

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

NOVEMBER 25, 1927.

### I.—GENERAL; PLANT; MACHINERY.

**Soft rubber filter-press plates and frames.** H. E. FRITZ and J. H. CLARK, JUN. (Ind. Eng. Chem., 1927, 19, 1151).—Plates and frames of semi-hard rubber were tested in the filtration of corrosive liquids at 65 lb./in.<sup>2</sup> and gave satisfactory results with the following: hydrochloric acid, caustic soda, ammonia solution, copper sulphate, etc. (all at any concentration), and sulphuric acid (up to 50%). The limit of temperature is, however, 40–65° according to the material undergoing filtration. No trouble from leakage or distortion occurred. Hard rubber, though resistant, is too fragile and liable to cold-flow for use in this connexion.

C. IRWIN.

**Stainless iron in chemical plant construction.** MITCHELL.—See X.

#### PATENTS.

**Hardness testing device.** C. H. WILSON (E.P. 277,517, 15.10.26).—A test point is caused to penetrate the test piece under a definite load. The point and piece are caused to approach under a minor load until they engage, this motion being effected independently of the size of the test piece. A major load is then applied, causing the penetration, and is kept on long enough to take the reading. The point is then withdrawn ready for another test. All the above motions are performed automatically.

B. M. VENABLES.

**Apparatus for analysing substances by means of Röntgen or cathode rays.** F. DESSAUER. Assee. of METALLBANK & METALLURGISCHE GES. A.-G. (E.P. 253,090, 18.5.26. Ger., 5.6.25. Addn. to E.P. 252,207; B., 1927, 735).—In apparatus constructed in accordance with the prior patent, the primary Röntgen or cathode rays are caused to fall obliquely upon, or to brush against, the substance to be analysed, so that the electronic emission of the characteristic secondary radiation exceeds that of the non-characteristic radiation.

J. S. G. THOMAS.

**Solidification of liquid substances.** I. G. FARBERINID. A.-G., Assees. of K. RAST (G.P. 442,358, 12.12.24).—Liquids, *e.g.*, methyl alcohol, castor oil, liquid hydrocyanic acid, are converted into solids by treatment with magnesium ethoxide in the presence of small quantities of water.

B. FULLMAN.

**Filters.** H. A. VALLEZ (E.P. 277,546, 1.2.27).—The filter is of the type in which a number of leaves are rotated within a casing, slowly when cake building and rapidly when discharging, the filtrate being withdrawn through a hollow shaft. The filter leaves are arranged radially and longitudinally, and besides being attached directly to the shaft they are supported by transverse discs.

B. M. VENABLES.

**Bag filter with outflow pipes disposed on one side for filter bags closed on all sides.** W. H. SCHEIDT (F. SCHEIBLER) (E.P. 272,209, 30.5.27. Ger., 7.6.26).—A method of connecting the outflow pipes to the external pipes is described.

B. M. VENABLES.

**Controlling the saturation temperature of gases and apparatus therefor.** CARRIER ENGINEERING CO., LTD., and S. L. GROOM (E.P. 276,221, 11.1.27).—The gas is passed through a chamber containing sprays of water the temperature of which is controlled by a thermostat fixed in the gas stream. Various methods are described: (a) the water from the sprays is cooled by a liquid, *e.g.*, brine, the flow of which is regulated; (b) the water is cooled in an evaporator, the evaporation of the refrigerating liquid being controlled by the flow of condensing water; (c) a regulated stream of cold water is added to the circulating water in a tank prior to the chamber and any excess water is withdrawn by a system of weirs; (d) the addition of cold water is made through a three-way valve controlled by the thermostat.

A. C. MONKHOUSE.

**Optical pyrometer.** G. KEINATH, Assr. to SIEMENS & HALSKE A.-G. (U.S.P. 1,644,340, 4.10.27. Appl., 23.11.25. Ger., 29.5.25).—See E.P. 244,691; B., 1926, 114.

**Working fluid for refrigeration.** A. A. KUCHER (U.S.P. 1,645,198, 11.10.27. Appl., 27.4.27).—See E.P. 208,547; B., 1924, 621.

**Apparatus for ascertaining the composition of gaseous mixtures.** H. HEINICKE, Assr. to P. JUNG (U.S.P. 1,646,247, 18.10.27. Appl., 16.5.24. Ger., 29.11.23).—See E.P. 225,549; B., 1925, 696.

**[Automatic system for] furnace regulation.** BRIT. THOMSON-HOUSTON CO., LTD., Assees. of A. R. SMITH (E.P. 264,852, 20.1.27. U.S., 20.1.26).

**[Economical firing of boiler] furnaces.** B. LJUNGSTRÖM (E.P. 277,396, 16.3.26).

**Machines for discharging and charging furnaces or retorts.** GIBBONS BROTHERS, LTD., and N. G. COOK (E.P. 278,081, 30.6.26).

**[Sealing device for elements of] heat-exchange apparatus.** SERCK RADIATORS, LTD. From ZIMMERMANN & CO., LUDWIGSHAFEN A./RHEIN MASCHINEN- & APPARATEBAU (E.P. 277,556, 16.2.27).

**Distributing cock for use in subjecting solid material to the action of fluids.** L. MOURGEON (E.P. 277,726, 21.5.26).

**Gas-fired furnaces** (E.P. 263,817).—See II.

**Volumetric gas-displacement apparatus** (E.P. 277,869).—See XXIII.



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

### Free-burning carbonised fuels for the open fire.

H. H. GREGER (J. Akita Min. Coll., 1927, No. 3, 52 pp.).—The theory of combustion in the open grate is discussed, and in connexion therewith an adsorption theory is suggested based on the assumption of fields of force in the micropores of the charcoal or other fuel similar to the magnetic field of force in a solenoid. The suitability of a smokeless fuel for burning in the open grate is dependent on other factors than the ignition temperature, such as, for example, the activation due to oxidation during the initial period of burning; it has been investigated by studying the combustion of a layer of the fuel about 1 cm. in depth, contained in an iron tube 3.55 cm. in diameter, in a regulated downward current of air. The fuel (10–20-mesh) rests on a layer of quartz sand (20–30-mesh), and is ignited by an electrically-heated nichrome spiral lying on the surface. The spiral is heated for 3 or 5 min. and a constant air current passed until the resulting combustion has ceased. The results show the existence of a critical rate of passage of air at which there is a sudden rise, from about 10 to about 80%, in the amount of fuel burnt. This, the "combustion threshold," is the minimum rate which will maintain combustion in the fuel layer. The series of fuels, "black" oak-charcoal, lignite semi-coke, "white" oak-charcoal, semi-coke from a bituminous coal, and a commercial charcoal show a progressive increase in the "combustion threshold" rate from 0.6 to 1.5 litres/min. No continued combustion is maintained with anthracite or high-temperature coke under the experimental conditions. The influence of various factors, *e.g.*, particle size, water content, activation, preheating, on the value of the critical rate has been investigated. A simple apparatus for the rapid determination of the ignition temperature has been designed, in which the fuel is heated by the passage through it of a current of air which passes first through an outer hot layer of sand.

A. B. MANNING.

**Gaseous fuels for furnace heating.** R. WIGGINGTON (J. Soc. Glass Tech., 1927, 11, 293–297).—In producer practice a moderate amount of steam in the blast improved the heating value of the gas, but a further increase was disadvantageous. Sufficient steam was necessary to prevent clinker trouble, but a gas rich in carbon monoxide was preferable to one rich in hydrogen, since it gave a longer flame and greater radiating power, and was less liable to surface combustion. Control of blast saturation temperature was essential, for which purpose a positive blower was better than the usual steam jet.

A. COUSEN.

**Differentiation of the conceptions coal, lignite, and peat.** G. STADNIKOV and N. PROSKURNINA (Brennstoff-Chem., 1927, 8, 305–306; cf. B., 1926, 729).—Lignite exhibits a much greater power of taking up ferric oxide from a solution of ferric chloride than coal of the same particle size. The extent of surface adsorption is probably about the same for both materials, but the humic constituents of lignite exert a chemical attraction for ferric oxide not shown by coal.

W. T. K. BRAUNHOLTZ.

**Relation between the physical nature and adsorptive capacity of coal samples.** D. J. W. KREULEN (Brennstoff-Chem., 1927, 8, 306).—It is emphasised in criticism of the work of Stadnikov and Proskurnina (cf. previous abstract) that the adsorptive capacity of a coal sample can be exactly defined only by relating it to unit area of adsorbing surface. It is also essential that only fresh coal samples are used, since oxidised coal will show similarities to lignite.

W. T. K. BRAUNHOLTZ.

**Formation of hydrocarbons from lignite coke at 500°.** F. FISCHER and H. PICHLER (Brennstoff-Chem., 1927, 8, 307–309).—When steam is passed over activated lignite semi-coke, prepared by carbonising lignite briquettes at 500° and activating by heating the product with a mixture of steam and air, in the presence of a metallic catalyst (*e.g.*, carbonates of manganese, cobalt, or iron, or manganese dioxide) traces of methane and of naphthalene are obtained. The former is, however, produced by the direct action of heat on the semi-coke and disappears on prolonged heating. Contrary to the results obtained by Hofmann and Groll (B., 1927, 289), no higher hydrocarbons are found in the gas. If steam is replaced by dry hydrogen, traces of hydrogenation products (*e.g.*, ethane) are formed.

W. T. K. BRAUNHOLTZ.

**Gas industry: past, present, and future.** W. J. A. BUTTERFIELD (J.S.C.I., 1927, 46, 399–407 r).—An address reviewing progress of public gas supply in Great Britain from 1812 onwards, and contrasting it with that of other public utility services. Official testings under the provisions of the Gas Regulation Act, 1920, covered, at the end of 1926, 71.5% of the gas supplied. They showed that throughout the general strike and coal stoppage of 1926 71.17% of the gas tested was not below its normal calorific value, and a further 18.76% was not as much as 5% below. Comparatively few and unimportant gas undertakings only have been found since 1922 to have supplied in any quarter gas of lower average calorific value than that on which their charge by the "therm" is based. The mean deficiency for all such cases is 1.25%, and in no quarter was more than 0.075% of the gas sold found to be deficient—apart from exceptional cases in the coal emergency period of 1926. Gas supplies now range from 200 to 600 B.Th.U. per cub. ft. in calorific value, but 90% of the whole fall between 440 and 560 B.Th.U. There are at least three gas works sending gas under relatively high pressure to rural districts 20 miles distant. Sales of gas in Great Britain in 1926 were more than four times as great as in 1882, and the consumption registered by each consumer's meter averaged 32,900 cub. ft. in 1926 as compared with 33,360 cub. ft. in 1882. On the basis of heat equivalents, the sales of gas increased between 1922 and 1925 at 2½ times the rate of increase in the sales of electricity by statutory undertakings.

**Disposal of liquor effluents from gas works.** INST. GAS ENG., RES. COMM. REP. I. A. PARKER. II. A. C. MONKHOUSE (Gas J., 1927, 179, 101–104).—I. The principal effluent from gas works arises from the manufacture of ammonium sulphate. This effluent, which contains phenols, thiocyanate, and thiosulphate, interferes with the self-purification of rivers into which



it is discharged and increases the difficulties of sewage purification when discharged into sewers. Of the 45–90 gals. of effluent produced per ton of coal carbonised, 90–85% forms the spent liquor discharged from the ammonia stills and 10–15% the "devil liquor" from the heat-exchangers which follow the sulphuric acid saturator in which the ammonia is absorbed. The oxygen absorption capacity for effluent liquors ranges from 300 to 1000 pts./10<sup>5</sup>, whereas the corresponding figure for average domestic sewage is 10 pts./10<sup>5</sup>. The volume of effluent liquor to be treated is usually 0.5–1.0% of the volume of sewage, and in this proportion it can be successfully treated if sudden changes in the proportion of effluent to sewage are avoided. Purification of the effluent by bacterial filtration reduces its oxygen absorptive capacity by 95%, but the initial cost of the process is large. Partial purification by the use of dephenolating towers reduces the oxygen absorptive capacity by only 20–30%. Admixture with sewage appears to be the only satisfactory method at present known, and it is suggested that arrangements should be made to run the effluent into sewers in regulated quantity.

