic acid and arsenite ions, Paris green was precipitated progressively as the solution lost acetic acid, poles being successfully treated with this solution at 30°. Useful results were obtained from a solution containing copper sulphate 2.80%, sodium carbonate 0.75%, arsenious oxide 1.25%, sodium acetate 1.55%, sodium chloride 4.00%, and sodium bisulphite 0.70%, which gave a mixture of Scheele's green and Paris green in the wood. Zinc meta-arsenite precipitated from sulphuric acid (d 1.84), 0.20%, zinc sulphate 2.00%, calcium acetate 1.40%, soda ash 0.05%, and arsenious oxide 1.50% was satisfactory when chestnut and cedar poles were treated, and proved to be soluble in the acid produced from fungi. Toxicity tests show that 4 pts. of Scheele's green in 10,000 of nutrient jelly inhibits the growth of *Fomes annosus*, whilst the insoluble arsenites generally have a toxicity towards this fungus which is several times greater than that of zinc chloride. Boiling tests show that these arsenites are highly resistant to leaching effects.

III. Basic substances which do not contain poisonous ions were investigated as wood preservatives because the wood-rotting fungus *Fomes annosus* produced in 14 days acid equivalent to 113 mg. of acetic acid. Compared with the corresponding chlorides, sodium and barium carbonates are 16 times as toxic towards *Fomes annosus*, presumably because of their alkalinity, whilst the carbonates of strontium and calcium are, respectively, 3 and 5 times as toxic as the corresponding chlorides. The hydroxide and oxychloride of zinc are as toxic as zinc chloride although less soluble. Sodium carbonate lacks permanence in rain, but barium carbonate, although about half as toxic as zinc chloride, is worthy of test as a wood preservative because of its non-corrosive nature, low electrical conductivity, and permanence to weather.

W. G. CAREY.

Use of shale oil as a wood preservative. A. M. SOWDER (Ind. Eng. Chem., 1927, 19, 1180—1182).—In order to determine the value of shale oil as a wood preservative the toxicity of pyridine and quinoline towards cultures of *Fomes annosus* was investigated. The fact that pyridine is much less toxic than quinoline and has a low b.p. and high volatility renders it unimportant when judging the suitability of shale oil as a preservative. Quinoline has been shown by Weiss (J.S.C.I., 1911, 30, 1348) to compare favourably in

toxicity with cresol, phenol, and mercuric chloride; shale oil containing from 2.43—3.06% of quinoline should inhibit the growth of destructive fungi when injected into wood to the amount of 12 lb. of oil per cub. ft. of timber.

W. G. CAREY.

Wood preservation. SCHWALBE.—See II.

PATENTS.

Manufacture of hydraulic cements. L. FORSÉN (E.P. 272,163, 19.11.26. U.S., 3.6.26).—A mixture of Portland cement, burnt lime, and a granular material is ground until the Portland cement, under the action of the granular material, has reached such a high state of sub-division that it reacts quantitatively with water, and the product is separated from the coarser particles by blast separation. The lime, with water, converts into colloidal solution any finely-ground granular material and, if this contains silica, hardening calcium silicates are formed.

W. G. CAREY.

Manufacture of fused cement and gases containing phosphorus. W. KYBER (E.P. 256,622, 4.8.26. Ger., 4.8.25).—A mixture of a material having a high content of alumina, e.g., bauxite, and a phosphate is melted with sufficient carbonaceous material to reduce the phosphorus.

W. G. CAREY.

Production of fused cement. STUDIENGES. F. NUTZ-BARMACHUNG SCHWEIZERISCHER ERZLAGERSTÄTTEN (Swiss P. 117,718, 4.7.25).—Mixtures of blast-furnace slag with quicklime are heated in an electrical induction furnace. The setting time of the cement is reduced by rapidly cooling the product.

L. A. COLES.

Impregnation of wood. MONTAN, INC., Assees. of J. R. COOLIDGE (E.P. 265,206, 26.1.27. U.S., 26.1.26. Cf. U.S.P. 1,556,570; B., 1925, 960).—Wood, fibre-board, etc. before impregnation under pressure with a wax of high m.p., e.g., montan wax, is treated under pressure at a raised temperature with a material which readily penetrates the wood, is miscible with or dissolves the wax, and is toxic, e.g., creosote oil or β -naphthol, after which vacuum treatment is applied to withdraw a large part of the liquid forced into the wood.

W. G. CAREY.

Wood preservative. RÜTGERSWERKE-A.-G. (E.P. 266,296, 17.12.26. Ger., 20.2.26).—Sodium fluoride is added to the preservative consisting of an aqueous solution of sodium silicofluoride, to prevent iron with which the preservative comes in contact from being attacked.

H. ROYAL-DAWSON.

Manufacture of bricks and tiles. E. W. TILLBERG, Assr. to S. HELLSTRÖM (U.S.P. 1,647,063, 25.10.27. Appl., 17.2.27. Swed., 9.6.26).—Suitable clay is heated at 300—900°, the temperature being so adjusted that a portion of the clay still remains in a plastic condition. The product is then crushed, moistened with water, pressed into bricks and tiles, and fired without previous drying.

H. ROYAL-DAWSON.

Manufacture of bricks, blocks, etc. from materials typifiable by dolomite. F. L. DUFFIELD (E.P. 278,120, 26.7.26).—A mixture of a flux (e.g., ferric oxide, clay, or basic slag) with granular dolomite is sintered at 1200—1700° and run directly into cast-iron or

steel moulds in which it is pressed to the required shape and density, after which the product is allowed to cool.

W. G. CAREY.

Separation of solids from liquids [in sand or gravel washers]. J. SOUTHALL (E.P. 277,500, 23.9.26).

Heat-treating of materials (E.P. 269,480).—See I.

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

Relation between the slagging of iron and of manganese in the basic open-hearth process. E. FAUST (Arch. Eisenhüttenwesen, 1927, 1, 119—126; Stahl u. Eisen, 1927, 47, 1871—1872).—The relation between the iron and manganese in the metal and slag at the end of the treatment in the open-hearth furnace is given by the equation $[Mn]/[Fe] = K(Mn)/(Fe)$, square brackets indicating the composition of the metal and round brackets that of the slag. Numerous experiments with widely varying charges and using different manganiferous materials showed that for the basic-lined furnaces the value of K was 247 within the normal working range of temperature, and for acid-lined furnaces and in the Bessemer converter, 1350.

A. R. POWELL.

Nitridation of special steels. L. GUILLET (Compt. rend., 1927, 185, 818—821).—Steels of varying compositions were tempered, reheated, and nitrided for 90 hrs. in a current of ammonia. Physical tests then showed that the type of fragility known as "Krupp's disease" was absent from chromium-aluminium-molybdenum steels containing a little nickel, and was most and least marked for chromium-aluminium-nickel steels and chromium-aluminium steels, respectively. Ordinary chromium-aluminium-molybdenum-nickel steels showed the disease to a slight extent, but were otherwise satisfactory, and in no case was the hardness decreased by the heating at 500° for 90 hrs.

J. GRANT.

Rapid determination of manganese in steels.

A. QUARTAROLI (Annali Chim. Appl., 1927, 17, 375—378).—A small quantity of the metal is dissolved in nitric acid, most of the latter being then expelled and the liquid diluted with water so that the solution contains 1 g. of the steel per 5 litres. If the proportion of manganese is fairly high, this liquid is further diluted twentyfold, and 20 c.c. of it are treated at 20° with 4 drops of ammonia solution (d 0.95), 0.5 g. of powdered glass, and 2 c.c. of hydrogen peroxide solution capable of giving about 22 c.c. of oxygen at 20° when completely decomposed. The liquid is kept at 20°, and is shaken at intervals of 5 min., the volume of oxygen liberated being read. In absence of manganese the volumes of oxygen after 5, 10, 15, 20, and 25 min. are 0.1, 0.3, 0.5, 0.7, and 0.8 c.c.; if 12 pts. of manganese are present per 100 pts. of iron, the volumes are 0.6, 1.4, 2.3, 3.6 and 4.5. Values are also given for 11, 10, 9, and 8 pts. of manganese, the volumes for the last amount being 0.3, 0.9, 1.4, 1.8, and 2.4 c.c. If the steel is comparatively poor in manganese, 100 c.c. of the solution containing 1 g. of iron per 5 litres are heated rapidly to boiling with 0.25 g. of sodium acetate and filtered, the determination being carried out as above

on 20 c.c. of the filtrate. The presence of the acetate modifies the result, and a table is given showing the results for different percentages of manganese, but the latter have been accidentally omitted from the table.

T. H. POPE.

Rapid determination of the silicon content of ferrosilicon by means of the density. M. VON SCHWARZ (Chem.-Ztg., 1927, 51, 815).—A curve is given showing the relation between the density of ferrosilicon at room temperature and its content of silicon. The density is taken as the average of ten determinations on different portions of each sample, either in solid or in powder form, in order to obviate any slight irregularities in composition. In the absence of impurities or of pores in the material, the silicon content can be determined with an accuracy of 1%.

F. R. ENNOS.

Determination of sulphur in ores. K. K. JÄRVINEN (Z. anal. Chem., 1927, 72, 81—100).—The sources of error in sampling and dissolving the ore and in precipitating the sulphuric acid by means of barium chloride or benzidine hydrochloride are discussed in detail, and the following method is recommended for the determination of sulphur in pyrites and other sulphide minerals. The finely-powdered sample (0.5 g.) is warmed on the water-bath with 10—15 c.c. of a mixture of 2 vols. of nitric acid (d 1.4), 2 vols. of hydrochloric acid (d 1.19), and 7 vols. of water. After 15—30 min. the solution is cooled, and any black specks of impure sulphur are dissolved by shaking with 1 c.c. of bromine and 1—2 c.c. of ether; finally the excess of ether and bromine is expelled by gentle warming, and the solution evaporated to dryness. The residue is evaporated again with 5 c.c. of hydrochloric acid (d 1.1) and then digested with dilute hydrochloric acid until all soluble compounds are dissolved. After removing gangue matter by filtration the clear solution is diluted to 150 c.c., treated with 2 c.c. of 10% hydroxylamine hydrochloride solution, heated to boiling, and treated with 60 c.c. of cold 0.25*N*-barium chloride added drop by drop over a period of 5 min., followed by a further 10 c.c. added at once. The precipitate is collected immediately on a close paper, washed with hot water, ignited wet, and weighed; a correction factor of 3.3 mg. is added for every 1 g. of barium sulphate obtained. Equally good results are obtained by precipitation with 0.3*N*-benzidine hydrochloride solution from a volume of 300 c.c. after reducing the iron as above, evaporating to 100 c.c., cooling, and filtering. The precipitate is washed with cold water, transferred to the precipitating vessel, boiled with 150 c.c. of water, and titrated with sodium hydroxide (free from carbonate), using phenolphthalein as indicator.

A. R. POWELL.

Electrolytic deposits of cadmium for the protection of metals and alloys against corrosion. J. COURNOT and J. BARY (Compt. rend., 1927, 185, 773—774; cf. B., 1927, 489).—Cadmium deposits on copper are superior to those of nickel or zinc in that they are adherent, non-permeable, and resistant to the corrosive effect of a salt-water fog, an exposure of 1 hr. of which is equivalent to two weeks in the normal atmosphere. On the other hand they lack hardness, and as a result rapidly lose their fine finish. Double-deposition experi-

ments using cadmium-nickel, copper-nickel, and zinc-nickel combinations showed that the presence of cadmium and of zinc produced the most and least satisfactory results respectively, the cadmium in particular producing a fine finish and a close deposit. J. GRANT.

Corrosion of lead by mineral waters. P. KAJA (*Z. angew. Chem.*, 1927, **40**, 1167—1168).—The lead covering of a telephone cable was found to be completely corroded away over a short length, principally on the under side. The cable was encrusted with calcium carbonate and the lead converted into carbonate and oxide. A short distance away a mineral spring containing calcium carbonate and sodium chloride in large quantities was found. Although mineral waters may be conveyed through lead pipes without corrosion taking place, conditions are different when the water is in free contact with the oxygen of the soil. The chlorides present appear not to have taken any appreciable part in corrosion. C. IRWIN.

Determination of traces of certain impurities in lead. B. S. EVANS (*Analyst*, 1927, **52**, 565—572).—

Antimony. The method recommended is a modification of that used for determining antimony in copper (B., 1922, 144 A). The lead nitrate solution is precipitated with sulphuric acid, evaporated until acid fumes are evolved, taken up with hydrochloric acid, and sodium hypophosphite added, and, after boiling, benzene. The filtrate is boiled with cleaned copper foil, the copper washed, covered with cold water, and sodium peroxide added. After 5 min. the liquid is poured off and zinc sulphide added to it and the washings. This liquid now contains all the antimony. Sulphur dioxide is passed into the acidified filtrate and gum arabic added, and after making up to volume the colour is compared with a standard. *Arsenic* is determined by a similar process and is dissolved in a measured excess of 0.01*N*-iodine. *Tin.* If 1—2% is present the sample is dissolved in hydrochloric acid and bromine, tin reduced to the stannous state and dissolved, and the solution titrated with iodine, but for small quantities use is made of the very different solubilities of lead in hot and cold aqueous media. Dissolution is effected in a percolator (A., 1926, 707) carrying an upright condenser, and so arranged that the lead chloride is continuously removed. Concentrated hydrochloric acid, potassium iodide, and platinum foil are now added to the tin solution, granulated zinc is dropped in, and the solution boiled in a current of carbon dioxide and eventually titrated with iodine in absence of air. *Bismuth.* Lead chloride is precipitated and to the filtrate, reduced in bulk, sulphuric acid is added, lead sulphate removed, the filtrate evaporated, dissolved in water, and the bismuth determined in the cooled solution colorimetrically with potassium iodide. *Sulphur.* The sample is digested with nitric and hydrochloric acids, evaporated to dryness, hydrochloric acid and hot water are added, and the solution is boiled. Sulphates are precipitated with barium chloride in the usual way, the sulphur present being calculated from the weight of precipitate obtained. D. G. HEWER.