II. Experiments made at Hinckley Gas Works relating to the problem are described. The total volume of effluent, of which 14% was "devil liquor," was 1.4 times the volume of ammoniacal liquor produced. The oxygen absorption capacity of the "devil liquor" was 20% of that of the total effluent. The circulation of liquor from a common tar and liquor well through the hydraulic main is unsatisfactory in that it produces a loss of ammonia and the formation of thiosulphate and thiocyanate. The storage of liquor and tar in a common well favours the dissolution by the liquor of higher tar acids, which should be avoided by early tar and liquor circulation. Preliminary experiments on the purification of effluent liquor by biological filtration have shown that with a rate of flow of 60 gals. per day per cub. yard of filtering material in each of two filters, 85% purification can be obtained with a mixture containing 8% of effluent liquor. Filter beds matured in this manner can be used for the treatment of effluent liquor diluted with bacterially purified effluent.

S. PEXTON,

**Cholesterol as parent of petroleum.** N. D. ZELINSKI (Ber., 1927, 60, 1793–1800).—A mixture of cholesterol and aluminium chloride is cautiously heated in a small flask with a free flame; the weight of the distillate is 66% of that of the cholesterol, and includes gaseous products condensed at –70°. The liquid distillate is treated with steam and the volatile portions are then distilled, yielding mainly a fraction, b.p. 60–150°,  $d_4^{20}$  0.7416,  $n_D^{20}$  1.4154, which is somewhat unsaturated. After hydrogenation in the presence of palladised asbestos at 170°, a saturated product, b.p. 60–150°,  $n_D^{21}$  1.4136, is obtained consisting mainly of cyclic compounds. From it a mixture of hexane and heptane hydrocarbons, 1:4-dimethylcyclohexane, and methylcyclohexane are derived. The less volatile oils have b.p. 150–400°/atmos. pressure without decomposition. In composition they closely resemble several natural petroleum products containing oxygen. With increasing b.p. the fluorescence of the individual fractions increases in intensity and the magnitude of the dextro-rotation

increases, corresponding to that of fractions of Campina and Schodnica petroleum. Treatment of the fractions of cholesterol oil with sulphuric acid causes diminution of the density of those of higher b.p., renders them almost inactive optically, and removes the fluorescence; precisely similar observations are made with natural petroleum products. The possible presence of traces of unchanged cholesterol in the higher fractions cannot be examined by the usual colorimetric tests. The carbonised residue which remains in the distillation flask yields a resin poor in hydrogen when extracted with benzene. Oleic, stearic, and palmitic acids are converted by aluminium chloride into mixtures of liquid and solid hydrocarbons which differ in character from those derived from cholesterol.

H. WREN.

**Esthonian oil shale: origin of oil shales.** P. N. KOGERMAN (Sitzungsber. Naturforsch.-Ges. Univ. Tartu, 1927, 34, 166–182; cf. B., 1927, 354).—Esthonian oil shale, "kukersite," contains approximately 43.4% of organic matter (kerogen). Analysis of the latter corresponds nearly to an empirical formula  $(C_6H_8O)_n$ . Kerogen from Scottish shale oil has an empirical formula  $(C_6H_{10}O)_n$ ; hence the kerogen of kukersite is less saturated. Some derivatives of hydroaromatic compounds, e.g., resins, unsaturated aldehydes, etc., have the same general formula as the kerogen of kukersite. The kerogen is insoluble in all ordinary organic solvents, and is apparently a highly polymerised "resinous substance," very resistant to the action of reagents (e.g., fusion with potash or sulphur) and depolymerised by the action of halogens and heat only. The character of the distillates obtained on thermal decomposition of the shale recalls the character of those obtained from resins and rubber.

R. C. ODAMS.

**Fluorescence of low-temperature distillation products of oil shales.** M. WITTLICH (Brennstoff-Chem., 1927, 8, 309–310).—Oils obtained from the low-temperature distillation of different oil shales exhibit, when examined by means of a quartz lamp, different fluorescence according to their origin and method of production and purification. The fractions can be arranged in order of their b.p. by means of their different fluorescence.

W. T. K. BRAUNHOLTZ.

#### PATENTS.

**Drying process for fuel.** A. V. LIPINSKI (Swiss P. 117,203, 22.10.25).—Fuel oil is completely burnt in a suitable combustion chamber, the products are mixed with several times their volume of air, and this gas mixture is passed into the drying chamber. In this manner the full heating value of the fuel is utilised, and the drying gases are free from sulphur dioxide, carbon monoxide, and other objectionable constituents.

A. B. MANNING.

**Apparatus for washing and treating coal.** ILLINGWORTH CARBONIZATION CO., LTD., and S. R. ILLINGWORTH (E.P. 276,382, 22.2.26).—Coal is treated with a liquid such as trichloroethylene in washing troughs through which run conveyors arranged to give an up-and-down motion to the coal. The clean coal passes to a pitch tank and from there to a dryer. The dirt is also dried and the vapours from the two dryers pass to a condenser, the water being separated and the liquid returned to



the tanks feeding the troughs. Any vapour left in the gas is recovered in a tower packed with charcoal.

A. C. MONKHOUSE.

**[Coal] grinding and drying apparatus.** J. E. BELL, Assr. to COMBUSTION ENGINEERING CORP. (U.S.P. 1,641,409, 6.9.27. Appl., 12.1.22).—The grinding mill is placed in a conduit through which a heating medium is passed.

T. S. WHEELER.

**Treatment of fuels.** L. B. DEVILLARS (Swiss P. 117,304, 23.10.25).—Coal, coke, or lignite, etc. is powdered, dried *in vacuo*, and mixed with a fluid material which increases its calorific value and which solidifies in the cold. A suitable material consists of 20–60% of tar, 3–20% of magnesite, 0.5–15% of naphthalene, and 10–35% of chalk. The treated fuel is dried and given a coating of waterproof material.

A. B. MANNING.

**Cleaning of carbonaceous materials.** R. LESSING (E.P. 276,723, 27.5.26).—After the removal of fine dust the coal is fed into the middle of a bath of liquid of suitable density, *e.g.*, an alkaline solution of calcium chloride. The liquid is stirred at the point of entry of the coal, and the "coal" and "dirt" are separated. The two fractions are removed to separate towers and the solution removed by downward displacement with a continuous flow of weak bath solution and then water. A definite line of demarcation between the liquids is obtained, and the solution is returned for further use, or, if necessary, first concentrated. The loss of solution is reduced to less than 1% of the weight of coal.

A. C. MONKHOUSE.

**Preparation of humin substances from coal.** M. PIETTRE (F.P. 582,400, 6.9.23).—Powdered coal is treated with concentrated nitric acid; the mixture is cooled during addition of the acid, is then warmed slowly to 70°, and maintained at that temperature for 60–80 hrs. The washed and dried nitrated product, which has an average nitrogen content of 4.65%, is dissolved in concentrated alkali and reduced by treatment with an excess of powdered zinc or iron. Reduction requires several days, and is complete when, on acidifying and drying the product, this no longer burns with the evolution of nitrous fumes. The washed and dried product is black and lustrous, has a conchoidal fracture, and possesses chemical properties similar to those of the humic acids. Both nitrated and reduced products are soluble in pyridine.

A. B. MANNING.

**Coke ovens.** A. W. MATHYS. From TAR & PETROLEUM PROCESS CO. (E.P. 277,256, 11.5.27).—A rectangular coke oven is heated by combustion flues under the sole of the coking chamber, and extending the entire length of the latter. Regenerators arranged beneath the flues supply preheated air alternately to each end of each flue, where also means for supplying gas are provided. Each combustion flue is provided with movable tunnel nozzles for distributing a portion of the air supply from the regenerators and for discharging waste-heat gases from the flue to other regenerators; these permit control of the uniformity of heating in the flues.

A. B. MANNING.

**Coke oven.** W. H. WRIGHT, Assr. to FOUNDATION OVEN CORP. (U.S.P. 1,643,532, 27.9.27. Appl., 7.6.21).—

A coke oven has coking chambers which are wider at one side than at the other. The combustion flues in the flue wall are arranged in groups, each of which communicates independently of the others with a pair of sole flues, which have a single opening at the end, to be used alternately for exhaustion of combustion gases and, on reversal, for admission of air. The effective areas of the passages from the sole flues to the combustion flues and the latter also are graduated in size continuously, increasing from one side to the other of the coking chamber as the width of the latter increases.

A. B. MANNING.

**Manufacture of hard coke.** P. DVORKOVITZ (E.P. 276,181, 15.10.26. Addn. to E.P. 249,901; B., 1926, 477).—An alkali, *e.g.*, lime or soda, is mixed with the coal or other material to be carbonised before or during the preliminary heating prior to carbonisation.

A. C. MONKHOUSE.

**Solid smokeless fuel.** ILLINOIS ANTHRACITE CORP., Assees. of C. S. LOMAX and W. M. GRANT (Can. P. 263,017, 19.10.25).—A bed of carbonaceous material of constant depth is maintained in a generator, the combustion in which is of such a nature that a relatively small proportion of the volatile matter, but no solid material, is consumed. The material in the generator is stirred continuously.

A. B. MANNING.

**Dry-cooling of gas-works coke.** P. GYGAX, and A.-G. KESSELSCHMIEDER RICHTERS WIL (Swiss P. 117,957, 13.11.25).—The coke is discharged into a jacketed container, the jacket of which is filled with some suitable liquid and is connected with a heat exchanger.

A. B. MANNING.

**Cooling granular combustible material such as coke, semi-coke, etc.** DEMAG A.-G. (G.P. 442,353, 26.6.24).—Separation into the various grain sizes and cooling of the separated material take place simultaneously during the course of conveyance, and the treated material is then partly or wholly remixed, also during its conveyance. The procedure economises in time and cost.

A. B. MANNING.

**Motor fuel.** G. MORETTI (F.P. 617,481, 12.6.26. Italy, 16.6. and 30.6.25).—A mixture of alcohol with a small quantity of acetone, hydrogen peroxide, potassium chlorate, resin, and grease is saturated with acetylene.

A. B. MANNING.

**Motor fuel.** H. P. BASSETT (U.S.P. 1,641,520, 6.9.27. Appl., 26.7.22).—Aniline (0.2%) and nitrotoluene (0.1%) are added to a hydrocarbon motor fuel to reduce the tendency to knock.

T. S. WHEELER.

**Pulverulent fuels for use in operating internal-combustion engines.** I. G. FARBERIND. A.-G. (E.P. 270,703, 2.5.27. Ger., 7.5.26).—To prevent the abrasive action of the mineral constituents the fuels are treated with hydrofluoric acid to remove wholly or partly the silicic acid.

A. C. MONKHOUSE.

**Apparatus for the purification of graphite by froth flotation.** A. GALLOIS (F.P. 617,697, 13.11.26).—A mixture of graphite with a special liquid is conveyed automatically from a mixer into frothing apparatus.

L. A. COLES.

**Manufacture of carbon granules for telephone transmitters.** F. O. BARRALET (E.P. 277,129, 17.6.26).



—A pure organic compound, *e.g.*, furfuraldehyde, sugar, is heated with a small quantity of a carbonising agent, *e.g.*, sulphuric acid, and the colloidal carbon so produced is subjected to further heat treatment, *in vacuo*, under pressure, or in a non-oxidising atmosphere. The dense product thus obtained is then crushed and graded.

A. B. MANNING.

**Production of hard homogeneous combustible material or objects such as picture frames, blackboards, lighting structures, etc. from peat, peat moss, lignite, and similar substances.** B. JIROTKA (E.P. 276,471, 30.3.26).—The material to be treated is first broken up by mechanical or chemical means and, after compression by a worm-feed, passes between rotating discs where it is subjected to a rubbing action and water is removed. The product obtained dries readily or can be moulded when plastic.

A. C. MONKHOUSE.

**Gas-fired furnaces, more particularly for coke- and gas-producing furnaces.** C. STILL (E.P. 263,817, 23.12.26. Ger., 28.12.25).—In a vertical-flued coke oven of the regenerative type two regenerators are arranged for each half oven. When poor gas is used as the heating gas the gas passes through one of the regenerators to a separate horizontal flue, and thence to the burner nozzles, while the other regenerator is used for preheating the air. When rich gas is being used, by an adjustment of dampers both regenerators are used for the preheating of air, the regenerators being connected to the same horizontal flues. This principle is applicable to furnaces where recuperative or regenerative devices are employed.

A. C. MONKHOUSE.

**Production of enriched illuminating or power gas from coal or other fuel capable of being distilled.** J. W. GIBSON (E.P. 276,065, 17.5.26).—In a water-gas plant the carburettor is dispensed with by mixing the oil for enrichment with the steam supply to the generator. The mixture passes through a superheater fixed in the generator wall before admission to the fuel bed.

A. C. MONKHOUSE.

**Manufacture of water-gas.** HUMPHREYS & GLASGOW, LTD., and J. C. STELFOX (E.P. 276,753, 4.6.26).—In a complete gasification plant in which the fuel in the retort is heated internally by the hot gases from the generator below, the formation of a central core of partially carbonised fuel is prevented by suspending a core of metal or refractory material down the centre of the retort.

A. C. MONKHOUSE.

**Generation of carburetted water-gas from bituminous fuel.** O. MISCH (E.P. 276,530, 8.11.26).—A retort which is superimposed on a generator is heated externally by burning water-gas under pressure in a jacket surrounding the retort. The temperature of the lower end of the retort is maintained at 700° or higher. The gases then pass in a spiral direction round the retort and are used for generating steam. The temperature of the upper part of the retort is regulated by a water jacket. Tar or oil for carburetting is admitted in a ring-shaped pipe half-way down the retort.

A. C. MONKHOUSE.

**Manufacture of gas from heavy oils.** C. CHILOWSKY (E.P. 271,907, 19.5.26. Belg., 16.7.27).—The heavy oil

is atomised by preheated air and steam, and the temperature raised to 900–1000° by partial combustion of the oil. The mixture is passed over an incandescent refractory or metallic catalyst, when a nearly complete transformation is effected into non-condensable gas and very light hydrocarbons.

R. C. ODAMS.

**Treatment of gases by the employment of solid catalytic and other reactive agents.** D. M. HENSHAW, S. G. WATSON, and W. C. HOLMES & Co. (E.P. 276,736, 12.6.26).—An apparatus for the continuous purification of gases (*e.g.*, removal of hydrogen sulphide by iron oxide, moisture or benzol vapours by silica gel, etc.) consists of an elongated casing with foraminous walls containing the purifying agent and situated inside a chamber to which the gases are admitted. Baffles to cause the gases to pass repeatedly through the purifying agent by a zig-zag path and means for renewing the agent as desired without discontinuing the process are provided.

C. O. HARVEY.

**Elimination of hydrogen sulphide from gases.** C. STILL (E.P. 271,805, 4.8.26. Belg., 25.5.26).—In a process for the removal of hydrogen sulphide from gases by washing with a suspension of ferrous and ferric hydroxides or carbonates, and regeneration of the latter by oxidation with air, the iron content of the suspension is maintained at not more than 2%, and the sulphur-containing foam from the regenerator is floated off into another vessel. After separation from the excess washing liquid, the sulphur is recovered from the foam by centrifuging. The regenerator consists of a tall narrow tower filled with the liquid, the oxidising air being introduced under pressure at the bottom. The wash liquid may be made alkaline, either by absorption of ammonia from the gas or by addition of alkali; the liquid is then circulated until a highly concentrated solution of thiosulphate is formed.

A. B. MANNING.

**Distillation of carbonaceous materials.** H. NIELSEN and B. LAING (E.P. 276,407, 6.3.26).—Carbonisation takes place in two stages in an internally-heated rotary retort. In the first stage (185–580°) the products of distillation flow in the same direction as the inert heating gas, thus preventing the cracking of the oils and deposition of graphitic carbon on the pores of the coke; in the second stage (580–1200°) superheated water- or producer-gas is used as the heating gas. The coke obtained contains 1–4% of volatile matter, and is easily ignited. The oil obtained has a static coefficient of friction of 0.100–0.185 at 12–18°, and is suitable for lubricating purposes.

A. C. MONKHOUSE.

**Low-temperature distillation.** BAMAG-MEQUIN A.-G., and O. HELLER (E.P. 268,745, 11.3.27. Ger., 1.4.26).—The material after passing through a dryer is fed into one of two rotary drums containing previously-heated refractory material made of tiles in lattice formation of varying designs. Carbonisation is effected at 450–850° and the distillation gases are withdrawn in an opposite direction to the moving charge. When one drum is in use the other is heated, and the heating gases pass subsequently to the dryer and then to a dust extractor, where the fuel recovered is used as pulverised fuel in the furnace.

A. C. MONKHOUSE.



**Low-temperature distillation of carbonaceous material.** SYNTHETIC AMMONIA & NITRATES, LTD., R. E. SLADE, and C. F. R. HARRISON (E.P. 276,522, 13.10.26).—The finely-divided material is preheated to 100–200° and fed into a horizontal rotary retort through which steam superheated to 450° is passed in counter-current flow. Very little permanent gas is produced, and the products of distillation are recovered without condensation of steam by injecting a small quantity of water and then washing with oil at 140–150°. The steam is returned to the superheater for re-use. A portion of the circulating gas is removed from time to time as the permanent gas accumulates. The system may be worked under increased or diminished pressure and catalysts mixed with the fuel. A. C. MONKHOUSE.

**Cracking of mineral oil and other hydrocarbon material.** J. F. P. DE LA RIBOISIÈRE (E.P. 276,532, 12.11. and 31.12.26).—Liquid or solid fuels are cracked under pressure, *e.g.*, 5–10 kg./cm.<sup>2</sup>, and the vapours produced are passed through a tower or column packed with Raschig rings or some highly porous material. The tower is maintained at a constant temperature by the introduction into the gas stream of a liquid, *e.g.*, water or benzol, under pressure. A. C. MONKHOUSE.

**Process of carrying out chemical reactions with liquids [cracking of hydrocarbons].** E. H. LESLIE and B. R. TUNISON (U.S.P. 1,644,736, 11.10.27. Appl., 4.9.20).—A supply of liquid hydrocarbons is uniformly and intermittently distributed over the surface of a second supply of liquid hydrocarbons, the temperature of the latter being higher than that of the former, so that a conversion temperature is attained at least as high as the b.p. of one of the oil fractions. R. C. ODAMS.

**Conversion [cracking] of oil.** W. M. CROSS, Assr. to GASOLINE PRODUCTS Co., INC. (U.S.P. 1,644,991, 11.10.27. Appl., 3.4.23).—Oil is cracked in the liquid phase, and the products are then vaporised and fractionally condensed, a regulated liquid level being maintained in the latter stages. Selected portions of the condensate are returned, together with the charging stock, to the heating and condensing stages.

R. C. ODAMS.

**Preparation and recovery of light oil or motor fuel from crude mineral or shale oil, tar oils, or carbonaceous materials.** E. SCHULTZ (E.P. 277,042, 9.3.26).—A modification of the process previously described (E.P. 254,011; B., 1926, 779) wherein oils or carbonaceous materials are heated in a retort, and the vapours are passed (at temperatures below that of the retort) through a converter containing salt, lime, zinc chloride, etc., consists in treating the heavier and lighter fractions separately and employing for the conversion substances such as montmorillonite, marialite, bentonite, lime and metallic sodium, aluminium chloride, or mixtures of these substances. C. O. HARVEY.

**Production of liquid hydrocarbons and derivatives thereof from coal, tar, etc.** I. G. FARBENIND. A.-G. (E.P. 277,273, 13.3.26. Addn. to E.P. 247,217, and 249,155; B., 1927, 601, 805).—In the conversion of coal, tars, mineral oils, etc. by treatment under pressure and at elevated temperatures with gases containing hydrogen or oxides of carbon, the cold as well

as the hot parts of the apparatus are made of a metal which does not react with carbon monoxide. Other reducing gases may be used. C. O. HARVEY.

**Transforming methane into a carburetting agent similar to petroleum.** H. SPINDLER, Assee. of A. GOUDET (E.P. 255,493, 19.7.26. Fr., 18.7.25).—Methane, either alone or with other gases capable of combining with hydrogen, is subjected to high pressure (70–200 atm.) and temperature (500–950°), when partial decomposition takes place with formation of hydrogen. The gaseous mixture is cooled to 250–350° and passed over a catalyst consisting of a mixture of zinc chloride, active carbon, aluminium and magnesium powders, and iron scale previously treated with alkali hydroxides. Liquefiable hydrocarbons of a higher order than methane are produced. R. C. ODAMS.

**Apparatus for the manufacture of carbonised fuel.** S. R. ILLINGWORTH, Assr. to ILLINGWORTH CARBONIZATION Co., LTD. (U.S.P. 1,645,861, 18.10.27. Appl., 27.9.24. U.K., 29.10.23).—See E.P. 223,624; B., 1925, 3.

**Manufacture of active carbon.** J. N. A. SAUER (U.S.P. 1,641,053, 30.8.27. Appl., 29.7.24. U.K., 9.8.23).—See E.P. 206,862; B., 1924, 548.

**Production of carbon [black].** C. MATLOCK, Assr. to MONROE-LOUISIANA CARBON Co. (Re-issue 16,765, 11.10.27, of U.S.P. 1,458,351, 12.6.23).—See B., 1923, 820 A.

**Process of refining mineral oils.** T. HELLTHALER, Assr. to H. STINNES-RIEBECK MONTAN- u. ÖLWERKE A.-G. (U.S.P. 1,645,530, 18.10.27. Appl., 1.8.25. Ger., 3.12.24).—See G.P. 421,858; B., 1926, 263.

**Manufacture of liquid fuels.** M. MÜLLER-CUNRADI and W. WILKE, Assrs. to BADISCHE ANILIN- & SODA-FABR. (U.S.P. 1,646,014, 18.10.27. Appl., 15.7.24. Ger., 14.1.24).—See E.P. 226,731; B., 1925, 163.