Thermal conductivity of light alloys. C. GRARD and J. VILLEY (*Compt. rend.*, 1927, **185**, 856—858).—Thermal, mechanical, and electrical data of a number of alloys of aluminium with copper or silicon are given.

Additions of copper to aluminium and magnesium gradually decrease and increase, respectively, their thermal and electrical conductivities till these become constant and almost equal for the two types of alloy. The ratios of the thermal and electrical conductivities confirm the Wiedemann-Franz law, and the former increase very slightly between 80° and 150°. Magnesium with 4% Cu (hammered) has thermal and mechanical properties superior to those of the alloy containing 12% Cu, and has a lower density.

J. GRANT.

Control of soldered joints by magnetic spectra.

A. ROUX (*Compt. rend.*, 1927, **185**, 859—861).—A perfect soldered joint gives the same magnetic spectrum (with continuous lines of force) as the steel of which its two portions are composed, whilst a faulty joint may be detected by means of the characteristic results obtained when the lines of force produced by the poles of a U-shaped electromagnet, placed symmetrically across the line of solder, are rendered visible by means of iron filings.

J. GRANT.

Electrolytic recovery of zinc from pyrites calcination residues rich or poor in copper.

H. PAWECK and H. WENZL (*Z. angew. Chem.*, 1927, **40**, 1106—1112).—In the Kellner process (Austr. P. 5484 of 1901) the residues are treated with sulphur dioxide, giving a solution of zinc, copper, and iron bisulphites, from which zinc sulphite is precipitated by boiling out of contact with air. The zinc sulphite is collected on a filter, and converted into sulphate by air oxidation, its solution being freed from iron by treatment with bleaching powder and zinc oxide, and is then further converted into zinc chloride with sodium chloride. The solution of zinc chloride is finally electrolysed. This process is modified by Paweck in that the zinc sulphite is converted into sulphate by the calculated quantity of warm 10% sulphuric acid, and the resulting solution is electrolysed in a Paweck diaphragm cell. These processes apply, however, only in the absence of appreciable quantities of copper, zinc sulphite being obtained free from copper only when the solution contains less than 0.3% Cu and more than 1.6% Zn. The yield of zinc sulphite increases with increasing concentration of bisulphite, and attains 75% with a concentration of 6.5% Zn. With residues rich in copper the precipitation of zinc sulphite is interfered with, and the copper must first be removed by heating the bisulphite solution to 75°, when the copper is precipitated as sulphide, the zinc remaining in solution. Traces of copper sulphate in the solution are removed by adding zinc, and the remaining sulphites of iron and zinc are oxidised by adding bleaching powder and blowing in air. Addition of zinc dust rich in oxide precipitates ferric hydroxide, and the clear zinc sulphate solution is electrolysed, with a lead anode and steel cathode. With residues rich in zinc and poor in copper the zinc sulphite is converted into sulphate by addition of the anolyte (containing 10—12% of sulphuric acid) from a previous electrolysis of zinc sulphate.

W. T. K. BRAUNHOLTZ.

PATENTS.

Manufacture of steel in open-hearth furnaces.

W. and H. MATHESIUS (E.P. 258,835, 28.6.26. Ger., 22.9.25).—Malleable scrap iron alone, or with the

addition of up to 20% of pig iron or its substitutes, is smelted and the temperature raised to 1900° or over; a material free from oxide is produced, and the usual boiling process can be omitted. H. ROYAL-DAWSON.

Production of [smelting charges of] coke and [iron] ore masses. R. TORMIN (E.P. 265,228, 28.1.27. Ger., 29.1.26).—Ground coal, coke, lime, etc. are intimately mixed with fine iron ore and degassed out of contact with air in separate retorts, closed on all sides, but not gas-tight. H. ROYAL-DAWSON.

Treatment of low-carbon steel and iron. F. KRUPP A.-G. (E.P. 274,016, 2.9.26. Ger., 7.7.26).—Low-carbon steel or iron is quenched from above 650° and afterwards drawn at temperatures up to 750°. Material so treated is more resistant to ageing influences and to the effects of liquids and vapours. C. A. KING.

Method and apparatus for refining steel and pig iron. J. MAXIMOFF, M. S. DE COSTA, and R. P. D. KREBS (E.P. 262,136, 27.11.26. Fr., 28.11.25).—Steel or pig iron is refined by means of centrifugal force in a heated rotary drum, the speed of rotation then being reduced and the collected impurities removed by gravity through a central orifice. The drum may be heated internally by gas or electricity and cooling may be effected by a current of air passing through tubes arranged within the drum and connected to annular distributing chambers. C. A. KING.

Casting of [steel] ingots. (Sir) C. A. PARSONS and H. M. DUNCAN (E.P. 278,032, 29.4.26).—Molten steel is cast in moulds having greater longitudinal than vertical dimensions. The bottom of the mould is a chill surface, and the sides are covered with refractory material of some thickness, the whole being heated to a high temperature before casting. Electrical heating units may be incorporated in the sides of the mould to assist in keeping the upper part of the ingot molten until the final stage of solidification. C. A. KING.

Method of annealing or heat-treating steel or other metals. F. W. GUIBERT (U.S.P. 1,644,828, 11.10.27. Appl., 24.10.25).—The metal is heated to the required temperature and then quenched, being maintained in a vacuum during the entire process.

M. E. NOTTAGE.

Production of sheets, bands, wires, etc. from ferrosilicon alloys for the improvement of their electrical properties. O. H. DÖHNER (E.P. 276,953, 22.4.27. Ger., 4.9.26).—For the production of an alloy having a low "watt loss," high-silicon iron is cold-worked within critical deformation limits and reheated to the recrystallisation temperature (600–900°) determined by the composition of the alloy. C. A. KING.

Rustless alloy steel. J. T. WHITELEY, Assr. to E. J. SPINKA (U.S.P. 1,647,491, 1.11.27. Appl., 19.7.26).—The alloy contains 8.75% Cr, 3% Ni, 1% Cu, 1.5% Si, 0.2% C, 0.75% Zr, 0.5% Ti, and the remainder iron.

F. G. CROSSE.

Erosion-resistant ferrous alloy. C. B. JACOBS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,647,096, 25.10.27. Appl., 11.1.23).—The alloy consists of 3–15% Mn, 1–5% Cr, at least 2% C, and the remainder iron.

F. G. CROSSE.

Preparation of low-carbon alloys. V. B. BROWNE (U.S.P. 1,645,126, 11.10.27. Appl., 9.1.23. Renewed 24.2.27).—A metal bath is heated by means of a carbon arc and alloying compounds are introduced, the absorption of carbon being prevented by means of an oxidising slag. M. E. NOTTAGE.

Treatment of machine parts made of austenitic manganese steel. BÖHLER GEBR. & Co., A.-G., Assees. of J. FUCHS (Austr. P. 106,326, 15.10.25).—Articles of manganese steel containing 0.5–1.4% C and 10–17% Mn are heated in a carbonising medium for several hours at 800–1100°, allowed to cool slowly, reheated to 900–1000°, and quenched in oil or water. The resistance to wear is considerably increased by this treatment. A. R. POWELL.

Mechanical ore-roasting furnace. MANUF. DE PROD. CHIM. DU NORD ETABL. KUHLMANN (Austr. P. 105,797, 7.7.24. Fr., 29.8.23).—A mechanical roasting furnace comprises a number of superimposed hearths alternately stationary and rotating, the latter being carried on rollers running on circular rails; both types are provided with water-cooled, rotating, metallic, toothed rables. A. R. POWELL.

Tunnel annealing furnace. R. SCHUBERT and C. PLETSCH (E.P. 277,541, 26.1.27).—To ensure uniform heating the flared orifices of the burners are provided only in the bed and side walls of the tunnel, and those in the same wall are so directed that the currents of hot gases intersect, thus minimising the tendency of the gases to rise. H. HOLMES.

Continuous roasting furnace for zinc or lead ores. L. TOCCO and M. LANDI (F.P. 619,660, 4.6.26).—A continuously operating, rotating, roasting furnace is provided with one or more separately revolving parts so as to allow of charging and discharging.

A. R. POWELL.

Alloys and their application to the manufacture of electrical conductors. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (E.P. 278,454, 10.7. and 24.9.26).—A quaternary alloy of high electrical resistance and high permeability contains 0–17% Fe, 1–5% Al, 1–5% of a fourth element, e.g., chromium, the remainder being nickel. A more particular composition shows 8–11% Fe, 2–3% Al, 2–3% Cr, the balance (not less than 84%) being nickel. C. A. KING.

Thermostatic metal. C. E. FRY, Assr. to H. A. WILSON Co. (U.S.P. 1,643,809, 27.9.27. Appl., 31.5.21).—A strip of Monel metal is fused to a similar strip of invar steel to give a couple, of which the shape alters with change of temperature. T. S. WHEELER.

Production of metallised surfaces on bodies containing sulphur. A. I. G. WARREN, and PRECIOUS METAL INDUSTRIES, LTD. (E.P. 278,437, 7.7.26. Addn. to E.P. 196,063; B., 1923, 565A).—Materials, e.g., tarry or bituminous substances, containing sulphur are placed in contact with metal or a metallic compound and heated until a film of metallic sulphide is formed. After removal of the excess metal the sulphide is reduced to metallic form. C. A. KING.

Coating of metals with aluminium or magnesium or aluminium or magnesium alloy. R. HOPFELT

and C. P. NOLDEN (E.P. 278,415, 1.7.26).—A coat of aluminium or magnesium or their alloys is applied to the surface of a metal in the presence of silica or siliceous material, and the whole is then heated at 800—1200°. The coating metal may be used in the form of powder mixed with silica, kaolin, and an organic binder to form a paste which may be brushed or sprayed on to the base metal.

C. A. KING.

Production of a weatherproof oxide layer on electron metal. W. PIEPER, Assr. to J. B. SOELLNER NACHF. REISSZUGFABR. A.-G. (U.S.P. 1,642,309, 13.9.27. Appl., 22.5.24. Ger., 4.7.23).—Electron metal (a manganese-zinc-aluminium alloy) is immersed in sodium hydroxide solution containing potassium nitrate for 20 min. at 110°.

T. S. WHEELER.

Improving the mechanical properties of aluminium containing magnesium. G. GIULINI (Austr. P. 106,021, 29.7.20).—Aluminium containing 0.5—1% Si and 0.2—1% Fe is alloyed with 0.1—0.5% Mg and the cast alloy is annealed for 1 hr. at 540—560°, quenched, and tempered at 160—170° for 10 hrs. The Brinell hardness of the alloy so treated is 90—100, and the tensile strength 3—3.5 tons/cm.² after rolling or drawing, compared with 35—45 and 1.4—1.8 tons/cm.² in the case of unalloyed aluminium.

A. R. POWELL.

Manufacture of a [non-tarnishing] silver-silicon alloy. M. G. CORSON, formerly KORSUNSKY, Assr. to ELECTRO METALLURGICAL CO. (U.S.P. 1,643,304, 27.9.27. Appl., 16.5.24).—The alloy comprises silver together with 1.5—6% Si and 1—50% Cd, Al, Zn, Sn, or Sb.

T. S. WHEELER.

Bearing metal. S. DEICHES (Austr. P. 106,211, 14.5.25).—An alloy for use as a bearing metal comprises 30—64% Pb, 10—15% Cu, and 30—40% Sb, with the addition of small quantities of cadmium, mercury, or arsenic.

A. R. POWELL.

Bearing metal. P. TUTZKY (Austr. P. 105,701, 15.5.25).—A bearing metal alloy contains 61.5% Pb, 13.5% Sb, 20.5% Sn, 2.5% Cu, 1% Ni, and 1% Mo.

A. R. POWELL.

Solder for aluminium and its alloys. STUTTGARTER VERSICHERUNGS-GES.M.B.H. (Swiss P. 118,501, 13.8.25. Ger., 15.6.25).—A solder for aluminium contains 30—70% Al together with tin, zinc, and small quantities of other metals. *E.g.*, it may contain 45 pts. of aluminium, 37 pts. of tin, 18 pts. of zinc, 2 pts. of copper, 1.5 pts. of silicon, 1 pt. of lithium, and 0.5 pt. of iron. Equal weights of copper and aluminium are first melted together, the iron and silicon added, followed by the remainder of the aluminium and the zinc, tin, and lithium; finally the molten alloy is deoxidised with ferric chloride, skimmed, and cast into sticks.

A. R. POWELL.

Uniting dissimilar metals. H. C. MOUGEY, Assr. to GEN. MOTORS RESEARCH CORP. (U.S.P. 1,644,741, 11.10.27. Appl., 2.1.23).—Articles of flexible copper and of a ferrous material may be united by placing between them a flexible brass sheet coated with zinc and heating it sufficiently to cause the brass and zinc to alloy.