**Apparatus for discharging coke ovens etc.** STETTNER CHAMOTTE-FABR. A.-G. (E.P. 276,325, 8.8.27. Ger., 17.8.26).

**Doors for gas retorts.** R. HADDAN. From STETTNER CHAMOTTE A.-G. VORM. DIDIER (E.P. 278,269, 9.4.27).

### III.—TAR AND TAR PRODUCTS.

#### PATENTS.

**Manufacture of organic compounds.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (E.P. 277,394, 13.3.26).—Aromatic hydrocarbons or phenols (or tar fractions containing these), when passed with excess of steam over hydrating and dehydrogenating catalysts or catalyst mixtures at 300–500°, are converted into lower homologues. *o*-Cresol over copper on bauxite gives phenol; sulphur-free naphthalene, or crude naphthalene containing phenols, over nickel and alumina on china clay gives benzene, toluene, *o*-xylene, etc. Nickel, cobalt, iron, and copper favour dealkylation, the first three being also suitable for removing fused nuclei. Catalyst poisons should be removed from the vapours. Oxygenated compounds may first be reduced with hydrogen to hydrocarbons. The side-chains are removed chiefly as carbon dioxide and hydrogen, with



a little methane and ethylene; the gases produced are thus suitable for further catalytic reactions, *e.g.*, the production of alcohol or of pure hydrogen.

C. HOLLINS.

**Light oil from tar oils** (E.P. 277,042 and 277,273).—See II.

#### IV.—DYESTUFFS AND INTERMEDIATES.

##### PATENTS.

**Manufacture of emulsions.** I. G. FARBENIND. A.-G. (E.P. 267,534, 10.3.27. Ger., 10.3.26).—Aryl dihydrogen phosphates, diaryl hydrogen phosphates, or their salts are used alone or with other emulsifiers for the production of emulsions of non-phenolic organic compounds in liquids in which the compounds are substantially insoluble. Examples are sodium ditolyl phosphate ("sodium dicresyl phosphate") for emulsification of cyclohexanone in water, oleic acid in water, aniline in water, and cyclohexanone and benzene in water; sodium ditolyl phosphate, disodium tolyl phosphate or sodium dinaphthyl phosphate for dispersion of Indanthrene Blue RS in water; sodium diphenyl or dixyl phosphate for dispersion of Indanthrene Golden Orange G in water.

C. HOLLINS.

**Production of quinone derivatives [leuco-esters of vat dyes].** B. WYLAM, J. E. G. HARRIS, H. A. E. DRESCHER, J. THOMAS, and SCOTISH DYES, LTD. (E.P. 277,398, 9., 22., and 23.4.26, and 10.1.27).—In the preparation of leuco-esters from vat dyes by the action of a metal, a tertiary base, and a substance (oleum, chlorosulphonic acid, sulphur trioxide, alkyl chlorosulphonates) which forms a sulphur trioxide compound of the tertiary base, it is found advantageous to add certain "assistants," namely, phthalic or succinic anhydride or imide, phosgene, ethyl chloroformate, acetyl chloride, benzoyl chloride, toluene-*p*-sulphonyl chloride, or other organic acid chlorides. The process is illustrated by 17 examples.

C. HOLLINS.

**Manufacture of [sulphide vat] dyes.** SOC. CHEM. IND. IN BASLE (GES. F. CHEM. IND. IN BASEL) (E.P. 270,348, 2.5.27. Switz., 30.4.26. Addn. to E.P. 199,360; B., 1924, 9).—In the thionation of indophenols from carbazole or *N*-alkylcarbazoles in presence of arylamines or their derivatives (cf. prior patent), markedly greener shades are obtained by adding urea to the sulphide melt.

C. HOLLINS.

**Manufacture of stable preparations of vat dyes.** I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 259,999, 18.10.26. Ger., 17.10.25).—A dry vat dye, with or without a dispersing or wetting-out agent, is mixed with a dry alkali-starch preparation; a dry hyposulphite may also be added. The preparations remain unaltered when kept, and give clear vats on addition of water (or of hyposulphite solution if hyposulphite is absent), which contain no excess of alkali and are thus suitable for wool dyeing.

C. HOLLINS.

**Production or development of azo dyes.** H. T. BUCHERER (E.P. 249,526, 16.3.26).—Nitrosoamines (*anti*-diazotates) can be made to couple with coupling components by using a slightly alkaline medium at 90–100°. Strongly alkaline nitrosoamine printing pastes are nearly

neutralised (*e.g.*, with calcium chloride) before steaming, the neutralising agent being added to the printing paste and/or to the fabric before printing.

C. HOLLINS.

**Manufacture of an [azo] dye [for acetate silk].** SOC. CHEM. IND. IN BASLE (GES. F. CHEM. IND. IN BASEL) (E.P. 256,205, 14.7.26. Switz., 1.8.25).—Diazotised aniline, coupled with 1-*o*-chlorophenyl-3-methyl-5-pyrazolone, gives a yellow dye for acetate silk. The dye paste is preferably ground with a protective colloid, such as sulphite-cellulose liquor.

C. HOLLINS.

**Manufacture of new intermediate compounds and of azo dyes therefrom.** BRIT. DYESTUFFS CORP., LTD., J. BADDILEY, P. CHORLEY, and R. BRIGHTMAN (E.P. 277,756, 25.6.26).—Unsymmetrical carbamides are obtained by phosgenating equimolecular mixtures of 2-amino-8-naphthol-6-sulphonic acid ( $\gamma$ -acid) and an aminoacetanilide or a sulphonated amine of the benzene or naphthalene series (excluding sulphonated diamines, aminoformanilides, and aminonaphtholsulphonic acids), in presence of sodium acetate etc. The products when coupled with diazo compounds give dyes for cotton and regenerated cellulose silks. The product from  $\gamma$ -acid and *p*-aminoacetanilide couples with diazotised dehydrothio-*p*-toluidine to give a bordeaux, with diazotised *p*-chloroaniline a red, on viscose silk; with diazotised *p*-aminoazobenzenesulphonic acid a claret, and with diazotised *m*-sulphobenzeneazo- $\alpha$ -naphthylamine a blue, on cotton. The preparation and couplings of 8-hydroxy-1' : 2-dinaphthylcarbamide-4' : 6-disulphonic acid, and 8-hydroxy-2 : 2'-dinaphthylcarbamide-6 : 6'-disulphonic acid are also described.

C. HOLLINS.

**Manufacture of pyrazolone-azo dyes.** I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 261,770, 19.11.26. Ger., 19.11.25).—Azo dyes are made by coupling a diazo compound with 1-(4'-chloro-6'-sulpho-3'-methylphenyl)-pyrazolones containing in the 3-position a methyl, carboxyl, or carbalkoxy-group. The preparation of fast yellow wool dyes from diazotised aniline-*o*-sulphonic acid, aniline, *p*-chloroaniline-*o*-sulphonic acid, and *o*-toluidine-3 : 5-disulphonic acid, and from tetrazotised  $\beta\beta$ -di-*p*-aminophenylpropane is described. The dye, *o*-toluidine-3 : 5-disulphonic acid  $\rightarrow$  the pyrazolone-3-carboxylic acid, gives a very greenish yellow lake on barium sulphate.

C. HOLLINS.

**Manufacture of cyclotrimethylenearylpyrazolones [1-aryl-3 : 4-trimethylene-5-pyrazolones].** C. MANNICH (E.P. 260,577, 18.10.26. Ger., 27.10.25).—The arylhydrazones of cyclopentanone-2-carboxylic esters are cyclised by sodium alkoxides, forming 1-aryl-3 : 4-trimethylene-5-pyrazolones, which are also obtainable directly from the components in presence of sodium alkoxides without isolation of the hydrazones. 1-Phenyl- (m.p. 183–184°), 1-*p*-bromophenyl- (m.p. 200°), and 1-*p*-tolyl- (m.p. 202°) derivatives are described.

C. HOLLINS.

**Manufacture of alkyl and aralkyl derivatives of cyclotrimethylenearylpyrazolones [1-aryl-2-alkyl- or aralkyl-3 : 4-trimethylene-5-pyrazolones].** C. MANNICH (E.P. 263,773, 1.12.26. Ger., 31.12.25).—By treatment with alkylating or aralkylating agents (*e.g.*,

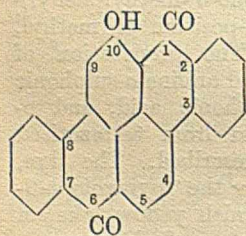


methyl sulphate, ethyl bromide,  $\beta$ -bromoallyl bromide, or benzyl chloride, in presence of alkali) the products of E.P. 260,577 (preceding) are converted into compounds having antipyretic and analgesic properties. 1-Phenyl-3-methyl-3:4-trimethylene-5-pyrazolone, m.p. 128°, the 1-phenyl-2-ethyl compound, m.p. 143°, the 1-p-tolyl-2-ethyl compound, m.p. 118°, the 1-p-bromophenyl-2- $\beta$ -bromoallyl compound, m.p. 109°, and the 1-phenyl-2-benzyl compound, m.p. 104°, are described. C. HOLLINS.

**Manufacture of anthraquinone nitriles.** I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS & BRÜNING (E.P. 261,422, 15.11.26. Ger., 13.11.25).—Halogenated anthraquinones are heated with cuprous cyanide and an alkyl or aralkyl cyanide (particularly phenylacetone nitrile). A complex compound separates on cooling or addition of alcohol, from which, by boiling with dilute nitric acid and removal of alkyl or aralkyl cyanide by steam-distillation, the cyanoanthraquinone is obtained free from copper. Halogen atoms in polyhalogenoanthraquinones may be replaced successively by cyano-groups. The preparation of 1:5-dicyanoanthraquinone, decomp. 380°; 1-cyanoanthraquinone, m.p. 245–247°; 8-chloro-1:4:5-tricyanoanthraquinone; 1:4-dicyanoanthraquinone, m.p. 389–390°; and 1:4:5:8-tetracyanoanthraquinone, is described. All the nitriles give green vats. C. HOLLINS.

**Manufacture of N-dihydro-1:2:1':2'-anthraquinoneazine.** J. H. SACHS, ASSR. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,640,724, 30.8.27. Appl., 23.1.25).—An aqueous suspension of the leuco-compound is treated with air at 100° to give a product of increased purity. T. S. WHEELER.

**Manufacture of benzanthrone derivatives.** I. G. FARBENIND. A.-G. (E.P. 263,845, 29.12.26. Ger., 30.12.25.



Addn. to E.P. 248,791; B., 1927, 550).—2-Aroyloxybenzanthrone is converted by acid condensing agents (sodium aluminium chloride) into hydroxydibenzpyrenequinone derivatives. 2-Benzoyloxybenzanthrone, m.p. 215–216° (prepared by Schotten-Baumann reaction), gives at 180–240° 10-hydroxy-

2:3:7:8-dibenzpyrene-1:6-quinone (annexed formula), the methyl and ethyl esters of which are yellow vat dyes. 2-p-Toluyloxybenzanthrone, m.p. 212–213°, similarly yields 10-hydroxy-4'-methyl-2:3:7:8-dibenzpyrene-1:6-quinone; the methyl ether is a yellow vat dye.

C. HOLLINS.

**[Manufacture of] black sulphurised naphthol dyes.** C. H. MARSCHALK (U.S.P. 1,641,182, 6.9.27. Appl., 11.3.24. Fr., 16.3.23).—See E.P. 212,936; B., 1925, 582.

**[Production of aromatic sulphonic acids by] substituting halogen.** K. SCHÖLLKOPF, ASSR. to RHEINISCHE KAMPFER-FABR. G.M.B.H. (U.S.P. 1,645,974, 18.10.27. Appl., 16.8.23. Ger., 23.8.22).—See E.P. 202,975; B., 1924, 937.