M. E. NOTTAGE.

Flux for welding and soldering. H. THEURER (Austr. P. 105,680, 14.8.25).—A flux for use in brazing

iron and steel and in the autogenous welding of cast iron, copper, and brass comprises an alcoholic solution of ethyl borate containing naphtha and an emulsifying agent, such as sulphonated castor oil.

A. R. POWELL.

Alloy for pen points. K. K. LEDIG, Assr. to AMER. PLATINUM WORKS (U.S.P. 1,647,301, 1.11.27. Appl., 1.3.26).—The alloy contains 60% Mo, 10% W, 10% Ru, together with 20% of a nickel-copper alloy consisting of 70% Ni and 30% Cu.

F. G. CROSSE.

Recovery of aluminium from oily metallic mixtures etc. J. G. G. FROST, Assr. to NAT. SMELTING CO. (U.S.P. 1,646,239, 18.10.27. Appl., 17.3.26).—After subjecting the oily mixture of finely-divided aluminium and iron to the action of a magnetic separator, the organic matter is burned in an oxidising flame, and the metallic residue again separated.

H. ROYAL-DAWSON.

Manufacture of metal polishes. F. E. GENGÉ (E.P. 278,078, 29.6.26).—Pyridine is used as part of the cleansing agent, with one or more of the abrasives in general use.

H. ROYAL-DAWSON.

Pickling of metals. J. H. GRAVELL (E.P. 278,398, 29.4.26).—See U.S.P. 1,632,833; B., 1927, 785.

Open-hearth furnace. G. L. DANFORTH, JUN., Assr. to OPEN HEARTH COMBUSTION CO. (U.S.P. 1,647,213, 1.11.27. Appl., 21.1.21).—See E.P. 183,247; B., 1922, 764 A.

Roasting of metallic ores. H. S. MACKAY (U.S.P. 1,647,050, 25.10.27. Appl., 23.4.23. U.K., 19.5.22).—See E.P. 205,528; B., 1923, 1230 A.

[Conveyor for use in] metallurgical operations [employing briquettes]. NEW JERSEY ZINC CO., Assees. of F. G. BREYER and E. H. BUNCE (E.P. 268,302, 15.2.27. U.S., 25.3.26).

Method and apparatus for degreasing metal etc. J. SAVAGE (E.P. 278,061, 23.6.26).

Recovery of lead from ores (E.P. 278,093) — See VII.

XI.—ELECTROTECHNICS.

Mercurous sulphate electrode for testing storage batteries. S. MAKIO (Amer. Electrochem. Soc., April, 1928. Advance copy. 7 pp.).—From measurements at temperatures between 11° and 28° the single potential of the normal mercurous sulphate electrode, $\text{Hg}|\text{Hg}_2\text{SO}_4, N\text{-H}_2\text{SO}_4$, is found to be given by $\epsilon_h = 0.676 - 0.00026(t - 18)$ volt at t° . Measurements have been made of the single potentials of the positive and negative plates of a lead accumulator during charge and discharge.

H. J. T. ELLINGHAM.

Explosion phenomena in the coating of mirrors—a warning. W. MEYER (Chem.-Ztg., 1927, 51, 804).—Explosions occurring in the silvering of mirrors are attributed to the formation at the liquid margins of silver amide and silver nitride ("fulminating silver") through the use of too concentrated solutions of ammoniacal silver nitrate and potassium hydroxide. The avoidance of over-production, the exclusion as far as possible of all organic impurities by lightly covering the reaction vessels, and the bringing into solution, by gradual addition of ammonia, of the brownish-black

specks of silver oxide precipitated on addition of the potassium hydroxide are further precautions advocated.

R. BRIGHTMAN.

Light sources for testing dyes. HADFIELD.—See VI.

Refractories for electric furnaces. KOTHNY.—See VIII.

Electrolytic protection of metals. COURNOT and BARY. **Corrosion of lead on cables.** KAJA.—See X.

Electrical properties of rubber-sulphur compounds. CURTIS and others.—See XIV.

Electrometric determination of ash in sugar. SANDERA.—See XVII.

PATENTS.

Electrolyser diaphragms of the filter-press type. R. PECHKRANZ (E.P. 271,043, 16.3.27. Switz., 14.5.26).—The diaphragm is mounted in a frame of circular, elliptical, or polygonal contour with no internal angle of 90° or less. A single metal member cast in one piece or bent to shape and soldered at its ends may be used.

H. HOLMES.

Electric furnace. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of D. F. NEWMAN and C. STEENSTRUP (E.P. 265,950, 29.1.27. U.S., 15.2.26).—Means are provided for inserting or withdrawing a portion of the furnace charge through the same normally-closed opening without the remainder of the charge in the furnace being exposed to the atmosphere.

J. S. G. THOMAS.

Electric furnace. F. A. J. FITZGERALD, Assr. to HARPER ELECTRIC FURNACE CORP. (U.S.P. 1,646,058, 18.10.27. Appl., 26.5.25).—The lining of the furnace chamber is made of coalescing carborundum blocks.

J. S. G. THOMAS.

[Electric] furnace. A. N. OTIS, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,646,213, 18.10.27. Appl., 16.7.25).—An arm supported at one side of the furnace can swing over the furnace, whilst a second arm pivoted on the first and extending lengthwise thereof is connected with the furnace cover, which is lifted by a lever pivoted on the end of the first arm and acting on the second.

J. S. G. THOMAS.

Electric furnace. J. A. SEEDE, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,646,221, 18.10.27. Appl., 23.3.25).—A pair of adjustable electrodes extend in arcing relation into an enclosed space enclosed by refractory walls in the furnace chamber and surrounded by the charge.

J. S. G. THOMAS.

Plates for electric accumulators. G. FROMONT (E.P. 250,985, 20.4.26. Fr., 20.4.25).—Chemically pure salts of alkali, alkaline-earth, and earth metals, produced by the action of sulphuric acid on bicarbonates or carbonates of the corresponding metals, are incorporated in the formation electrolyte and in the paste for filling the plates.

J. S. G. THOMAS.

Discharge electrodes for electrical gas-purifying apparatus. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of R. HEINRICH (G.P. 442,644, 22.6.24).—The electron-emitting properties of the usual metals or alloys used for the construction of the electrodes, such as copper, bronze, brass, nickel, and tungsten, are improved by the addition of about 0.3–3.0% Th,

or of one or more of the alkaline-earth metals, magnesium, or the rare-earth metals.

L. A. COLES.

Treatment of hydrocarbons (F.P. 615,581).—See II.

Flame-tinted glass (E.P. 271,481). **Resistant stoneware** (U.S.P. 1,642,754).—See VIII.

Alloys for electrical conductors (E.P. 278,454).—See X.

White lead (E.P. 277,723).—See XIII.

XII.—FATS; OILS; WAXES.

Linoleic acid content of bone grease. I. H. STADLINGER and E. TSCHIRCH (Chem.-Ztg., 1927, 51, 667–669; cf. Kaufmann, B., 1926, 165, 758).—Free cyanogen displaces iodine from iodides, is displaced from thiocyanates by bromine, and behaves in addition and substitution reactions like a halogen element. New titration methods for oils and fats are based on these facts, the thiocyanogen being used in solution in an indifferent solvent. So-called “rhodanometric” determinations of the iodine values of fats and oils have been made and compared with Hanus iodine values. In compounds with one ethylenic linking the results were the same by either method; when two such linkings were present the “rhodanometric” iodine value was only half the theoretical, *e.g.*, linoleic acid, iodine value 181.09, gave a “rhodanometric” value 90.545. Details are given for the preparation of the reagent and its titration with 0.1*N*-thiosulphate solution. The authors find that the solution is not constant when compared with other volumetric solutions, and tabulated results of its titre confirm this. Polymerisation sets in after some days, and it is not recommended to use a solution of the technical reagent after 8 days. The determination of linoleic acid in fats by Kaufmann’s rhodanometric method should be followed by determination of the Hanus iodine value.

H. M. LANGTON.

Linoleic acid content of bone grease. II, III. H. STADLINGER and E. TSCHIRCH (Chem.-Ztg., 1927, 51, 686–688, 706–708).—II. In the thiocyanate determination of the iodine value an 80% excess of thiocyanate and 16–24 hrs. treatment was found to be sufficient. The proportions of oleic, linoleic, and saturated fatty acids are calculated from the Hanus iodine value and the thiocyanate iodine value. The highest linoleic acid content of a trade sample of the acid, determined by this method, was 82.1%. A number of samples of bone fat extracted from fresh or cooked beef bones averaged 5.2% of linoleic acid. A series of mixtures of the two were prepared. This could not be done by melting them together as a precipitate appeared, and analysis of such mixtures gave discordant results. With cold-mixing, however, the determined linoleic acid contents were within 1% of the quantity taken and the accuracy of the method was confirmed.

III. A large number of trade samples of bone grease were examined for Hanus iodine value and thiocyanate iodine value, and the linoleic acid content was deduced. Certain discrepancies led to the conclusion that a preliminary acid treatment was essential to the securing of reliable results. Taking only acid-treated samples, the mean figure for “refined bone grease” was 8.9% of linoleic acid and for “crude bone grease” 7.3%.

This compares with 5.2% of linoleic acid in laboratory-prepared bone grease, and the cause of the difference requires further research. C. IRWIN.

Changes in the characteristic indices of fats during the production of rancidity. K. TÄUFEL and J. CEREZO (Anal. Fis. Quím., 1927, 25, 349—362).—The development of rancidity in fats is of two types, viz., the ketonic or aromatic type as in the case of coconut oil, and the oleic acid type, as in the case of unsaturated glycerides. Data obtained for a twenty years' old sample of colza oil, by the usual methods for characterising fats, are given and are discussed from the point of view of the authors' theories.

G. W. ROBINSON.

Determination of the purity of olive oils. C. MILANI (Annali Chim. Appl., 1927, 17, 389—390).—When 5—6 c.c. of olive oil are shaken in a test-tube with 1 c.c. of a 1% solution of eosin in acetone, a pale pink coloration develops which disappears either almost immediately or when the tube is heated in a water-bath. Under similar treatment, a seed-oil, such as sesame, cottonseed, almond, castor, etc., gives at once a red or sulphur-red coloration, which tends to become deeper, even when the tube is heated. Olive oil adulterated with a seed-oil also gives the permanent red or pink coloration. This test is disturbed by the presence of water or other extraneous substance. T. H. POPE.

Fruits of Siberian cedar and the cedar oil. W. RUTSCHKIN (J. Oil Fat Ind., Russ., 1926, No. 4—5, 13—16; Chem. Zentr., 1927, I, 2783).—The seeds of the Siberian cedar (*Pinus cembra* L.) contain about 60% of a light yellow drying oil (d 0.93, n 1.485, saponif. value about 192, iodine value (Hübl) 130—160, Reichert-Meissl value 0.44—3.77) which is edible and can also be used for the preparation of a slow-drying varnish. E. H. SHARPLES.

PATENTS.

Extraction of oil from vegetable seeds etc. C. DOWNS and R. A. BELLWOOD (E.P. 278,145, 23.8.26).—Vegetable seeds etc., travelling in the form of a thin layer of meal along an endless band of porous material in an enclosed chamber, are alternately sprayed with a suitable extracting solvent and subjected to the action of an evacuating device whereby oil and solvent are withdrawn into sumps situated beneath the conveyor band. S. S. WOOLF.

Extraction of oil from the blubber of marine animals. A./S. FORSØKSDRIFT (E.P. 257,582, 3.8.26. Norw., 31.8.25).—Whale blubber etc. is finely ground and a substantial proportion of the water present is removed forthwith by heating *in vacuo*. The oil is then extracted from the tissue residuum by pressure.

S. S. WOOLF.

Treatment of wax. Paint remover containing wax. O. E. ENELL, Assr. to CHADELOID CHEMICAL CO. (U.S.P. 1,646,280—1, 18.10.27. Appl., [A, B] 24.12.24).—(A) A normally solid animal or vegetable wax is purified by treatment with hot trisodium phosphate solution. (B) A paint remover comprises suitable solvents and treated wax (*v.s.*). The wax is softer, has lower sp. gr.,

and tends less to granulation and separation from the solvents than untreated wax of like kind.

S. S. WOOLF.

Hydrogenation process (G.P. 441,164).—See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Constitution of artificial ultramarines. II. Ultramarine-blue with high silica content and silver, silver-sodium, and silver-selenium ultramarines. F. M. JAEGER and F. A. VAN MELLE (Proc. K. Akad. Wetensch. Amsterdam, 1927, 30, 479—498; cf. A., 1927, 715).—The variations in the X-ray powder spectrogram of ultramarine-blue Guimet No. 7553, as silver is substituted for sodium, giving finally 46.5% Ag, have been investigated. For comparison, several other silver compounds were prepared, *e.g.*, from ultramarine-pink Guimet OVR, giving 34.4% Ag; ultramarine-blue No. 13 of the Vereinigte Ultramarinfabriken, 34.2%; and ultramarine-green No. X from the same source, 51.0%. These silver ultramarines give practically identical spectrograms. The replacement of sodium by silver results in a diminution of the constant a_0 of the fundamental lattice from 9.06 Å. to 8.93 Å., but the original periodic structure is preserved. The relative intensities of corresponding interference images in the spectrograms of the sodium- and the silver-ultramarines, respectively, are, however, appreciably modified. The substitution of sulphur by selenium appears to leave the general structure unchanged. The general character of the relative intensities of corresponding diffraction images and their sequence also appear to remain the same, with a few exceptions. The film of the selenium derivative, however, is more clouded, probably due to the greater diffracting power of the selenium as compared with the sulphur atoms. The lattice constant $a_0 = 9.10$ Å. of the selenium derivative is only very slightly greater than that of the sulphur derivative. Similar behaviour is observed when selenium substitutes sulphur in silver-ultramarine. The results suggest that the sodium and silver atoms take a more important part in the formation of the "rigid" periodic structure of the ultramarines than the sulphur or selenium atoms, which may be statistically dispersed through the whole structure. Rotation spectrograms of the minerals nosean and hauyne have also been obtained, in order to confirm previous conclusions as to the fundamental grating and space-groups of these compounds. In addition, the mineral analcite has been examined, but, although closely related chemically to the other silicates studied, its structure appears to be quite different. M. S. BURR.