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

**Analysis of cotton.** Effect of disruption of the cotton hair on the extraction of fat, wax, and resin. R. G. FARGHER and L. HIGGINBOTHAM (J. Text. Inst., 1927, 18, 283–287 T).—The effect of destroying the structure of the cotton hair is considerably smaller than was indicated by Knecht and Streat (B., 1923, 345 A), the extracts being due, not to material which cannot otherwise be removed, but to the easier removal of substances which are only slowly extracted from normal hairs. There is no evidence for the existence of relatively large amounts of wax and resin which are protected from attack by their position within the hair. B. P. RIDGE.

**Deterioration of cotton during damp storage.** A. C. BURNS (Ministry Agric., Egypt, Tech. & Sci. Service, 1927, Bull. No. 71, 92 pp.).—A full account of work previously described (B., 1925, 666). Spraying the unginned cotton with bactericides or fungicides might be used to prevent deterioration, but presents difficulties. Toluene, formaldehyde, and naphtha are too inflammable, whilst carbon tetrachloride has too powerful a solvent action, and, unless applied under strictly controlled conditions, affects the spinning qualities of the cotton. Hydrocyanic acid gas is probably the most suitable (except for poison risks to workmen), and comparative tests show that the spinning qualities of the treated cotton are unimpaired. B. P. RIDGE.

**Influence of humidity on the breaking load of cotton at 20°.** J. C. MANN (J. Text. Inst., 1927, 18, 253–264 T).—Tests show that the strength of single cotton hairs increases with increasing humidity, the increase being of the order 10–15% of the breaking load at 44% R.H., and that above 66% R.H. the effect of humidity is negligible. B. P. RIDGE.

**Mothproofing of fabrics and furs.** L. E. JACKSON and H. E. WASSELL (Ind. Eng. Chem., 1927, 19, 1175–1180).—By submitting a large number of possible moth-repelling chemicals to an exhaustive series of tests, the cinchona alkaloids and their derivatives have been found to be particularly effective as mothproofing agents. Application to the fabric is made by spraying or by immersion in a solution of the alkaloid. These substances possess the added advantages that they are inodorous, non-toxic to human beings, and non-injurious to fabrics. They adhere to the material on which they are used without being apparent, and may be employed in a variety of solvents by making use of salts of different solubilities. For use with solvents ordinarily employed in "dry-cleaning" plants, the fatty acid compounds are very suitable as, unlike the majority of the cinchona derivatives, they are readily soluble in these solvents. Quinidine salts have so far proved the most economical for industrial use. F. R. ENNOS.

**Analysis of cotton.** Action of hot dilute sodium hydroxide solutions on modified cotton cellulose. D. A. CLIBBENS, A. GEAKE, and B. P. RIDGE (J. Text. Inst., 1927, 18, 277–284 T).—Oxycelluloses suffer a considerable fall of viscosity in cuprammonium solution after boiling for 6 hrs. with 1% sodium hydroxide,



whilst hydrocelluloses and normal bleached cotton are but slightly affected. The magnitude of this fall varies with the method of preparation of the oxycellulose, but is very great for neutral hypochlorite oxycelluloses—the most frequently occurring type of technically oxidised cotton. An exactly similar behaviour is shown when the alkali boil is replaced by the treatment given during the determination of copper number by Braid's method. This test for the nature of the chemical attack of cotton is, therefore, conveniently made by comparing the viscosity of a sample with that of the material remaining after the determination of its copper number. The copper numbers of modified celluloses are reduced to one fifth of their original value by treatment of the material with boiling 1% sodium hydroxide solution for 6 hrs. under atmospheric pressure, and to about one tenth of the value by similar treatment at 20 lb./in.<sup>2</sup> excess pressure. The Methylene Blue absorption generally rises as a consequence of such treatment, the rise being connected in some way with the concurrent fall in copper number. The percentage loss of weight suffered by modified cottons after treatment for 4 hrs. with boiling 1% sodium hydroxide under atmospheric pressure is about six times the copper number when the latter does not exceed 2.5, irrespective of the manner of modification. For values exceeding 2.5, hydrocelluloses suffer a greater, and oxycelluloses a smaller, loss of weight than corresponds to this relation, the divergence increasing with increasing cellulose modification.

B. P. RIDGE.

**Photographic method of investigating the colour of light sources, and the reflecting power of coloured fabric and other surfaces.** P. W. CUNLIFFE and F. D. FARROW (*J. Text. Inst.*, 1927, 18, 291—302 T).—The method depends upon the production of equal densities on a photographic plate by exposures to a standard source of radiation (a gas-filled electric lamp) and to the lamp it is desired to investigate. Exposures are made for the same length of time, and the light falls on to the plate through an Ilford wedge screen and a spectral screen consisting of seven strips of Wratten gelatin filters, each passing a small range of wave-lengths. Attached to the spectral screen is a black slotted mask which allows light to pass through widths of 5 mm. of each filter. An exposure to the standard lamp is first made for one set of 5 mm. strips, and by a lateral movement of the mask these portions are covered and adjacent ones then exposed to the light from the unknown source, with the wedge reversed. The position of the point of equal density of the adjacent strips on the developed plate is found, from which, with a knowledge of the wedge constant, the relative intensities of any colour in the two exposures can be calculated. Undimmed lamps are found to be redder in the order gas-filled, vacuum, and carbon, the relative efficiency of the carbon lamp being low, and a marked loss of luminosity occurring when a gas-filled lamp is screened to give light of daylight quality. For the examination of the reflection from surfaces, a zinc white surface is taken as standard, and exposures are then made to light reflected first from the standard and then from the surface under investigation, the angle of incidence of light from a standard lamp to the surface being 45° and of reflection 0°.

Results of the examination of both printed papers and dyed cotton cloths show the colour expressed in the form of curves, fading in the case of some of the dyed fabrics being indicated by a general increase in reflecting power. Reproducible results are obtained with both Wratten and Ilford filter screens.

B. P. RIDGE.

**Relation between fluorescence and the reddening of sulphite cellulose. VIII. Chemistry of the sulphite-cellulose digestion process.** E. HÄGGLUND and T. JOHNSON (*Z. angew. Chem.*, 1927, 40, 1101—1104).—The reddening of sulphite cellulose is due to the presence of lignosulphonic acid, and if this acid is eliminated from the carbohydrate portion by hydrolysis, the remaining material is no longer turned red by oxidation. The fluorescence of sulphite cellulose and of sulphite waste-liquor also depends on the presence of lignosulphonic acid, and there is a close relationship between the two phenomena. Cellulose which shows the strongest fluorescence also shows, on oxidation, the deepest red coloration, whilst that which no longer becomes red shows no violet fluorescence. The same group of the lignin which is responsible for the reddening also produces the fluorescence. This group, or constitutional ingredient, is formed or liberated through the sulphonation of the lignin during the cooking process, and, by treatment with oxidising agents, by air oxidation, or the action of alkalis or dilute acids at raised temperatures, it is irreversibly changed so that it no longer produces the luminous fluorescence. The latter must be distinguished from the violet colour which results from the treatment of wood with acids or acid-reacting liquids, or which appears after the removal of the violet fluorescence of the sulphite cellulose or the lignosulphonic acid by oxidation. This violet colour is changed to a fluorescent greenish-yellow by the action of alkalis. Neither the violet colour nor the greenish-yellow fluorescence appears when the cellulose ceases to give the red colour. The results stated apply not only to pine wood, but also to Scotch fir and to the sulphite celluloses obtained from both.

B. P. RIDGE.

**Cellulose in the paper and artificial silk industries.** G. RAIMONDO (*Notiz. chim. ind.*, 1927, 2, 247—250).—Fir-wood cellulose, when boiled with 1% aqueous sodium hydroxide solution, reaches its maximum solubility, with loss of 18% by wt., in 2 hrs.; at the ordinary temperature in 16—20% sodium hydroxide solution, it lost 15.3% (maximal) in  $\frac{1}{2}$  hr. Digestion for 1 hr. with 18% sodium hydroxide solution should dissolve all the hemicellulose, even from straw and hemp. The following are values of the percentage dissolved by cold 18% and by hot 1% sodium hydroxide solution, respectively: German bleached, 15.1, 17.2; Czechoslovakian crude, 14.9, 17.7; Swedish crude, 15.8, 19.6; Norwegian bleached, 17.5, 19.2; Swedish bleached, 14.9, 17.3; poplar wood, 22.6, 22.5; esparto, 25.3, 21.2; straw, 38.5, 35.7; hemp stalk, 28.1, 23.8. The percentage soluble in boiling dilute sodium hydroxide solution is greater for soda or sulphite cellulose than for Italian (chlorine) cellulose.

CHEMICAL ABSTRACTS.

**Grinding of immature eucalypts for mechanical pulp, and possibilities of manufacturing newsprint in Australia.** L. R. BENJAMIN (Commonwealth of



Australia Council for Sci. and Ind. Res., 1927, Bull. No. 31, 37 pp.; cf. B., 1923, 923 A).—Mill tests show that eucalyptus chemical pulp when beaten sufficiently to ensure successful running on an ordinary paper machine gives a paper which, though of satisfactory strength, lacks the opacity, softness, and ink absorbency characteristic of high-grade newsprint. In view of these facts, attempts have been made to improve the finish of the paper by the addition of eucalyptus mechanical pulp. Small-scale grinding tests indicate that under carefully-controlled conditions of grinding the more important light-coloured eucalypts are capable of being converted into mechanical pulp of satisfactory colour and freeness. Laboratory papermaking trials showed that a furnish of eucalyptus sulphite pulp and eucalyptus mechanical pulp in the ratio 70:30, possibly with addition of a small quantity of long-fibred sulphite pulp, gave a paper somewhat stronger than newsprint, and closely resembling it in finish. No difficulty should be experienced in running such a paper on an ordinary paper-making machine, and estimated costs of production indicate that it should now be possible, especially with the assistance already proffered by the Australian Government, to establish a newsprint industry in Australia.

D. J. NORMAN.

**Testing bleaching quality of wood pulp.** H. L. JOACHIM (Zellstoff u. Papier, 1927, 7, 361—362).—For the rapid testing of bleaching quality an application of the permanganate method is described whereby results may be obtained in 20 min. and converted into terms of bleaching liquor by reference to a graph. The apparatus is a butter machine consisting of a thick glass jar of 2 litres capacity fitted with a mechanical stirrer with a curved glass rod substituted for the wooden paddle. A quantity of moist centrifuged pulp corresponding to 10 g. of air-dry fibre is stirred up with 1 litre of water, and the temperature is regulated in a constant-temperature bath to 37°, 25 c.c. of dilute sulphuric acid (1:4) are added, and then, without stopping the stirrer, 25 c.c. of *N*-permanganate solution are run in through the lid. The action of the permanganate is timed at exactly 7 min. at 37° with continuous stirring, and then by means of a burette inserted through the lid of the jar the excess of permanganate is discharged by *N*-oxalic acid added in portions of 0.5 c.c. at a time, at 30 sec. intervals until the end-point is approached. The correlation between bleach liquor and permanganate values is established by independent standardisation for different types of pulp.

J. F. BRIGGS.

**Application of vat colours.** WILSON. Use of indicator-dyed wool. KING.—See VI.

## PATENTS.

**Formation of colloid products.** S. A. NEIDICH (U.S.P. 1,643,080, 20.9.27. Appl., 3.3.26).—Artificial threads consisting of a solid core and an outer tubular sheath are obtained by extruding a colloid filament containing more than 8% of hydrated cellulose into a sulphuric acid bath the acidity of which is at least 50% greater than is normally employed. The duration of the acid treatment is such that a complete skin is formed on the exterior of the thread leaving a colloid core in contact therewith. Reversion and dehydration of this

colloid core are then brought about so that the core separates from the sheath to form an independent filament, whilst the outer sheath becomes shrivelled and thus capable of reflecting light with a scintillating effect.