Testing of nitrocellulose lacquers. E. VON MÜHLENDAHL and H. SCHULZ (Z. angew. Chem., 1927, 40, 1185—1187).—In the Karussell test the samples are mounted on a vertical wheel which makes one revolution per 15 min. During a revolution they are immersed in water, then exposed in turn to radiant heat, giving a temperature of 60—70°, and to the rays of a mercury arc lamp. It is shown that one day in this test produces results closely parallel to one month of weathering. Another test consists in producing a film of lacquer of known thickness on a mirror, removing it by softening it with water, drying, and determining on a series of

days the breaking strain and breaking elasticity of the film. Increase of softening medium in the lacquer reduces the initial strength, but gives a more elastic film with a longer life. Another method of determining elasticity consists in bending sheets of known thickness and breadth through an angle of 180° around rods of different diameters. The rod which causes fracture gives a measure of the elasticity. These tests are all somewhat unsatisfactory, but taken in combination together with practical experience they give important indications of what improvements are desirable.

C. IRWIN.

Rôle of solvents and diluents in varnishes. B. SCHEIFELE (Farben-Ztg., 1927, 33, 207—209).—A dissertation on the structure of varnishes and lacquers and their films from the colloidal point of view, the significance of "solvate sheaths" as transition regions between pure disperse phase and pure dispersion medium being stressed. The original size and structure of the disperse phase and the solvent power of the solvent are factors influencing the formation of such sheaths and the resulting viscosity changes. Mechanical properties of films are not necessarily enhanced by complete interpenetration of disperse phase and dispersion medium. Even when drying oil and similar films are excluded, film formation cannot be regarded as the reverse of the dissolution process, effects due to vapour pressure, latent heat of evaporation, surface tension, and the formation of adsorbed layers being superimposed. S. S. WOOLF.

Damar Penak. R. W. BLAIR and F. E. BYRON (Malayan Forest Rec. No. 4, 1926, 3, 1—12).—The chemical and physical properties of 5 grades of Damar Penak (pale, yellow, amber, dust coarse, and dust fine) and 3 grades of "dead" (opaque) Damar, all obtained from *Balanocarpus Heimii*, are tabulated and compared with the results of earlier observers. The acid value of pale Damar Penak increases with age, light or absence thereof having no effect on this change. Details of varnish trials and of methods of purification of the resin are given. S. S. WOOLF.

Cedar oil. RUTSCHKIN.—See XII.

PATENTS.

Manufacture of white lead by electrolysis. R. S. CARRERAS (E.P. 277,723, 17.5.26).—In the manufacture of white lead in a single-chamber tank by the electrolysis of, e.g., sodium chlorate solution saturated with carbon dioxide, exhausted electrolyte is run off and fresh regenerated electrolyte is supplied continuously, the degree of saturation and the amount of electrolyte being kept constant. S. S. WOOLF.

Manufacture of zinc oxide. NEW JERSEY ZINC Co., Assees. of J. A. SINGMASTER, F. G. BREYER, and E. H. BUNCE (E.P. 268,301, 12.2.27. U.S., 27.3.26).—A charge of zinciferous and carbonaceous material is heated under substantially non-oxidising conditions, and the metallic zinc so formed meets a blast of relatively cool oxidising gas. The stream of gases, containing in suspension fine zinc oxide particles at about 700° , passes to an appropriate collecting room, being maintained at a temperature not below 125° during the collection of zinc oxide and separation of exhaust gases. Deposition of sulphur compounds on the pigment is thereby inhibited. S. S. WOOLF.

Application and treatment of varnish. G. R. CLOVER, Assr. to COOPER HEWITT ELECTRIC Co. (U.S.P. 1,646,010, 18.10.27. Appl., 18.7.23).—Varnished articles conveyed through a tunnel chamber are exposed to heat and to light rich in ultra-violet rays. S. S. WOOLF.

Coating compositions. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of W. P. DAVEY (E.P. 257,243, 28.7.26. U.S., 24.8.25).—The tendency to scumming in "water japans" consisting of an aqueous emulsion of asphaltic material, oleaginous material, and rubber latex, is inhibited by the addition of lamp black, clay, or other loading material of suitable density. S. S. WOOLF.

Application of coatings of nitrocellulose lacquers. WOLFF & Co., and H. I. SCHULZ (E.P. 262,440, 1.12.26. Ger., 4.12.25).—Priming coats for cellulose lacquers are based on nitrocellulose of higher nitrogen content and lower ether-alcohol solubility than the nitrocellulose used in the subsequent lacquer coats (which may differ progressively among themselves in these respects). Guncotton nitrocellulose containing more than 12.6% N and incompletely soluble in ether-alcohol is preferred for use in primers. S. S. WOOLF.

Manufacture of condensation products of carbamide and formaldehyde. H. BARTHÉLEMY, Assr. to SOC. IND. DES MAT. PLASTIQUES (U.S.P. 1,645,848, 18.10.27. Appl., 23.11.25. Fr., 16.12.24).—An alkaline-earth carbonate is added to a mixture of carbamide and formaldehyde and the whole is boiled. S. S. WOOLF.

Manufacture of a synthetic resin [shellac substitute]. J. McINTOSH and E. Y. WOLFORD, Assrs. to DIAMOND STATE FIBRE Co. (U.S.P. 1,642,078—9, 13.9.27. Appl., [A] 12.8.20; [B] 20.2.25).—(A) Equal parts of a phenol and glycerol are heated, first under reflux with a small quantity of sulphuric acid for 6—8 hrs. and then with hexamethylenetetramine at 125° for 24 hrs. (B) A phenol and glycerol are condensed without addition of hexamethylenetetramine. T. S. WHEELER.

Production of purified rosin. W. B. LOGAN, Assr. to ACME PRODUCTS Co., INC. (U.S.P. 1,643,276, 20.9.27. Appl., 21.4.26).—Wood rosin is heated at 300° for 2 hrs. to render it more suitable as a substitute for gum rosin. T. S. WHEELER.

Nitrocellulose for lacquers (E.P. 252,382).—See V.

Treatment of lead chloride (E.P. 278,093).—See VII.

Paint remover containing wax (U.S.P. 1,646,281).—See XII.

Resin from rubber (U.S.P. 1,642,018).—See XIV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Density and electrical properties of the system, rubber-sulphur. I. Density of rubber-sulphur compounds. A. T. McPHERSON. II. Electrical properties of rubber-sulphur compounds. H. L. CURTIS, A. T. McPHERSON, and A. H. SCOTT (U.S. Bur. Stand., Sci. Paper No. 560, 1927, 22, 383—397, 398—418).—Vulcanisation of rubber with sulphur is accompanied by a distinct increase in density; a maximum increase is observed at 19% of combined sulphur, which corresponds with the formation of a compound $(C_5H_8)_2S$. The dielectric constant and the

power factor curves show minimum values at this composition, whilst the curves showing variation in resistivity and in coefficient of thermal expansion with percentage of combined sulphur also undergo a marked inflexion. The changes in density accompanying vulcanisation are not simple, one complicating factor being the transformation of sulphur into allotropic forms. As the proportion of combined sulphur in rubber is increased from 0 to 32% the dielectric constant increases to a maximum at 10.5%, decreases to 19%, and then increases slightly up to 32%. The power factor behaves similarly except that the maximum occurs at 13% of combined sulphur; the resistivity increases up to 26% and then decreases up to 32%, inflexions occurring in the curve at 10.5, 13.5, and 19%. These results indicate the possibility of three additional compounds, viz. $(C_5H_8)_4S$, $(C_5H_8)_3S$, and $(C_5H_8)_4S_3$. The electrical breakdown is affected to such an extent by circumstances of the test that no definite relations can be indicated with the proportion of sulphur. The effect of free sulphur on the electrical properties of rubber (vulcanised or not) is very small relative to the effect of combined sulphur.

D. F. TWISS.

PATENTS.

Production of vulcanised rubber and accelerators therefor. H. W. ELLEY and D. H. POWERS, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,643,205, 20.9.27. Appl., 5.3.25).—A *p*-nitrosoaryldialkylamine, e.g., *p*-nitrosodimethylaniline, is heated at about 100° for 1–2 hrs. with 0.5–2 mols. of a primary, secondary, or tertiary aliphatic or heterocyclic amine, e.g., *n*-butylamine or pyridine, with or without addition of a solvent, e.g., ethyl alcohol, to give a product of value as an accelerator.

T. S. WHEELER.

Vulcanisation of rubber articles and apparatus therefor. NEW LIVERPOOL RUBBER CO., LTD., and F. AMENDE (E.P. 278,064, 26.6.26).—In the vulcanisation of rubber articles, e.g., shoes of rubber or with rubber soles, in an atmosphere of compressed air or other gas, the gas is circulated inside the vulcanising chamber and is preheated before admission, thereby ensuring a more even temperature than is obtained with the use of internal heating coils only.

D. F. TWISS.

Method of reclaiming rubber and fabric from scrap. HERCULES POWDER CO., Asses. of L. T. SMITH (E.P. 274,797, 25.9.26. U.S., 20.7.26).—Scrap rubber is heated at from 100° to 180° with a solvent consisting mainly of dipentene. This solvent may be the fraction b.p. 160–200°, d_{40}^{15} 0.86, from the distillation of pine wood or may consist of chemically dehydrated pine oil. The resulting solution is filtered to remove fabric and fillers, and is then evaporated for the recovery of the rubber. By heating with caustic soda under pressure used solvent may be rendered fit for re-use.

D. F. TWISS.

Manufacture of a rubber conversion product [resin]. H. L. FISHER, Assr. to B. F. GOODRICH CO. (U.S.P. 1,642,018, 13.9.27. Appl., 28.12.26).—Rubber is heated with trichloroacetic acid at 100° for 2 days, with or without addition of a solvent, e.g., benzene, and a phenol.

T. S. WHEELER.

XV.—LEATHER; GLUE.

Bactericidal action [in tanning processes] of chromium salts and its general origin. S. HILPERT, L. PANETH, and E. SCHLUMBERGER (Z. angew. Chem., 1927, 40, 1086–1089).—Two factors must be considered separately, viz., the direct action of acidity on bacteria, and the influence of acidity on the action of chromium salts on protein. With *Staphylococci* the action of acidity persists up to p_H 3, and with *B. coli* up to p_H 2; beyond this the action of the chromium salt supervenes. With *Staphylococci* and a 0.01% solution of chrome alum there is a feeble bactericidal action up to p_H 4, due to acidity. At p_H 4.29 the reaction between the chromium salt and protein begins, and reaches a pronounced maximum at p_H 4.46. As neutrality is more nearly approached this action disappears again. *B. coli* gives a maximum at the same p_H value, but is more resistant to acidity. Commercial green chromium sulphate, which is a stronger tanning agent than chrome alum, exerts a more powerful bactericidal action at the optimum p_H value than the latter.

W. T. K. BRAUNHOLTZ.

PATENTS.

Treatment [tanning] of animal tissue. F. W. WEBER (U.S.P. 1,642,054, 13.9.27. Appl., 19.3.25. Renewed 31.1.27).—Prepared hides, after treatment with a solution of soap, e.g., sodium stearate, are immersed in a solution containing a rare-earth chloride, or a mixture of such compounds, cerium chloride excepted, sodium chloride, and, preferably, zinc sulphate. They are then treated with sodium phosphate solution.

T. S. WHEELER.

Treatment of hides and skins preliminary to tanning. R. BOTSON (U.S.P. 1,647,953, 1.11.27. Appl., 21.5.27. Belg., 2.6.26).—See E.P. 272,195; B., 1927, 758.

Synthetic tanning agents (G.P. 441,432).—See XX.

XVI.—AGRICULTURE.

Phenomena of absorption by soil colloids, studied by means of manganese salts. A. QUARTAROLI (Annali Chim. Appl., 1927, 17, 384–389).—When a small quantity (1 g.) of fine soil is left in contact, with occasional shaking, with 100 c.c. of a solution of a manganese salt containing 1 pt. of manganese per 50,000 of water at the ordinary temperature, appreciable proportions of the salt are absorbed. This absorption is not an ordinary precipitation in stoichiometric proportions. Subsequent treatment of the soil with successive quantities of water shows that part of the manganese salt is readily given up, whereas part is held tenaciously either as absorption compounds not easily decomposed by water or as true insoluble compounds.

T. H. POPE.

Influence of soil treatment on the assimilability of nutrients, in the seedling method of Neubauer. P. HAUSCHILD (Z. Pflanz. Düng., 1927, 10A, 37–53).—Drying increases the apparent amount of available nutrients in soils, as determined by the Neubauer method. The effect is greater as the temperature of drying increases. The possibility that changes in the proportion of calcium bicarbonate present in soils under

different conditions affect the solubility of phosphates is discussed. Drying may also assist in the decomposition of native phosphate.

A. G. POLLARD.

Decomposition of hydrogen peroxide by soils. K. SCHARER (Biochem. Z., 1927, 189, 125—149).—In the gas-volumetric determination of the power of a soil to decompose hydrogen peroxide, neutral peroxide must be used, as the catalytic power of the soil, although not a direct function of its p_H , is much greater in neutral or alkaline than in acid soils. The greater the manganese, iron, and total calcium content of the soil, the greater is its activity. Sandy soil has the least and loam and clay soil the greatest activity. Soils containing humus give, usually, higher values than mineral soils of similar composition, and high moor soils, on account of their acidic character, give smaller values than the more alkaline lower moor soils. The loss on ignition of soils involves loss of catalytic power only in so far as it reduces the carbonate content, and thereby the alkalinity of the soil. No relationship was detected between the hygroscopic nature of the soil and its activity. When a soil is sieved, the coarser grain shows less activity than the finer. Treatment of the soil with hydrochloric acid, aluminium chloride, mercuric chloride, or copper sulphate decreases, and with sodium hydroxide increases its activity. Treatment with potassium cyanide also increases its activity by virtue of its alkalinity. Boiling and heating the soil at 100° until it becomes constant in weight decreases its activity, whilst ignition results in considerable loss of activity except in moor soils rich in iron oxide. The activity is also decreased by irradiation with ultra-violet light. Manuring the soil only affects the catalytic power in so far as it alters its reaction. The soil containing the lowest number of bacteria possessed also the lowest catalytic activity, but there was no direct relationship between bacterial count and activity. Manganese compounds have the greatest catalytic power, and iron oxide comes next.

P. W. CLUTTERBUCK.

Movement of water in soils and subsoils. A. F. LEBEDEV (Z. Pflanz. Düng., 1927, 10A, 1—36).—Experimental work is described showing the mechanism of the transference of water from one soil horizon to another, and, in particular, the accumulation of subsoil water. The movement of soil water may result from the influence of gravitational and capillary forces or by the distillation of "hygroscopic" moisture brought about by changes in temperature and humidity at varying depths from the soil surface. Factors controlling these water movements, their limitations and effectiveness in soils and subsoils of different types are discussed in the light of existing theory and of practical results obtained.

A. G. POLLARD.

Neubauer method for determining the solubility of rock phosphates [in soils]. R. RAUSCHER (Forts. Landw., 1927, 2, 85; Bied. Zentr., 1927, 56, 395—397).—In experiments with Gafsa phosphate, the total intake of phosphorus by the seedlings increased with the amount of phosphate added, but the percentage utilisation of the added phosphate decreased. Fineness of grinding was of great importance in determining the solubility of rock phosphates, but the relative effect

of fineness on solubility was specific to each type of phosphate. The method does not offer a common basis of comparison for rock phosphates. The fertiliser value of these can only be ascertained by individual experiment.

A. G. POLLARD.

New method for the ready determination of manganese in soil. A. QUARTAROLI (Annali Chim. Appl., 1927, 17, 379—383).—Two grams of the soil are treated with boiling hydrochloric acid, most of which is then expelled, and the solution made up to 250 c.c. In 100 c.c. of this liquid the iron is determined approximately by precipitating with ammonia in presence of ammonium chloride, and washing the precipitate successively with boiling water, boiling 10% sodium hydroxide, and water. Part of the original 250 c.c. is then diluted to contain 1 g. of iron in 20 litres, 100 c.c. being used for the determination of the manganese by the method employed for steel (B., 1927, 910). If the proportion of manganese is very small, the hydrochloric acid solution of 2 g. of the soil is rendered almost neutral, and the iron, aluminium, and phosphoric acid are precipitated with sodium acetate. The liquid is made up to known volume and filtered, one half of the filtrate being concentrated to a small volume on a water-bath, treated with bromine water, and again concentrated to expel the bromine. The manganese hydroxide is collected on a small filter and dissolved in hydrochloric acid, the solution being evaporated to dryness and the residue made up in water to 100 c.c., or, for a very small amount of the precipitate, to 20 c.c. The catalytic determination of the manganese is then carried out on 20 c.c. of the solution. If only the readily assimilable manganese is required, 2 g. of the soil are treated with 2 c.c. of acetic acid on a water-bath until effervescence ceases, the liquid being filtered, the filter washed, and the filtrate evaporated to dryness on a water-bath. With a calcareous soil, the residue is moistened several times with hydrochloric acid and evaporated to dryness to expel most of the acetic acid. Finally the residue is made up to 250 c.c. with water, and 20 c.c. are used for determining the manganese catalytically. A table is given which shows the volumes of oxygen evolved for solutions containing from 1 g. of manganese in 125,000 to 1 g. in 4,000,000 c.c.

T. H. POPE.

Colorimetric determination of potassium in aqueous extracts of soils as an indicator of lack of manuring. A. NĚMEC (Biochem. Z., 1927, 189, 50—56).—A colorimetric method for the determination of potassium, depending on the brown colour obtained with potassium chloroplatinate and zinc chloride in hydrochloric acid solution, is described and used to demonstrate that an approximately direct relationship exists between the potassium content of aqueous extracts of a large number of soils of different sources and the amount of potassium in these soils assimilable by plants. The method can be used to determine the potassium supply of a soil and to separate soils rich from those poor in assimilable potassium.

P. W. CLUTTERBUCK.

Napier grass. W. G. FRIEDEMANN (Georgia Agric. Exp. Sta. Press Bull., 1927, No. 257).—Values for immature and mature Napier grass (*Pennisetum purpureum*),

respectively, are: moisture 90.16, 85.83; ash 1.54, 1.67; crude protein 2.02, 1.30; crude fibre 2.96, 5.41; crude fat 0.14, 0.29; nitrogen-free extract 3.18, 5.50%.

CHEMICAL ABSTRACTS.

Portable calomel electrode for the determination of p_H values in the field. P. KAMERMAN (J.S. African Chem. Inst., 1927, 10, 32–35).—In the portable calomel electrode described, special devices to prevent the contact of the calomel with the platinum are detailed. Trials showed that the instrument was reliable for the determination of the p_H value of soils, and gave results within 0.1 of those obtained by using a standard instrument.

R. A. PRATT.

Utilisation of sulphite-cellulose waste liquor for manurial purposes. J. GÖRNING (Papier-Fabr., 1927, 25, 573–575, 633–638, 653–658, 671–673; cf. Bokorny, B., 1919, 218; 1920, 167A).—The possibility of making use of sulphite-cellulose waste liquor as a fertiliser has been investigated. The preliminary difficulty of preparing a product which could be used under agricultural conditions was finally overcome by mixing the partly concentrated liquor with ground quicklime and peat dust. *E.g.*, 40 kg. of quicklime and 11 kg. of peat dust fixed 220 kg. of waste liquor (d 1.21) and yielded a readily-distributable powder containing about 23% of organic matter. The detailed results of a large number of experiments with this material on a great variety of crops and soils, both in pots and in the field, are given. In almost all the experiments the cellulose manure was either without effect or actually reduced the yield and caused injury to the plants. The favourable results previously obtained by Bokorny could not be confirmed.

C. T. GIMINGHAM.

Application of Ridsdale's modification of Pemberton's method for the volumetric determination of phosphoric anhydride to fertilisers. A. M. CAMERON and W. T. DOW (Analyst, 1927, 52, 576–580).—After destruction of any appreciable amounts of organic matter a weighed quantity of the sample is boiled with 100–150 c.c. of water and 20–30 c.c. of concentrated nitric acid, and the liquid decanted. The residue is heated with 8 c.c. of nitric acid and 5 c.c. of concentrated hydrochloric acid until evolution of oxides of nitrogen and chlorine ceases, the diluted solution is added to the decanted liquid, and the whole filtered. Sodium hydroxide is added to 25 c.c. of filtrate till a permanent precipitate is just obtained, which is redissolved by adding, drop by drop, nitric acid (d 1.2), followed by a further 4 c.c. of acid, a few drops of 5% permanganate solution, and, to the warm solution, one No. 7 Analoid tablet (ammonium nitrate 1.75 g., ammonium chloride 1.6 g., ammonium oxalate 0.25 g.). The resulting solution is diluted to 60 c.c. and boiled, a mixture of 25 c.c. of standard molybdate solution and 20 c.c. of water is added, and the whole shaken. After 10 min. the liquid is decanted, the precipitate washed with 0.1% potassium nitrate solution ("Laval") into a Nessler tube of about 65 c.c., which is placed in a Leffmann-Beam centrifuge and whirled for 1 min. The supernatant liquid is decanted through a cotton-wool plug, the Nessler tube nearly filled with wash liquor, closed with a stopper, and shaken. The tube is then filled up with "Laval,"

again centrifuged, and the process repeated until the precipitate is free from acid. To the precipitate, cotton-wool plug, and washings is added an excess of 0.5*N*-sodium hydroxide, and when dissolution of the yellow precipitate is complete the excess alkali is titrated with 0.5*N*-nitric acid against phenolphthalein. Water-soluble phosphate is determined on 25 c.c. (10–15 c.c. if superphosphate) of the aqueous solution obtained by the official method by just producing a precipitate with the sodium hydroxide solution and proceeding as above, and citric-soluble phosphates by adding 6 c.c. of nitric acid (d 1.2) to 25 c.c. of solution, boiling for 5 min., and completing oxidation by adding three No. 4 Analoids (potassium permanganate 0.25 g.) followed by a No. 7 Analoid, and, after diluting to 60 c.c., completing as before. Results agreed well with those obtained by the official methods.

D. G. HEWER.

PATENTS.

Artificial drying of crops. B. J. OWEN (E.P. 278,474, 29.7.26).—The material to be dried is exposed to a stream of hot combustion gases and air, the former being derived preferably from liquid fuel. The combustion chamber opens into a supply duct in communication with the atmosphere at one end, and having a suction-fan at the other, and is so constructed that combustion of the fuel is complete before admixture of the gases with air drawn in by the fan. The temperature of the mixed gases is controlled by the quantity of air admitted.

C. T. GIMINGHAM.

Seed-pickling materials. I. KREIDL (Austr. P. 105,704, 18.2.25).—The materials consist of slightly soluble basic copper salts used in the dry state. *E.g.*, the seed is thoroughly mixed with finely ground basic copper chloride or sulphate so as to give an even coating on the grains.

C. T. GIMINGHAM.

[Plant] stimulant and fertiliser. I. G. FARBERIND. A.-G., Assecs. of G. PFÜTZER (G.P. 443,130, 5.2.25).—Iron formate is claimed for use alone or with a fertiliser (*e.g.*, sodium nitrate) to increase crop yields.

C. T. GIMINGHAM.

Manufacture of a fungicide containing copper. J. D. JENKINS and E. F. BERGER, Assrs. to PITTSBURGH PLATE GLASS Co. (U.S.P. 1,642,370, 13.9.27. Appl., 17.11.25).—Precipitated copper hydroxide mixed with gum arabic and maize syrup is heated at 100° to give a product which forms in water a colloidal solution containing metallic copper and cuprous oxide.

T. S. WHEELER.

Copper-arsenic dust [fungicide]. G. E. SANDERS, Assr. to RICHES, PIVER & Co. (U.S.P. 1,642,511, 13.9.27. Appl., 9.8.24).—See U.S.P. 1,541,753; B., 1925, 689.

XVII.—SUGARS; STARCHES; GUMS.

Precipitation of sugar solutions with lime. **Steffen process for manufacture of sugar from molasses.** H. I. WATERMAN and J. S. A. J. M. VAN AKEN (J.S.C.I., 1927, 46, 411–413 T).—Experiments involving the precipitation of sucrose solutions with increasing quantities of lime show that the phenomena observed are of a colloid-chemical character, and correspond with the view that colloidal solubility in presence of a peptiser depends

on the quantity of colloid undissolved (cf. Ostwald; also von Buzágh, A., 1927, 310), and the existence of definite compounds between sucrose and lime, *e.g.*, the so-called calcium trisaccharate, is open to doubt. A sugar solution after treatment with lime was filtered at 0°, and the filtrate at room temperature gave a precipitate of a gel character, transparent under the microscope, when air was excluded. For each mol. of sugar only 1.8 mols. of calcium oxide were removed from the solution (cf. Dedek and Tchechov, B., 1926, 927; Aten, van Ginneken, and Engelhard, B., 1927, 23).

W. J. POWELL.

Deterioration of cane juices and its prevention.

J. H. HALDANE (Int. Sugar J., 1927, 29, 367—370).—Mixed mill juice, treated with 2, 4, 6, 8, and 10 c.c. per litre of a solution of "E.C." (electrolytic chlorine) containing 2% of available chlorine, showed after 6 hrs. a decrease of purity of 1.08, 0.57, 0.20, 0.14, and 0, respectively, whereas the untreated sample indicated one of 3.49. After a "wash-down," and periodically during milling operations, the mill beds, mill cheeks, gutters, intermediate carriers, etc. should be sprayed with "E.C." containing 2% of available chlorine. Further, a continuous trickle of the antiseptic, diluted 1:500 at the beginning of the season, and increased to 1:200 towards the end of it, should be maintained in all the juice gutters as a preventative against deterioration.

J. P. OGILVIE.

Amino-acids and related compounds in sugar products. J. A. AMBLER (Int. Sugar J., 1927, 29, 382—385, 437—441).—Riffart's method (cf. B., 1922, 841 A) of determining amino-acids colorimetrically by means of ninhydrin (triketohydrindene hydrate) has been applied to follow the elimination of amino-acids in the refining of raw sugar, char being found to remove 70—80% of them. Raw sugar contains 0.025% N as amino-acids in a concentrated condition in the surface films of the crystal, whence they are readily removed by washing.