D. J. NORMAN.

**Washing or emulsifying agents [for textile material].** C. A. AGTHE (E.P. 253,554, 14.6.26. Ger., 13.6.25).—A normal or acid salt of pyrophosphoric acid is used, preferably in conjunction with known washing and emulsifying agents, as a washing agent for woollen, silk, and other soft fabrics.

D. J. NORMAN.

**Machines for washing, milling, scouring, or otherwise treating fabrics with liquids.** I. MITCHELL (E.P. 276,154, 3.9.26).—Two, or a series of, pairs of nipping rollers, driven at successively slower speeds, are arranged with their faces almost in contact so that the fabric is accommodated in the substantially enclosed space formed between each pair of rollers and the next.

D. J. NORMAN.

**Washing or cleaning of wool.** E. C. DUHAMEL, and COMP. GÉN. DES IND. TEXTILES (E.P. 274,100, 3.5.26. Fr., 4.5.25. Addn. to E.P. 240,477).—The process of the main patent, dealing with the use of concentrated suint liquor for cleaning or reconditioning wool, is modified to eliminate the step of purifying the suint for re-use after treating wool in bulk or in the form of fleeces. This is effected by washing the wool, after a preliminary de-suinting operation if necessary, with sodium carbonate or soap solution. The wool is then treated with concentrated suint liquor on the counter-current principle, and is finally rinsed with water.

D. J. NORMAN.

**Treatment [finishing] of fabrics, paper, etc.** R. H. CHATHAM, ASSR. to CELANESE CORP. OF AMERICA (U.S.P. 1,640,596, 30.8.27. Appl., 18.7.21).—The material is padded with a solution of a cellulose ester or ether, e.g., cellulose acetate, in a mixture of phenol, formaldehyde, and ethyl alcohol, then immersed in water to precipitate the cellulose derivative, and dried.

T. S. WHEELER.

**Ornamentation of fabrics, leather, etc.** BRIT. BEAD PRINTERS, LTD., J. C. VREDENBERG, and F. A. H. HEYNERT (E.P. 277,091, 8.6. and 13.7.26).—Carbamide-aldehyde condensation products in the plastic intermediate condition are applied to fabrics etc. and are converted into the hard, final product after surface-decorating materials, e.g., glass particles, metal dust, pearl dust, mica flakes, have been introduced. Alternatively, the decorative materials may be incorporated with the plastic condensation product before its application to the fabric.

S. S. WOOLF.

**Manufacture of cellulose derivatives.** H. DREYFUS (E.P. 277,721, 26.3.26).—Cellulose is intimately mixed with alkali and an agent for removing water (calcium oxide) in presence of a diluent (benzene), and treated with ethylene oxide or chlorohydrin (2.5—5 mols. for each  $C_6H_{10}O_5$  taken) preferably at 0—15°. In place of the ethylene oxide or chlorohydrin there may be used halogen esters of glycols, glycerol, and other polyhydric alcohols, or of ethers of these, also the products of action of hydrogen chloride on formaldehyde and acetaldehyde. The amount of water present should



preferably be not more than half the weight of cellulose; the alkali should be at least equal to and preferably 3–19 times the weight of water. Cellulose derivatives containing one or more free hydroxyl groups may be used in place of cellulose.

C. HOLLINS.

**[Non-inflammable] cellulose acetate compositions.** S. J. CARROLL, Assr. to EASTMAN KODAK CO. (U.S.P. 1,641,411—3, 6.9.27. Appl., [A, B], 27.4.25; [C], 21.12.25).—Cellulose acetate is rendered non-inflammable by addition of (A) 1-bromonaphthalene (5–30%), (B) bromobenzene or a monobromotoluene (5–50%), or (C) *s*-tribromoaniline or a homologue (4–16%).

T. S. WHEELER.

**Treatment of cellulosic materials.** H. E. POTTS. From ARNOLD PRINT WORKS (E.P. 276,877, 21.1.27).—Cellulosic material, particularly cotton, is superficially modified, *e.g.*, linenised, by padding it with caustic soda solution of such strength (*d* 1.095 or less) that, under the conditions of time and temperature of the treatment, caustic soda is adsorbed without causing any substantial mercerising effect, and then subjecting it to the action of, *e.g.*, cuprammonium hydroxide solution of such strength that, during the limited time of treatment given, it would of itself have no appreciable action on the cellulose. No substantial quantity of caustic soda diffuses from the cellulose during the operation, and the stability of the cuprammonium solution is not, therefore, adversely affected.

D. J. NORMAN.

**Production of cellulose and paper from straw, esparto, reed, and similar raw materials.** E. L. RINMAN (E.P. 269,154, 23.3.27. Swed., 7.4.26).—Esparto, straw, etc. are digested with caustic soda either alone or in admixture with sodium sulphide at 100–140°. The resulting pulp is separated from the black liquor at a temperature not exceeding 50° and washed with water at about 30°, these operations being carried out on a rotary filter with addition, if necessary, of high-boiling petroleum oils to prevent the formation of scum. In this way the glutinous substances present in the raw material are retained with the cellulose, and are present in the paper made therefrom. The washed pulp gives on beating a close sheet, and is suitable for making greaseproof papers, kraft papers (optionally with addition of 10% of long-fibred pulp), newsprint (in admixture with about 75% of mechanical pulp), etc. When bleached, which operation must be conducted at a temperature below 30°, the pulp may replace bleached sulphite pulp in fine papers of all descriptions. If it is found necessary in the manufacture of heavy greaseproofs to heat the pulp suspension to cause it to run more freely on the machine the temperature should not exceed 40°.

D. J. NORMAN.

**Manufacture of paper.** H. A. SMITH, Assr. to HAMERSLEY MANUF. CO. (U.S.P. 1,644,451, 4.10.27. Appl., 15.8.25).—Paper containing hydrated cellulose (produced by beating) is impregnated with a solution of starch at 50–100°, pressed, and dried.

D. J. NORMAN.

**Treatment [separation of fibres] of paper pulp.** C. R. ROBINSON, Assr. to ROBINSON FIBRE CORP. (U.S.P. 1,644,447, 4.10.27. Appl., 6.2.25).—A solution of sodium

peroxide is added to the pulp under conditions permitting free reaction of the peroxide; when the mixture is alkaline to litmus it is agitated to complete the separation of the fibres at a temperature somewhat above normal, but insufficiently high to injure the fibres. W. G. CAREY.

**Rendering articles moulded from wood pulp waterproof.** J. J. H. STURMEY (E.P. 276,395, 19.4.26).—Containers and the like produced by the Drake process (*cf.* E.P. 115,587) are rendered waterproof by sizing the pulp in the beater, *e.g.*, with resin and aluminium sulphate, and subjecting the formed article, before removal from the mould, to a high temperature, *e.g.*, up to 400°, in order to fuse the sizing agent.

D. J. NORMAN.

**Production of artificial fibres, silk, hair, films, etc. from viscose.** E. C. R. MARKS. From KÖLN-ROTTWEIL A.-G. (E.P. 277,716, 16.3.26).—See F.P. 612,879; B., 1927, 387.

**Degreasing textile material.** G. B. ELLIS. From H. KOHNSTAMM & CO., INC. (E.P. 276,121, 22.7.26).—See U.S.P. 1,598,305; B., 1926, 945.

**Stencil sheet.** S. HORII (U.S.P. 1,645,141, 11.10.27. Appl., 18.5.26).—See E.P. 250,798; B., 1926, 534.

**Manufacture of strawboard.** M. E. PENNINGTON and A. B. DAVIS (U.S.P. 1,645,997, 18.10.27. Appl., 3.3.27).—See U.S.P. 1,625,090; B., 1927, 406.

**Dyeing of cellulose esters and ethers** (E.P. 258,611).—See VI.

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

**Recent advances in the application of [anthraquinone] vat colours.** J. WILSON (J. Soc. Dyers and Col., 1927, 43, 319–321).—Vat dyes may be applied to silk without deteriorating the fibre by the same methods as are used for dyeing cotton, and the resulting shades have excellent fastness, being unaffected by a degumming treatment (immersion in boiling soap for 8–12 hrs.). Vat dyes may be satisfactorily applied to wool from an ammonia-soda-hyposulphite vat such as is used in dyeing indigo. Dyeing is effected at 30–60°, the cold-dyeing colours (for cotton) yielding superior results when dyed on wool at 60°. An ordinary quality of wool is not deleteriously affected by immersion in solutions containing 4.5, 4.0, 3.5, 3.0, 2.5, 2.0, and 1.5 g./litre of caustic soda at 30°, 35°, 40°, 45°, 50°, 55°, and 60°, respectively; these quantities may be increased by 20% for a low-quality harsh wool, but should be decreased by 20% for a good-quality soft wool. Vat dyes may be applied to paper by adding the colour paste to the beater, followed by alum to promote exhaustion. The brightness of the resulting shade is proportional to the degree of fineness of the dye particles. For the colouring of oils and varnishes it is necessary to use vat dyes suspended in a fine state of division in non-aqueous medium such as linseed oil, turpentine, butyl alcohol, and ethyl acetate, since vat dyes after drying have but little tinctorial value when subsequently added to oils. The shade of a varnish coloured by the presence of a vat dye can be rendered lighter only by the addition of a white pigment and not by dilution of the varnish. A. J. HALL.

**Use of indicator-dyed wool to control textile**



**operations.** A. T. KING (J. Soc. Dyers and Col., 1927, 43, 321—324).—Wool dyed with sulphonephthalein indicators (e.g., Thymol Blue, Bromophenol Blue, Cresol Red, etc.), and also lacmoid, satisfactorily indicates the degree of acid or alkaline treatment to which it is subjected, although the sensitivity of these indicators when dyed on wool is much less than when used in solution. E.g., Thymol Blue in solution suffers a colour change with 0.25% sulphuric acid, whereas 4% sulphuric acid is required to effect the same change when the indicator is dyed on wool. The indicators are suitably dyed on wool from a bath acidified with 1% of acetic acid, 0.02% of Phenol Red (or other indicators in proportion to their mol. wt.) yielding sensitive shades; 0.8% of lacmoid is used. The indicators dyed on wool have reasonable fastness to light, but the fastness to alkalis and acids varies, and the indicator may be removed from the wool by prolonged immersion in an acid or alkaline liquor. Indicator-dyed wool has yielded especially useful results in detecting faults occurring during the drying, scouring, and carbonising of wool materials. A. J. HALL.

## PATENTS.

**Process for bleaching and like purposes.** C. B. THORNE (U.S.P. 1,642,978, 20.9.27. Appl., 6.12.24).—The disintegrated material is mixed with bleaching solution and fed into a bleaching tank of such design that, despite constant discharge during operation, the mass undergoing bleaching is not agitated.

D. J. NORMAN.

**Dyeing of wool and/or silk.** L. B. HOLLIDAY & Co., LTD., and C. SHAW (E.P. 277,833, 26.11.26).—Red-brown shades on wool, fixed by after-chroming or boiling with a solution of copper or iron sulphate, are obtained by dyeing from an acid bath with 2 : 5-dichloro- or 2 : 5-dibromo-*p*-benzoquinone. C. HOLLINS.

**Dyeing of cellulose esters and ethers.** I. G. FARBERIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 258,611, 17.9.26. Ger., 17.9.25).—Primary disazo dyes suitable for acetate silk are obtained by coupling a mono- or di-hydroxy-compound of the benzene or naphthalene series with 2 mols. of diazo compounds, provided that the final product contains at least one free or substituted amino-group, not more than one sulphonic group, and not more than one naphthalene nucleus. Examples are: sulphanilic acid → phenol ← *m*-phenylenediamine (yellow). Resorcinol gives orange to brown dyes. C. HOLLINS.