J. P. OGILVIE.

Decoloration of sugar juice by sulphuring before evaporation. L. CHALOUPKA (Z. Zuckerind. Czechoslov., 1927, 51, 543—548, 554—558).—After passing through a triple-effect pressure evaporator with concentrator, the juice was found to be more coloured than when working under vacuum, but by sulphuring the juice after the third carbonatation, previous to evaporation, so as to reduce its alkalinity from 0.03 to 0.02% CaO, and then again carbonating to 0.01%, a much better decolorising effect was obtained than in the absence of this treatment.

J. P. OGILVIE.

After-darkening of the juices [in the carbonatation process in beet factories]. R. BRADA (Z. Zuckerind. Czechoslov., 1927, 51, 548—550).—Determinations of the colour of the juice throughout clarification in a beet factory show that after the second carbonatation it is about 20% higher than at the end of the first.

J. P. OGILVIE.

Electrical determination of the ash content of sugar factory products. K. ŠANDERA (Z. Zuckerind. Czechoslov., 1927, 51, 603—612).—A portable form of the author's apparatus (B., 1927, 312) has been designed for use wherever A.C. of usual voltage is available,

by means of which the indication is obtained photo-metrically and the ash content read off directly, the temperature being compensated by the introduction of a resistance. A truer idea of the salt content of sugar solutions is obtained electrically than by the incineration method, since the latter determines only inorganic matter, but the former both inorganic and organic anions.

J. P. OGILVIE.

Sachs-Le Docte versus Krüger method of determining sugar in the beet. A. LE DOCTE (Int. Sugar J., 1927, 29, 387).—Krüger's method, in which 78 c.c. of liquid are used per 26 g. of rasped pulp, gives a result that is 0.16% lower than the truth, the sugar content of the roots being 16%. Compared with the Sachs-Le Docte procedure, it is much more liable to give rise to various errors in routine work.

J. P. OGILVIE.

Determination of dry substance in after-product syrups and molasses. J. MIKOLÁŠEK (Z. Zuckerind. Czechoslov., 1927, 51, 9—12).—Determinations carried out during the past four seasons show that results obtained refractometrically and by desiccation (on sand) approximately agreed, those found by the former being slightly the higher. On the other hand, determinations made by the pycnometer were irregular compared with the results obtained by the other two methods. It is considered that the refractometer gives results closest to the truth.

J. P. OGILVIE.

Error in polarisation [of beet sugar solutions] produced by evaporation. C. R. KARGL (Z. Zuckerind. Czechoslov., 1927, 51, 519—523).—Normal weight solutions of raw beet sugars, clarified with 1.5 c.c. of basic lead acetate, were filtered, and the loss of weight due to evaporation was ascertained by weighing the whole apparatus. When using an ordinary stemless funnel uncovered there was an average loss of 0.05 g. in 10 min., corresponding to an increase in the polarisation of about 0.05° V., but when covered the loss was about 0.004 g.—a negligible amount. A covered filtering device, permitting more rapid filtration than the stemless funnel, whilst obviating the error due to evaporation, is described.

J. P. OGILVIE.

Adsorption [from sugar juice] in a layer of active carbon. J. DĚDEK and K. L. KÁČL (Z. Zuckerind. Czechoslov., 1927, 51, 523—532).—Animal charcoal was found to adsorb practically no nitrogenous matter, contrary to certain of the decolorising carbons (here designated as C, N, and SN) tested. Adsorption of calcium oxide and mineral constituents forms generally no point of difference between animal charcoal and active carbon, which latter retains relatively large amounts of ash.

J. P. OGILVIE.

PATENTS.

Purification of [sugar] liquors and manufacture of sugar. E. E. BATTELLE (U.S.P. 1,646,079, 18.10.27. Appl., 22.7.24).—The impure sugar liquor is passed through a battery of char-containing filters arranged in series, and at intervals the filter which has been longest under liquor pressure is cut out and another one containing revived char is inserted next to the filter which has been the shortest time under liquor pressure.

F. R. ENNOS.

Manufacture of syrup. W. F. COLE (U.S.P. 1,647,082, 25.10.27. Appl., 1.5.25).—Lime is added to raw wash sugar dissolved in steam, then to every 100 lb. of syrup in the mixture is added $\frac{1}{2}$ oz. of salt, after which the solution is filtered through bone charcoal.

H. ROYAL-DAWSON.

Boiling and evaporation of sugar solutions. BERTEN & Co., G.M.B.H. (E.P. 276,563, 31.1.27. Ger., 12.11.26).—Refined sugar is dissolved with syrup and water in a boiler and then introduced into a collecting reservoir from which the solution is pumped in turn to a succession of boilers, which latter are brought successively into communication with a source of vacuum.

J. P. OGILVIE.

Removal of the fine grain contained in syrup and molasses. RAFFINERIE TIRLEMONTAISE SOC. ANON. (E.P. 276,527, 2.11.26. Ger., 20.9.26).—Syrup and molasses are freed from fine grain by employing the surface action of particles of materials, *e.g.*, kieselguhr, paper, sugar crystals, etc., exposed to high centrifugal force. These materials may be mixed before the centrifugal action with the syrup or molasses, or may be inserted into the centrifugal in which they form a layer through which the syrup or molasses must pass.

J. P. OGILVIE.

Cleaning of molasses for the manufacture of compressed yeast. R. KUSSEROW (U.S.P. 1,642,929, 20.9.27. Appl., 29.10.25; cf. G.P. 439,498; B., 1927, 666).—Crude molasses diluted with water is made alkaline with calcium hydroxide, and after addition of a trace of potassium permanganate is kept at 25° for 24 hrs. A culture of lactic acid bacteria active at higher temperatures is then added and the mixture is fermented at 60° for 48 hrs., calcium hydroxide being added at intervals. Finally the liquid is heated at 80°.

T. S. WHEELER.

Manufacture of a true maple flavouring product. J. W. SALE and J. B. WILSON, ASSRS. to U.S.A. (U.S.P. 1,642,789, 20.9.27. Appl., 1.4.27).—Maple sap, concentrated to *d* 1.24, is treated at 80° with barium hydroxide in quantity sufficient to precipitate all the sugar present. The liquid is filtered, and the filtrate is freed from barium with sulphuric acid, concentrated to one tenth of its original volume, and re-filtered after neutralisation with calcium carbonate. It is finally concentrated to *d* 1.3.

T. S. WHEELER.

Heat-treatment of spent bone-char and similar materials [used in sugar refining]. C. W. BURGHOUS (E.P. 255,079, 8.7.26).—Drying, heat-treatment, and cooling are conducted on the material while it is passing vertically down as a continuous thin layer through an annular space in which it first receives a drying heat from an impervious member forming the inner wall of the space. Its moisture is given off freely through passages formed in the outer wall into a chamber forming the second wall, through which air is preferably circulated. It is then subjected to heat treatment by receiving more intense heat from the impervious member, whereupon all gaseous products evolved pass similarly into a second chamber surrounding the outer wall, through which escape products of combustion of fuel

or other inert gases. Finally it passes between the impervious member and a cooling jacket to an outlet.

J. P. OGILVIE.

Purification of crude starch. K. BRATRING (G.P. 441,697, 16.1.24).—By treatment of starch-protein mixtures with dilute hypochlorous acid (1.6 pts. of available chlorine for 1 pt. of the starch), the protein matter is completely removed.

C. HOLLINS.

Extraction of juice from [sugar] cane stalks. W. H. MORGAN, SEN., ASSR. to W. H. MORGAN, JUN. (U.S.P. 1,646,762, 25.10.27. Appl., 7.12.25).—See E.P. 267,235; B., 1927, 538.

Glycerin from sugar (E.P. 278,086).—See XVIII.

Oxidation of sugars to osones (G.P. 440,389).—See XX.

XVIII.—FERMENTATION INDUSTRIES.

Malt extracts. Time of saccharification and diastatic power; phenomena hindering their determination. D. SCHENK (Chem.-Ztg., 1927, 51, 814–815).—The time of saccharification of a malt extract, which is the time in minutes required by 20 c.c. of a 2% solution of the malt extract to effect the complete hydrolysis of 50 c.c. of a 3% solution of arrowroot, is useful as a control in the determination of the diastatic power. Examination of the time of saccharification and diastatic power, expressed in Pollak units, of 50 malt extracts showed that a diminution in the former was accompanied by an increase in the latter throughout the series. In certain determinations the diastatic power was lower than was to be expected from the time of saccharification. This was shown to be due to the poisonous action on the diastase of traces of copper in the distilled water, the diastatic power being diminished in some instances to one half or even one third of its normal value, whilst the time of saccharification was hardly affected. F. R. ENNOS.

PATENTS.

Production of yeast. AKTIEBOLAGET BÄSTA (F.P. 614,037, 20.3.26. Swed., 28.3. and 1.10.25).—Sulphite waste-liquors are partially neutralised by ground limestone and fermented after removal of the precipitate.

C. RANKEN.

Drying of yeast. A. K. BALLS (U.S.P. 1,643,047, 20.9.27. Appl., 13.12.23. Renewed 16.8.27).—Compressed yeast is mixed with pure cellulose (10%) and dried at 20–40° for 6–8 hrs.

T. S. WHEELER.

Improving the flavour of yeast. E. B. BROWN, ASSR. to FLEISCHMANN Co. (U.S.P. 1,642,537, 13.9.27. Appl., 12.3.25).—Compressed yeast mixed with sugar (1 pt.) is left for 8 hrs., and the sugar is then removed by diluting the mixture with water and inducing fermentation.

T. S. WHEELER.

Production of glycerin from sugar by fermentation in an alkaline medium. K. and N. LÜDECKE (E.P. 278,086, 30.6.26).—When the fermentation of the sugar in an alkaline medium is entirely or partially finished, the yeast is separated and the volatile fermentation products are distilled off. The residual mash, which contains glycerin, is again fermented after the addition

of fresh quantities of sugar. The process is repeated as often as is required (cf. E.P. 138,328; B., 1921, 57 A).

C. RANKEN.

Distillation of alcohol. E. C. R. MARKS. From U.S. INDUSTRIAL ALCOHOL CO. (E.P. 278,211, 29.11.26).—In an apparatus which comprises a distilling column, an intermediate dephlegmator, and a rectifying column, high-strength alcohol, substantially free from impurities, is produced by the distillation of liquids containing alcohol and lower-boiling impurities. The liquid alcohol and alcohol vapour of the distillate are separately conveyed from the dephlegmator to the rectifying column by valved pipe-lines in such a way that any part or all of the distillate may be introduced into the rectifier with any desired ratio between the liquid and vapour so introduced.

C. RANKEN.

Unfermentable and bacteria-resisting alcoholic liquids. C. O. BERTIN (F.P. 617,945, 21.6.26).—Wine, beer, etc. are deprived of their content of mineral substances by means of a secondary micro-organism.

C. RANKEN.

Fermentation of molasses (U.S.P. 1,642,929).—See XVII.

XIX.—FOODS.

Decomposition of free and combined cystine, with special reference to certain effects produced by heating fish flesh. L. H. ALMY (J. Amer. Chem. Soc., 1927, 49, 2540—2545).—Little or no hydrogen sulphide can be detected in fresh fish flesh after heating in a sealed tube at 120° for 45 min., but relatively large amounts of hydrogen sulphide are produced if the fish flesh is somewhat stale. This effect is due to the ability of the fresh flesh to destroy hydrogen sulphide, apparently by oxidation. Added cystine, as well as that present in combined form in the fresh flesh, is destroyed by heating. The presence of considerable hydrogen sulphide in canned fish therefore probably indicates a poor condition of the material when canned.

F. G. WILLSON.

Detection of prohibited vegetable and coal-tar colours in foodstuffs. J. R. NICHOLLS (Analyst, 1927, 52, 585—589).—To an ammoniacal extract of the foodstuff 1 drop of Methyl Orange is added, the solution neutralised, then acidified to about 0.01—0.02N, extracted with methylated ether, and the ether extract treated with successive quantities of about 5—10 c.c. of approximately 0.01N-sodium hydroxide solution until no more colour is removed. An equal quantity of light petroleum is next added to the methylated ether and the mixture again extracted with sodium hydroxide. Absence of colour in alkaline layers indicates absence of prohibited dyes. The petroleum-methylated ether extract is orange-red if Aurantia is present; the methylated ether is yellow with picric acid, yellow-orange with Victoria and Manchester Yellows and gamboge, and red with Aurin. Addition to the solution of 1 c.c. each of alcohol, methylated ether, and 30% sodium hydroxide solution produces on shaking a pink soda layer with Aurin and yellow ether layer with the others. Alternatively, if to 1 c.c. of solution 2 c.c. of 30% sodium hydroxide solution are added, mixed, kept for 1 min., and 1 c.c. of methylated ether and 1 c.c. of

alcohol added with shaking, the ethereal solution is colourless and soda layer yellow with picric acid and pink with Aurin. By acidifying the original solution and extracting with light petroleum, a yellow ethereal solution is produced with gamboge, whilst on removing the petroleum and adding light petroleum which has been shaken with ammonia a yellow turbidity is obtained with Victoria and Manchester Yellows. Details are given of 22 supplementary and confirmatory tests.