**Production of new fabrics [by effects on textile materials containing cellulose acetate].** BRITISH CELANESE, LTD., G. H. ELLIS, and R. J. MANN (E.P. 277,414, 15.6.26).—The cellulose acetate textile is treated locally (e.g., by printing) with an aliphatic carboxylic acid and then steamed. In some cases (succinic and citric acids) delustring is accelerated by the acid; in others (acetic, propionic, chloroacetic acids) it is retarded. Hydroxy-acids are particularly advantageous, and formic, acetic, propionic, butyric, chloroacetic, aminoacetic, glycollic, lactic, citric, and succinic acids are specially claimed. C. HOLLINS.

**Means for increasing the wetting and cleaning power of aqueous liquids used for treating fibrous**

**materials.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (E.P. 277,391, 10.3.26).—Sulphonic acids of polynuclear aromatic hydrocarbons, carrying as nuclear substituent a hydroaromatic radical with at least five carbon atoms, are used as wetting-out agents. In particular the sulphonated products of condensation of naphthalene with cyclohexanol or methylcyclohexanols in concentrated sulphuric acid are used. C. HOLLINS.

**Production of fast dyeings.** F. PETERHAUSER, Assr. to DURAND & HUGUENIN S.A. (U.S.P. 1,645,925, 18.10.27. Appl., 12.3.25. Ger., 20.3.24).—See E.P. 231,189; B., 1926, 785.

**Dyeing acidylcelluloses.** P. RABE and W. SCHEPSS, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,645,450—1, 11.10.27. Appl., [A], 3.3.24; [B], 10.12.24. Ger., [A] 28.5.23, [B] 29.5.23).—See E.P. 216,838; B., 1925, 627.

**Production of combined shades of azo and vat dyes on vegetable fibre.** I. G. FARBERIND. A.-G., Assees. of G. PETZOLD and H. RITTNER (U.S.P. 1,640,802—3, 30.8.27. Appl., [A], 15.12.24; [B], 24.6.25. Ger., [A] 29.1.24; [B] 1.2.24).—See [A] E.P. 228,510 and [B] E.P. 228,514; B., 1925, 844, 845.

**Stable preparations of vat dyes** (E.P. 259,999).  
**Development of azo dyes** (E.P. 249,526).—See IV.

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

**Composition of commercial chemical lime.** J. S. ROGERS (Ind. Eng. Chem., 1927, 19, 1157—1158).—Analyses of 38 samples of lime representative of those used in various industries are given, and the results tabulated. A maximum limit of 1% of silica is not unreasonable, and a ferric oxide content of less than 0.5% is easily secured, whilst alumina in 65% of the samples was less than 1%. The results show that a lime free from phosphorus is possible, and that manganese, though found in nearly all the samples, is generally present in minute amount, sufficient, however, to affect the colour of the lime, giving it a greyish tint. Sulphur (as SO<sub>3</sub>) varied from 0.04—3.33%, and may have been introduced or increased by burning the limestone with coal containing sulphur. The quicklimes varied from soft friable material to hard dense lumps, and in a few of the latter silica particles  $\frac{1}{8}$  in. in size were present. Some quicklimes hydrated immediately, whilst others required 1 hr. or more, and generally the slaked products were soft, fine powders with less variation in colour than the corresponding quicklimes. W. G. CAREY.

**Determination of potassium in mixtures of salts, especially in Chile saltpetre, by the perchlorate method.** G. LEIMBACH (Caliche, 1926, 8, 254—261).—For technical analysis, a single evaporation with hydrochloric acid (concentrated) and perchloric acid is sufficient. For more exact work the extraneous salts after the first evaporation are eliminated with alcohol (96%), and the residue is reprecipitated from water with perchloric acid; sulphates must be removed, but iodates do not interfere. In presence of boric acid an excess of perchloric acid is maintained.

CHEMICAL ABSTRACTS.



**Unusual impurity in sodium bromide.** V. ZANOTTI (Boll. Chim. farm., 1927, 66, 513—514).—Attention is drawn to the presence of a phosphite as impurity in sodium bromide intended for medical use. The presence of sulphate is also detected. M. CARLTON.

**Molten ammonium chloride.** W. MERTNER (Chem.-Ztg., 1927, 51, 638).—When ammonium chloride is heated at about 400° in a sealed tube it melts to a clear mobile liquid, which, on cooling, solidifies to a hard glassy mass similar in appearance to commercial "sublimed sal-ammoniac." This suggests that in the commercial method of making this salt true sublimation does not occur, but that, owing to the pressure inside the sublimation vessel, the ammonium chloride melts and then distils unchanged. A. R. POWELL.

**Mechanism of the preparation of "sublimed sal-ammoniac."** Molten ammonium chloride. W. SIEGEL (Chem.-Ztg., 1927, 51, 759).—Polemical against Mertner (cf. preceding abstract). The pressure and temperature inside a sublimation vessel are much too low to cause any part of the substance to melt; the greater output of sublimed salt obtained by working with pressed tablets of crude salt is due to the greater surface obtained and to the greater ease with which the vapours can escape from the mass. Apparently the first sublimate consists of loose fibrous crystals, which, under suitable conditions of temperature and pressure, become coarser in texture and denser. A. R. POWELL.

**Determination of the neutralising value of acid calcium phosphate [in baking powder].** C. W. HERD (Cereal Chem., 1927, 4, 347—369).—The amount of alkali required for the neutralisation of acid calcium phosphate depends on the conditions of the experiment, owing to the fact that there is an equilibrium in solution between the mono-, di-, and tri-salts. The mono-acid salt has no neutralising power in the cold, but on heating equilibrium is gradually reached, so that the hot solution has a distinctly acid reaction. Direct titration of acid calcium phosphate with sodium hydroxide both in hot and cold solutions is unsatisfactory, as the end-point is dependent on the rate of addition of the alkali. The back-titration method involving addition of excess of alkali and titration of this excess with acid gives results in agreement with those obtained electrometrically, provided that the mixture is boiled until equilibrium is reached (about 10 min.), and that Thymol Blue or not more than 0.1 c.c. of a 0.2% solution of phenolphthalein is used as indicator. This method gives a value which is characteristic of the particular phosphate powder, but is too high for the commercial practice of the preparation of acid phosphate-bicarbonate mixtures for self-raising flour, since the mixtures based on this titration would be alkaline. Bailey's method of determining the  $p_H$  value of the actual baking ingredients may be used for checking the correct mixing of a raising powder, and can be used to correlate the above titration value with the most satisfactory acid phosphate-sodium bicarbonate ratio for baking practice. F. R. ENNOS.

**Influence of form and proportion of lime used and of method of mixing on the resulting Bordeaux mixture.** E. B. HOLLAND, C. O. DUNBAR, and G. M. GILLIGAN (J. Agric. Res., 1927, 34, 677—686).—The use

of different forms of calcium hydroxide in preparing Bordeaux mixture has been studied. Judging by the ratio of the volume occupied by the precipitate to the total volume of the liquid after 1, 2, and 3 hrs., it is concluded that, with chemically-precipitated calcium hydroxide, the best results are obtained if the equivalent of 1.5 lb. of calcium oxide are used with 4 lb. of copper sulphate and 50 gals. of water. Corresponding figures are 2.0—2.25 lb. for quicklime (made into milk of lime), and 2.0 lb. for commercial hydrated lime. The best method of mixing, similarly judged, is by pouring dilute copper sulphate solution into concentrated milk of lime, or the two simultaneously into a third receptacle. C. T. GIMINGHAM.

**Dehydration of hydrated salts. IV.** M. A. RAKUZIN and D. A. BRODSKI (Z. angew. Chem., 1927, 40, 836—840; cf. B., 1926, 1011; 1927, 187).—When borax (undamaged crystals) is heated in an oven at 98° it is rapidly converted into the pentahydrate. The decahydrates of sodium carbonate and sulphate are completely dehydrated when treated in the same way, whereas the heptahydrate of magnesium sulphate loses only 4.5 mols. of water in 3 hrs.; of zinc sulphate, 6 mols. in 8 hrs.; of nickel sulphate, 4.5 mols. in 5 hrs.; and of cobalt sulphate, 6 mols. in 3 hrs. The pentahydrate of copper sulphate loses 4 mols. of water in 5 hrs., and potash alum 15 mols. in 2 hrs. A stream of dry air at 98° likewise completely dehydrates sodium carbonate, and removes 6 mols. from cobalt sulphate; it also brings about further dehydration of alum, 19 mols. in all being removed. Boiling ethyl alcohol removes 6 mols. from borax and from zinc sulphate, 8½ mols. from sodium carbonate, completely dehydrates sodium sulphate, removes only 2 mols. from magnesium sulphate, 2½ mols. from nickel sulphate, 2 mols. from cobalt sulphate, practically none from copper sulphate, and only 10 mols. from alum. The dehydration of borax by boiling alcohol depends on the strength of the latter. The products of dehydration by heating in air are crusty masses, which would need grinding before marketing. The various processes of dehydration are compared with one another from the economic point of view. W. A. SILVESTER.

**Danger of explosion in the manufacture of bleaching liquor from liquid chlorine.** E. SCHÖNBERG (Papier-Fabr., 1927, 25, 581).—An explosion in an asphalted cast-iron tower in which a stream of milk of lime was treated with a counter-current of chlorine is attributed to the decomposition of calcium hypochlorite formed through supercooling of the reaction mixture. Calcium hypochlorite is unstable and, on warming, decomposes with the formation of chlorate and chloride, a reaction which may be completely without danger. In the presence of catalysts (e.g., phosphorus, arsenic, sulphur, iron oxide) or of light, however, the reaction  $\text{Ca}(\text{OCl})_2 \rightarrow \text{CaCl}_2 + \text{O}_2$  takes place with explosive violence. If supercooling of the reaction mixture is avoided by ensuring complete vaporisation of the liquid chlorine before it comes into contact with the milk of lime, no danger of explosion exists. B. P. RIDGE.

**Simple method for determination of active chlorine [in bleach liquor].** J. HAUSNER (Zellstoff u.



Papier, 1927, 7, 363–364).—Although the use of sodium indigo sulphate as a quantitative measure of active chlorine in bleach liquor is not to be recommended, it may be employed to advantage as an indicator when mixed with the thiosulphate to a standard solution. For works' use the titration is carried out in a graduated stoppered cylinder having a zero mark near the bottom to indicate a known volume of bleach liquor; a drop of acetic acid is added to this liquor, and the blue-tinted thiosulphate solution is introduced gradually until the colour is no longer discharged. J. F. BRIGGS.

**Ammonia as a source of nitrogen oxides for chamber acid plants.** D. H. KILLEFFER (Ind. Eng. Chem., 1927, 19, 1153–1156).—A description of American types of converter for the oxidation of ammonia in this connexion. Anhydrous ammonia is there used as frequently as aqueous ammonia as raw material. It is stored in tanks of 1½-in. hammered steel, and vaporised by steam or by waste heat in a double-pipe heater, and then passes through an expansion valve to an air mixer. The heat-exchanger piping conveying the ammonia-air mixture is of aluminium, as iron has been found to catalyse the oxidation of ammonia to nitrogen under these conditions. C. IRWIN.