D. G. HEWER.

Determination of benzoic acid in foods. G. W. MONIER-WILLIAMS (Analyst, 1927, 52, 572—575).—The method depends on the fact that when steam containing benzoic acid vapour is passed over moist metallic magnesium in a reflux apparatus, the benzoic acid combines with the magnesium to form a soluble benzoate extractable by washing with hot water. The success of the determination depends largely on the thoroughness of the extraction. The extract is made strongly alkaline with 40% caustic soda solution, cooled to 40—50°, oxidised with a saturated aqueous solution of potassium permanganate, excess of which is destroyed by sulphur dioxide or sodium sulphite, and the precipitated manganese dioxide dissolved in sulphuric acid. This solution, after saturation with salt, is extracted with a mixture of methylated ether and light petroleum, the solvent evaporated, and the benzoic acid sublimed. The reflux apparatus used is described. Results compared well with those obtained by steam distillation, but the method was found not so generally applicable as the latter.

D. G. HEWER.

Analytical study of roasted coffee berries. J. M. CLAVERA (Anal. Fis. Quím., 1927, 25, 369—373).—If the percentage of ash in a roasted coffee exceeds by more than 0.5% the percentage of ash in raw coffee berries, it may be concluded that more than 10% of sugar has been added in roasting.

G. W. ROBINSON.

PATENTS.

Removal of undesirable foreign flavours and odours from milk, cream, and other milk products. M. B. MACDONALD, ASSR. to UNIV. TENNESSEE (U.S.P. 1,644,842, 11.10.27. Appl., 23.12.26).—The milk or milk product in liquid form is mixed with a mineral oil, and the latter is afterwards removed from the treated liquid, taking with it foreign odours and flavours.

F. R. ENNOS.

Treatment of butter cream. G. A. GRAY and M. B. NEWBURGER (U.S.P. 1,643,301, 27.9.27. Appl., 16.7.23).—The cream is treated with sulphur dioxide (0.03% or the equivalent amount of sulphite) prior to shipment in order to prevent fermentation, the excess of the latter being oxidised after shipment by means of hydrogen peroxide or during the pasteurising or aerating processes. The cream is then fermented and churned into butter.

F. R. ENNOS.

Sterilisation of cream. B. H. WEBB, ASSR. to U.S.A. (U.S.P. 1,646,671, 25.10.27. Appl., 16.4.27).—Cream is heated to 80° (approx.), then rendered homogeneous at 2500—3000 lb. pressure, sealed in air-tight containers at about 118°, and sterilised.

H. ROYAL-DAWSON.

Preparation of water-soluble albumin. E. M. MEYER (E.P. 260,224, 22.9.26. U.S., 20.10.25).—A

finely-granular, acid-precipitated milk casein is washed with water completely to remove acid, and agitated for a few minutes with approximately ten times its weight of water containing 3% by weight of lime. After allowing the suspended material to settle, it is separated and the translucent solution evaporated at a temperature below 52°. The partly soluble flakes thus obtained are then rendered completely soluble in water by storing for some days with finely-powdered dibasic calcium phosphate, which is afterwards separated from the flakes by sifting through a fine mesh. F. R. ENNOS.

Manufacture of dry-powdered jelly base containing pectin and sugar. H. T. LEO (U.S.P. 1,646,157, 18.10.27. Appl., 14.3.22).—A jelly base is made by mixing powdered pectin and finely-divided sugar, the proportion of the latter being varied in accordance with the jellifying capacity of the pectin and also with its ability to effect the dissolution of the pectin in the liquid into which the composition is placed. F. R. ENNOS.

Supplemental poultry food material. W. H. LAPP, Assr. to CONCENTRATE PRODUCTS Co. (U.S.P. 1,645,703, 18.10.27. Appl., 28.12.25).—A supplemental poultry food comprising organic and inorganic materials, of which the latter contains calcium and phosphate in the proportion of 6–8 pts. of calcium to 1 pt. of P_2O_5 , less than 0.2% Mg, and no fluorine. F. R. ENNOS.

Production of preserves of vegetable matters of unlimited durability. A. FAITELOWITZ (U.S.P. 1,647,089, 25.10.27. Appl., 8.3.24. Ger., 29.7.21).—See E.P. 229,100; B., 1925, 296.

Freezing apparatus for use in the concentration of fruit juices. W. A. HEYMAN (E.P. 278,799, 13.7.26).

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Manufacture of benzaldehyde and benzoic acid on a large scale. R. FREUND (Chem.-Ztg., 1927, 51, 803–804).—A description of plant and process affording by the chlorination of toluene a daily production of 150–200 kg. of pure benzaldehyde and 50–100 kg. of sublimed benzoic acid. Sublimation is performed in an aluminium vessel at 160–180°. R. BRIGHTMAN.

Pharmaceutical incompatibility of phenol. D. MIGLIACCI and R. GARGIULO (Annali Chim. Appl., 1927, 17, 402–406).—When mixed with either naphthalene or β -naphthol, phenol immediately forms a liquid mass. The system phenol– β -naphthol exhibits a eutectic point at 26°, corresponding with 78% of phenol. Below this temperature solid solutions containing 0–40% of phenol and simple crystalline mixtures containing 40–100% of phenol are formed; at higher temperatures only pasty or liquid masses are obtained. Contrary to Yamamoto's statement (A., 1908, ii, 928), the system phenol–naphthalene has the eutectic point 29°, corresponding with 80% of phenol. At lower temperatures only crystalline mixtures are formed, although pasty masses may result owing to absorption of moisture by the phenol; at higher temperatures pasty or liquid masses are formed. Neither system forms molecular compounds. T. H. POPE.

Extraction of ergot. I. Liquid extract of ergot. W. H. LINNELL and D. G. RANDLE (Pharm. J., 1927, 119, 423–427).—The preparation of alkaline, acid, and neutral alcoholic extracts of ergot has been examined. An alcoholic menstruum is preferable to water, but both are inefficient as extractive media and the addition of either boric acid or calcium hydroxide does not increase their activity. Alcohol (50 or 60%) acidified with not less than 0.5% of tartaric acid is the most effective solvent, and this yields a solution which contains a maximum of about 0.1% of ergotoxine in a 1 in 2 extract. It is unnecessary to prepare extracts stronger than 1 in 2 since 0.1% represents nearly the limit of solubility of ergotoxine. The efficiency of the extraction on continuing the percolation with unacidified dilute alcohol is very small and the labour involved is not justified. Methods of extraction adopted in nine foreign pharmacopœias are reviewed, and it is shown that with the exception of the Dutch and United States extracts all are practically inactive owing to the choice of unsuitable solvents. Defatting should precede extraction and all extracts should be 1 in 2. Projected work is outlined. E. H. SHARPLES.

Butyrates of linalol and the linalyl acetate contents of lavender oil. A. KAUFMANN and F. KJELSBURG (Perf. Ess. Oil Rec., 1927, 18, 418–421).—Linalyl butyrate (97.86% of ester, d_{15}^{15} 0.8970, $[\alpha]_D^{20}$ –10.02°, n_D^{20} 1.4518) and isobutyrate (94.49% of ester, d_{15}^{15} 0.8926, $[\alpha]_D^{20}$ –11.89°, n_D^{20} 1.4487) are much more stable than is the acetate. The butyrate requires boiling for 4 hrs. and the isobutyrate for 7 hrs. with a small excess of 0.5N-alcoholic potassium hydroxide before saponification is complete. The rates of hydrolysis of some esters of linalol adjusted to an ester content of about 50% (comparable with that of a good lavender oil) by addition of linalol have been examined. The results show that, owing to the low rates of hydrolysis of the two butyrates, lavender oil cannot contain large amounts of these two esters. Determinations of the linalyl butyrate and isobutyrate contents of lavender oils from various sources by a modification of Schimmel's fractional hydrolysis method (Gildemeister and Hoffmann, III, p. 74) show that, whereas natural lavender oil may contain these two esters, they are only present in small amounts (below 7%) and may occur combined with alcohols other than linalol, e.g., geraniol. The effect of different distillation methods for the production of the oil on the linalyl acetate and butyrate content is discussed (cf. also Langlais and Goby, B., 1927, 92, 267). The presence of the methyl ether of umbelliferone in lavender oil (Pfau, *ibid.*, 571) is confirmed. E. H. SHARPLES.

Amino-acids in sugar products. AMBLER.—See XVII.

Determination of azides. COPEMAN.—See XXII.

PATENTS.

Preparation of glycols. F. H. UNTIEDT (U.S.P. 1,641,710, 6.9.27. Appl., 11.6.26).—An olefine oxide, e.g., propylene oxide, is treated with an equivalent quantity of water in presence of a trace of sulphuric acid. T. S. WHEELER.

Preparation of succinic anhydride. I. G. FARBENIND. A.-G., Assees. of F. WEBEL (G.P. 441,002, 18.12.24).—Maleic anhydride vapour is passed with hydrogen over a catalyst (nickel at 180°, copper at 300°) other than platinum; or the anhydride is reduced with hydrogen in ethyl acetate in presence of a nickel catalyst at 15°.

C. HOLLINS.

Preparation of β -hydroxybutyric acid. DR. A. WACKER GES. FÜR ELEKTROCHEM. IND., M.B.H., Assees. of G. BASEL and F. KAUFER (G.P. 441,003, 28.5.25).—Crotonic acid is heated under reflux with dilute mineral acid (sulphuric or hydrochloric acid, or aluminium sulphate), and the resulting β -hydroxybutyric acid is purified by vacuum distillation. The ethyl ester, b.p. 179–180°, and the *butyl* ester, b.p. 209–211°, are useful softening agents in varnishes.

C. HOLLINS.

Manufacture of higher aliphatic acids. I. G. FARBENIND. A.-G., Assees. of R. WIETZEL and O. KÖHLER (G.P. 441,272, 26.9.24).—The oily products from the catalytic reduction of carbon oxides or from the condensation of alcohols with or without carbon monoxide, preferably freed from lower alcohols, are heated with alkali or alkaline-earth hydroxides. From the fraction of b.p. above 140° a mixture of acids $C_6H_{12}O_2$ and $C_7H_{14}O_2$ is obtained. The fraction, b.p. 160–170°, gives almost exclusively α -methylisohexic acid, b.p. 205°. Acids $C_5H_{10}O_2$ to $C_8H_{16}O_2$ are formed from the fraction, b.p. above 120°, obtained by passing water-gas mixed with methyl and ethyl alcohols over a mixed catalyst at 450° and 200 atm.

C. HOLLINS.

Preparation of organic acids [synthetic tanning agents]. I. G. FARBENIND. A.-G., Assees. of K. DAIMLER (G.P. 441,432, 14.9.20).—Fossil vegetable materials, freed or not from bitumen, or bitumens of these in so far as they are not purely hydrocarbon, are oxidised with air or oxygen in presence of nitric acid. Brown coal residues, e.g., extracted with benzene, are heated at 90° in a covered vessel in a current of air, red fuming nitric acid being allowed to percolate through the mass. The waste gases are washed successively in 50% and 25% nitric acid and water, the wash-liquors being kept at about constant strength by dilution with the next weaker liquor. When samples show no further increase in solubility, the remaining nitrous gases are removed by a current of hot air. If desired, the product is freed from insoluble matter by dissolution in water. It consists of a mixture of organic acids (38% CO_2H) and not more nitrogen (2.5%) than the starting-material. There is also formed an acid mixture soluble in alkalis or alkali acetate. Ozokerite gives a water-insoluble, alkali-soluble, acid mixture, the sodium salt of which has soap-like properties. Humic acids may also be oxidised in like manner.

C. HOLLINS.

Manufacture of vanillin [and azobenzene]. R. H. BORS (U.S.P. 1,643,804—5, 27.9.27. Appl., [A] 25.1.26; [B] 25.5.26).—The product obtained by treating oil of cloves with potassium hydroxide (potassium isoeugenoxide) is dissolved in aniline and heated at 100° for 2 hrs. with nitrobenzene in presence of concentrated sodium hydroxide solution, to form vanillin and azobenzene.

T. S. WHEELER.

Di-*p*-xylylguanidine. W. SCOTT, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,642,180, 13.9.27. Appl., 27.11.22).—Di-*p*-xylylthiocarbamide is converted by treatment with lead monoxide and ammonia into di-*p*-xylylguanidine, m.p. 170°.

T. S. WHEELER.

Preparation of organic compounds by means of catalytic gas reactions. I. G. FARBENIND. A.-G., Assees. of A. MITTASCH, K. WINKLER, and M. PIER (G.P. 441,433, 27.9.23).—As catalysts, especially for reduction of carbon oxides, are used in addition to oxide of zinc or cadmium difficultly reducible oxygen compounds of at least two metals of the higher groups of the periodic system. Such mixed catalysts are very resistant to catalyst poisons, and when used in the reduction of carbon oxides yield alcohols almost exclusively. A mixture of 70% of hydrogen and 30% of carbon monoxide at 200 atm. passed at 400–420° over a catalyst containing zinc oxide (1–2 mols.) and chromium trioxide (1 mol.) gives nearly pure methyl alcohol. Addition of alkali hydroxides to the catalyst leads to formation of higher alcohols. Other combinations of catalysts are oxides of: zinc, chromium, and uranium or vanadium; cadmium, chromium, and tungsten; zinc, manganese, and vanadium; zinc, vanadium, and uranium; zinc, cadmium, chromium, and uranium. The metals copper, silver, lead, or thorium, or their oxides may be added, but iron compounds are to be avoided. Ethylene is reduced quantitatively to ethane when led with hydrogen at 380–400° over a mixture of zinc oxide (3 mols.), chromic oxide (2 mols.), and uranium oxide (1 mol.). The reduction of nitrobenzene to aniline, the oxidation of toluene to benzaldehyde and benzoic acid, and of methyl alcohol to formaldehyde, and the preparation of higher alcohols from alcohol, carbon monoxide, hydrogen, and nitrogen, are described.

C. HOLLINS.

Hydrogenation of organic compounds. WERSCHEN-WEISSENFELSER BRAUNKOHLEN A.-G., A. FÜRTH, and G. HILDENBRAND (G.P. 441,164, 18.1.24).—The substance to be hydrogenated is passed with steam over artificially activated carbon at 300–350°. Oleic acid thus gives 68% of stearic acid, and 32% of oleic acid is returned to the process with more steam. Benzene from brown coal is completely reduced to saturated compounds after three such hydrogenations.

C. HOLLINS.

Oxidation of sugars to osones. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), Assees. of A. KRAISY (G.P. 440,389, 19.9.24).—From the mixture obtained by oxidation of invert sugar or laevulose with hydrogen peroxide and ferrous sulphate (G.P. 439,115; B., 1927, 619), made strongly acid with 50% sulphuric acid, the iron is precipitated by addition of the calculated quantity of potassium ferricyanide and filtered through talcum. The filtrate after removal of sulphuric acid as calcium sulphate is evaporated. The products have an improved appearance and taste. The Turnbull's blue must be adsorbed on talcum or alumina, or on silver chloride precipitated in the solution by addition of sodium chloride and silver nitrate.

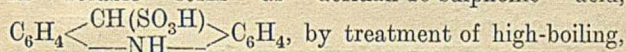
C. HOLLINS.

Manufacture of therapeutic agents. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 251,651, 30.4.26. Ger., 1.5.25).—

Hydrazoic acid derivatives, especially organic azides, are active hypotonics, but since their action is fugitive they are used in the form of their molecular compounds with other hypotonic and/or antispasmodic agents. *E.g.*, benzyl *p*-iodoethoxybenzoate (G.P. 412,699) and ψ -saccharin azide (benzazide-*o*-sulphonic acid) are dissolved together in acetone and the solvent is removed in a vacuum. C. HOLLINS.

Isolation of acridine and acridines with free meso-position from mixtures with other substances.

Preparation of 9-cyanoacridine. E. WIRTH (G.P. 440,771—2, 18.1.25).—(A) Acridine etc. is removed in soluble form as acridan-10-sulphonic acid,



by treatment of high-boiling, basic tar oils with warm sodium bisulphite solution, and may be regenerated by the action of alkali on the isolated sodium salt. From 0.25 to 0.3% of acridine is thus recovered from an anthracene oil, b.p. above 300°, *d* 1.10. (B) 10-Cyanoacridine, m.p. 186°, is obtained by treating acridan-10-sulphonic acid with alcoholic alkali cyanide. The intermediately formed 10-cyanoacridan (10-cyanodihydroacridine) is very quickly oxidised in air to 10-cyanoacridine, which finds application in the preparation of therapeutic compounds. C. HOLLINS.

Manufacture of organic compounds of arsenic.

R. W. E. STICKINGS, and MAY & BAKER, LTD. (E.P. 278,444, 8.7.26).—An arsinic acid of an *o*-aminophenol is treated with an α -halogenoacetyl halide (chloroacetyl chloride) in presence of caustic alkali, and the product is converted separately or in the same operation into a hydroxybenz-1:4-oxazinearsinic acid by hot alkali. From 3-amino-4-hydroxybenzene-1-arsinic acid and chloroacetyl chloride are obtained first the 3-chloroacetamido-derivative, then 3-hydroxybenz-1:4-oxazine-6-arsinic acid. The 8-acetamido-derivative of this is prepared from 5-amino-3-acetamido-4-hydroxybenzene-1-arsinic acid (obtained by nitration and reduction of 3-acetamido-4-hydroxybenzene-1-arsinic acid). C. HOLLINS.

Manufacture of protective and curative agents from glandular organs. G. SCHRÖDER (E.P. 263,155, 14.12.26. Ger., 16.12.25).—Agents for the cure of, and

protection against, infectious diseases are obtained from cell suspensions from glandular organs (spleen, thymus, etc.) by keeping them in a frozen state, then thawing and filtering. The therapeutic action of the preparations is increased by inoculating them with appropriate bacteria (tuberculosis culture etc.), which are killed by the action of the preparations, while specific vaccines are produced. B. FULLMAN.

Manufacture of a pharmaceutical product containing arsenic. W. SCHOELLER and M. GEHRKE,

ASSRS. to CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (U.S.P. 1,642,830, 20.9.27. Appl., 9.8.26. Ger., 2.7.24).—See E.P. 236,563; B., 1925, 783.

Alcohols from gases containing olefines (E.P. 249,834).—See II.

Carbazides and thiocarbazides of the naphthalene series (E.P. 278,037). **Heterocyclic compounds** (E.P. 258,553). **Chlorides of *o*-hydroxycarboxylic acids of polynuclear hydrocarbons** (E.P. 278,463). **Oxida-**

tion and reduction of organic compounds (G.P. 441,163 and 441,179).—See IV.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Preparation and properties of some synthetic photohalide emulsions. E. P. WIGHTMAN,

A. P. H. TRIVELLI, and S. E. SHEPPARD (J. Franklin Inst., 1927, 204, 491—499).—A method of preparing photohalide emulsions, somewhat different from the method of Lüppo-Cramer, is described using either colloidal silver or colloidal silver sulphide as sensitiser. To remove the excess colloidal silver in the case of the former emulsion, chromic acid is used, preferably added to the emulsion before coating. This treatment results in a considerable lowering of sensitivity, which can be restored to a large extent by bathing in a very dilute solution of ammonia containing a little potassium bromide. The emulsions show in general a sensitivity extending further into the long wave-length region than emulsions prepared without colloidal silver or silver sulphide, an observation which is in accordance with those of previous workers. The spectral sensitivity of the photohalide emulsions differs from that of those sensitised with allylthiocarbamide in being more sensitive in the red and less sensitive in the green region of the spectrum. The work supports the strain and ionic deformation theories of sensitivity, but it does not exclude the halogen acceptance theory of sensitising by silver sulphide. W. CLARK.

Development of silver halide-gelatin emulsions.

F. BÜRKI (Helv. Chim. Acta, 1927, 10, 782—784).—Although sodium hyposulphite may be used as a photographic developer and formaldehyde as an accelerator, the double compound "Rongalite" is inert. Fixers behave differently towards the "exposure nuclei" of the latent image. Exposed plates after fixing in sodium thiosulphate solution blacken on physical development, whereas exposed plates which have been treated with ammonium thiocyanate solutions or desensitisers do not. J. S. CARTER.

XXII.—EXPLOSIVES; MATCHES.

Replacement of nitroglycerin in ammonium nitrate explosives. H. KAST (Z. ges. Schiess- u. Sprengstoffw., 1927, 22, 208—213, 243—247, 279—283).—Ammonium nitrate explosives in Germany and elsewhere must contain, by law, not less than 5% of nitroglycerin in order to ensure detonation. For reasons of economy and safety in handling, it is desirable to investigate the possibility of obtaining the same effect

either by increasing the percentage of aromatic nitro-compounds, replacing nitroglycerin by nitrocellulose, or by replacing ammonium nitrate by other suppliers of oxygen such as potassium perchlorate. In the case of donarit, 8% of T.N.T. or 10% of potassium perchlorate could be present without undue sensitiveness, although initiation was less easy. When nitroglycerin is replaced by ammonium perchlorate the resulting mixtures are equal in sensitiveness to those containing potassium perchlorate. Substitution of wood meal for wheat flour yielded a less dense explosive. S. BINNING.

Glycol dinitrate as basis of gelatinous blasting explosives. A. SCHMID (Z. ges. Schiess- u. Sprengstoffw., 1927, 22, 273—276).—Attention is called to the extended use in America of glycol dinitrate as a constituent of gelatinous blasting explosives, which remain plastic when exposed to temperatures low enough to freeze the ordinary nitroglycerin explosive. Glycol is not more difficult to nitrate than glycerin and the glycol dinitrate separates quickly from the waste acid and wash-water. Owing to the toxicity of the volatile dinitrate, a continuous nitration process is recommended. Gelatinisation of nitrocotton is effected more quickly by glycol dinitrate than by nitroglycerin, so that a slow-gelatinising nitrocotton must be used or gelatinisation will be uneven. Gelatinisation should take place at 0—1.5°, when homogeneous, transparent "gelatins" will be obtained. Compared with "gelatin" obtained from nitroglycerin, those from glycol dinitrate are stickier and not so stiff, but an increase in nitrocotton and in the absorbent used subsequently removes this disadvantage. Decrease in weight of the glycol dinitrate explosive when cartridged is practically equal to that of a nitroglycerin explosive.

S. BINNING.

Determination of azides. D. A. COPEMAN (J.S. African Chem. Inst., 1927, 10, 18—22).—Existing methods are reviewed and two satisfactory methods described. In the first method, sodium azide is treated with sulphuric acid and the resulting hydrazoic acid is distilled over into a definite volume of standard caustic soda. The decrease in alkalinity is found by titration with standard acid, using phenolphthalein as indicator. In the second method a solution of ceric ammonium nitrate is allowed to act on the azide and the resulting nitrogen is collected in a nitrometer. Both methods yield results of great accuracy.

R. A. PRATT.

Analysis of nitroglycerin waste acid. A. S. WEBB (J.S. African Chem. Inst., 1927, 10, 13—14).—Sulphuric and nitrous acids are determined by titration methods, total nitrogen in the nitrometer, and the organic matter by oxidation and subsequent titration with potassium iodide and sodium thiosulphate. The method of calculating results is given.

R. A. PRATT.

XXIII.—SANITATION; WATER PURIFICATION.

Use of colloids for boiler-scale prevention. E. SAUER and F. FISCHLER (Z. angew. Chem., 1927, 40, 1176—1183, 1276—1279).—The use of boiler-scale preventives containing tannin, hydrocarbons, albuminous substances, etc. may have a scientific foundation, as it may be that such substances act as protective colloids and modify processes of crystallisation. A series of experiments was carried out on the precipitation of calcium carbonate from its acid solution by heating with and without addition of colloids. Without any addition, the rate of precipitation was found to increase with the temperature from 40° to 98°, and with the rate of stirring up to 1200 r.p.m. These factors being uniform, it was found that addition of increasing proportions of dextrin from 0.1% to 0.8% steadily reduced the rate of precipitation. At 98° precipitation was very rapid, but the presence of 0.8% of dextrin reduced the quantity of precipitate by 70%. Gum arabic had a much slighter

effect, but agar-agar and gelatin were even more effective than dextrin. In the last case a thick foam formed on the surface, and such precipitate as was deposited was a non-crystalline slime which contained organic groupings from the colloid. Similar tests under a pressure of 1—10 atm. gave analogous results. With 0.8% of dextrin, up to 85% of the calcium carbonate was retained in solution; with 0.2% of agar-agar a precipitate was at first formed but redissolved. Tannin in the proportion of 0.1% also totally prevented precipitation. In all these cases the calcium in solution is in combination with organic radicals. With smaller quantities of colloid the carbonate precipitated was of a semi-colloidal nature and not coherent.

C. IRWIN.

Influence of free chlorine on the elimination of manganese from water. O. WEBER (Chem.-Ztg., 1927, 51, 794—795).—The elimination of manganese from water by aeration and filtration of the oxidised manganese compounds depends not so much on chemical action as on biological influence. Tabulated results from a waterworks in Hanover show that the manganese content is not lessened by aeration and filtration when free chlorine is added to the raw water; considerable improvement is seen when the chlorine is added after filtration, but the manganese is entirely eliminated only when unchlorinated raw water is used for washing the coke filters. A filter requires time to attain its highest efficiency, and its manganese dioxide content is important.

W. G. CAREY.

Determination of the chlorine value of effluents. H. BACH and K. GLÄSER (Z. angew. Chem., 1927, 40, 1115—1116).—"Chloramine" is used instead of potassium hypochlorite as source of chlorine. Its 0.1N-solution is stable if kept in a dark bottle. To the filtered effluent, containing a small quantity of 10% sodium hydroxide (to fix any carbon dioxide and so prevent the liberation of chlorine from the "chloramine"), is added a known volume of 0.1N-"chloramine" solution and the mixture is gently boiled under specified conditions. A small quantity of 10% potassium iodide is added, then concentrated hydrochloric acid, and, after some minutes, starch-zinc iodide solution, and the iodine is titrated with 0.1N-thiosulphate. A blank experiment must be made. A comparison of this method with the potassium permanganate and biochemical oxygen-absorption methods shows no general agreement in the results.

W. T. K. BRAUNHOLTZ.

PATENTS.

Apparatus for sewage treatment by activated sludge in combination with sludge digestion. K. IMHOFF (U.S.P. 1,642,206, 13.9.27. Appl., 25.8.24; cf. G.P. 418,319; B., 1926, 470).—The sewage is aerated in a tank, into which it is introduced at the bottom. At the top it overflows into an inner settling basin from which the effluent is removed. The sludge falls to the bottom, whence a part is returned to the aeration tank, the remainder being passed to a digestion chamber.

T. S. WHEELER.

Treatment [softening] of hard water. G. PETROFF and P. SHESTAKOFF (U.S.P. 1,642,594, 13.9.27. Appl., 30.12.25).—Sulphonic acids of high mol. wt., e.g., a naphtholsulphonic acid, are employed.

T. S. WHEELER.