**Behaviour of carbon and sulphur at high temperatures; [solid] carbon sulphides.** J. P. WIBAUT (Z. angew. Chem., 1927, 40, 1136–1137).—By the interaction of sulphur with amorphous or activated carbon at high temperatures, or by heating tetraiodothiophen, a solid heat-resistant carbon sulphide is obtained. This contains (approx.) 5% S, and is not an adsorption product, but its stoichiometric composition is not yet established. The preparation of this and other solid sulphides of carbon is described. S. S. WOOLF.

**Determination of carbon disulphide in its emulsions.** H. J. FISHER (Ind. Eng. Chem., 1927, 19, 1201–1202).—2 g. of emulsion are added to a tared glass-stoppered 100 c.c. flask containing 40 c.c. of 8% alcoholic potassium hydroxide. The solution is made up to 100 c.c. with 95% alcohol, and 5 c.c. are added to a mixture of 50 c.c. of water, 50 c.c. of 3% hydrogen peroxide, and 10 c.c. of 10% potassium hydroxide. It is heated for 1 hr., acidified, and the sulphur weighed as sulphate. Results given are about 96% of the theoretical assuming the purity of the carbon disulphide used. C. IRWIN.

**Determination of sulphur trioxide in the presence of sulphur dioxide, together with some analyses of commercial liquid sulphur dioxide.** J. R. ECKMAN (Sci. Papers U.S. Bur. Stand. No. 554, 1927, 22, 277–285).—A nearly saturated and slightly acid solution of barium sulphite is used for precipitating the trioxide; the solution must be made and kept and the operations carried out in an oxygen-free atmosphere. Apparatus for carrying out the determination is described. Eight samples of commercial liquid sulphur dioxide were examined by this method; in all cases the gas obtained from the liquid was found to be free from sulphur trioxide, and only in three cases was the trioxide found in the residues. S. I. LEVY.

**Corrosive sublimate as wood preservative.** MOLL.—See IX.

**Steels for ammonia synthesis.** VANICK.—See X.

**Absorption of nitrogen oxides by phosphate rock.** MORRIS.—See XVI.

**Ammonia by fermentation.** WOODRUFF.—See XVIII.

**Size of silver bromide particles.** LOVELAND and TRIVELLI.—See XXI.

#### PATENTS.

**Manufacture of sulphuric acid.** Y. LE MONIÈS DE SAGAZAN (F.P. 618,560, 12.11.25).—Gases from pyrites or sulphur burners pass through apparatus comprising three sections in which they are brought in contact successively with nitrosulphuric acid and dilute sulphuric acid, with water or dilute sulphuric acid, and with sulphuric acid, *d* 1.71, for the recovery of oxides of nitrogen. L. A. COLES.

**Production of concentrated nitric acid.** C. UEBEL (G.P. 442,037, 12.1.26).—In a cyclic process for producing the acid by treating ammonium nitrate with concentrated sulphuric acid, the residual ammonium bisulphate, which may contain some nitric acid and water, is re-converted into ammonium nitrate by neutralisation with aluminium hydroxide with the addition of potassium nitrate in such quantity that potassium alum mixed with two equivalents of potassium sulphate crystallises out on cooling the solution. The crystalline product is treated with ammonia to recover aluminium hydroxide, potassium ammonium sulphate also being formed. Alternatively, the ammonium bisulphate is neutralised with ammonia in the presence of an equivalent weight of aluminium nitrate, whereby ammonium nitrate, aluminium hydroxide, and ammonium alum are formed. A further recovery process is described using manganic hydroxide or hydrated manganese dioxide, aluminium nitrate, and potassium nitrate. L. A. COLES.

**Production of volatile acids, acid anhydrides, and chlorine.** H. MEHNER (G.P. 442,166, 11.4.22).—Salts and other compounds destined for thermal decomposition, with or without the addition of steam, are sprayed (fused or in solution) through the hearth of a reverberatory furnace, and the heating gases are introduced elsewhere. The steam may be formed by the combustion of hydrogen or water-gas. The hearth is constructed of cement clinker for the decomposition of sulphates, and of loose heaps of fluorspar plaster for the manufacture of hydrogen fluoride. L. A. COLES.

**Utilisation of waste acid liquors in chemical processes.** C. A. KLEIN and R. S. BROWN (E.P. 277,769, 8.7.26).—Sulphuric acid obtained, for example, in the manufacture of titanium pigments (cf. E.P. 243,081; B., 1926, 99) is decolorised with animal charcoal, sulphur dioxide, or a small amount of a barium salt to carry down the colour with the resulting precipitate, and is then treated with barium oxide or carbonate to precipitate the acid as *blanc fixe* without precipitation of iron. A small quantity of a weak acid, e.g., hydrochloric, nitric, or acetic acid, may be added to attack the oxide or carbonate before it acts as the precipitating agent. W. G. CAREY.



**Production of commercial lumpy ammonium carbonate.** I. G. FARBENIND. A.-G. (G.P. 442,176, 17.3.25).—Ammonium bicarbonate, alone or mixed with ammonium carbamate or with ammonium carbonate, or a mixture of ammonia, carbon dioxide, and water in the correct proportions, is sublimed or heated under pressure.

L. A. COLES.

**Hydration of lime.** H. DITTLINGER, Assr. to DITTLINGER CROW PROCESS Co. (U.S.P. 1,640,602, 30.8.27. Appl., 5.11.23).—Calcium hydroxide of increased plasticity is obtained by slaking calcium oxide with a dilute solution of potassium permanganate, sodium chloride, or similar compound.

T. S. WHEELER.

**Treatment of crude nitrate of soda.** I. G. FARBENIND. A.-G. (E.P. 266,744, 25.2.27. Ger., 25.2.26).—Oxides of nitrogen are obtained from crude sodium nitrate too low in nitrate to be economically worked, by heating it to red heat preferably in a current of air or other gas. The process is accelerated if gas which facilitates the decomposition of the nitrate is used, or if a small quantity of a non-volatile acid solid, *e.g.*, acid silicate, is added to expel the more volatile acids.

W. G. CAREY.

**Simultaneous manufacture of iron phosphide and fused cement.** W. KYBER (E.P. 267,518, 8.3.27. Ger., 10.3.26).—Sufficient iron ore or scrap iron is added to a reduction melt or slag containing phosphates to combine with all the phosphorus, with the further addition of alumina, calcium oxide, and/or silica, the amounts depending upon the quantity already present and the required composition of the fused cement. Aluminium phosphate may be used as the primary material, in which case alumina is not added.

W. G. CAREY.

**Production of zinc carbonate.** N. A. LAURY (U.S.P. 1,640,708, 30.8.27. Appl., 23.12.25).—Roasted zinc ore is leached with hot ammonium sulphate solution, zinc carbonate being recovered by cooling and saturating the resulting clear liquor with carbon dioxide.

T. S. WHEELER.

**[Production of sulphur compounds from ores containing] sulphur.** M. F. COOLBAUGH and J. B. READ (Can. P. 264,357, 30.3.25).—The ore is roasted in air and the gases obtained, which contain sulphur dioxide, sulphur trioxide, and excess oxygen, are brought in contact with a chloride. Alternatively, a chloride is mixed with the ore before roasting.

L. A. COLES.

**Production of synthetic ammonia.** OMNIUM DES IND. CHIM. (PROC. TOCCO & LANDI), Assees. of L. TOCCO and M. LANDI (E.P. 262,090, 16.11.26. Fr., 27.11.25).—See Addn. to F.P. 607,059; B., 1927, 814.

**Production of ammonia.** OMNIUM DES IND. CHIM. (PROC. TOCCO & LANDI) (E.P. 272,842, 17.11.26. Fr., 15.6.26).—See F.P. 607,059; B., 1927, 218.

**Nitrating mixture** (U.S.P. 1,640,737).—See XX.

**Shadow producing agent** (U.S.P. 1,644,446).—See XXI.

## VIII.—GLASS; CERAMICS.

**Properties of soda-lead oxide-silica glasses.** S. ENGLISH, W. E. S. TURNER, and F. WINKS (J. Soc. Glass Tech., 1927, 11, 300—303).—In a series of glasses

of the molecular type  $6\text{SiO}_2, x\text{PbO}, (2-x)\text{Na}_2\text{O}$  and ranging from 72.86%  $\text{SiO}_2$ , 3.75%  $\text{PbO}$ , 22.62%  $\text{Na}_2\text{O}$  to 59.34%  $\text{SiO}_2$ , 27.77%  $\text{PbO}$ , 12.31%  $\text{Na}_2\text{O}$ , the annealing temperature decreased continuously from 478° to 446°, and the linear thermal expansion coefficient (25° to 90°) from  $1090 \times 10^{-8}$  to  $842 \times 10^{-8}$ , whilst the density at 25° increased from 2.5045 to 2.9960. From the results the coefficient of cubical expansion for lead oxide in glass was determined as  $3.18 \times 10^{-7}$ .

A. COUSEN.

**Brittleness of opal glass.** G. GEHLHOFF and M. THOMAS (J. Soc. Glass Tech., 1927, 11, 347—358).—The brittleness of fluoride opal bulbs, as measured by impact strength, was found to be a function of wall thickness. With decreasing temperatures of working-out the glass, a rapid decrease of strength occurred over the range 1040—1030°, indicating 1050° as the limiting working temperature for the glass investigated. Confirmation was obtained by thermal resistivity tests involving the rapid chilling of spheres worked-out at varying temperatures. The glass showed a distinct heat absorption when, on heating, a temperature of 1050° was reached. Samples kept for some time at successively increasing temperatures and then rapidly chilled became clear and transparent at above 1050°, but opaque below this point. With three opal glasses of varying composition the critical zone occurred at points of approximately equal viscosity.

A. COUSEN.

**Electrical conductivity of glasses at high temperatures.** F. F. S. BRYSON (J. Soc. Glass Tech., 1927, 11, 331—346).—Conductivities were determined over a temperature range from the softening point of the glass to 1150°, by means of a high-frequency alternating current. In a series of soda-silica glasses the conductivity increased as soda substituted silica, and the greater the soda content the greater the increase in conductivity for a given rise of temperature. With soda-magnesia-silica glasses the conductivity increased as magnesia replaced silica. Soda-alumina-silica glasses progressively decreased in conductivity as the alumina replaced soda. With an apparatus devised for obtaining continuous records of conductivity of glass as it flowed from a tank furnace, variations were obtained which were in agreement with the limited variations of viscosity occurring under the system of control used in the works.

A. COUSEN.

**Noxious properties of [vitreous] enamels containing antimony.** F. FLURY (Z. angew. Chem., 1927, 40, 1134—1136).—The effect of introducing various antimony salts into the food of rats, dogs, etc. is studied with a view of determining the desirability or otherwise of using enamelled cooking utensils containing antimony oxide or other compounds of antimony. The results are inconclusive, but the use of such articles is considered dangerous.

S. S. WOOLF.

**Analysis of refractory materials.** I. H. J. VAN ROYEN (Stahl u. Eisen, 1927, 47, 1696—1697).—Silica in quartzite and silica brick is determined by loss in weight after the usual hydrofluoric acid treatment; the residue is dissolved in fused pyrosulphate and the iron determined by titration with titanous chloride. In a separate portion, also treated with hydrofluoric acid, the titanium and aluminium are precipitated together as



phosphate and weighed; the precipitate is dissolved in fused pyrosulphate and the titanium determined colorimetrically, the amount so found multiplied by 3.15 being subtracted from the weight of mixed phosphates to obtain the weight of aluminium phosphate. Manganese is determined by the Volhard method, and lime and magnesia as usual. The analysis of clays and grog is carried out in a similar manner, except that the sample is decomposed by fusion with alkali carbonate and the mixed aluminium and titanium phosphates obtained are redissolved in hydrochloric acid and reprecipitated to remove adsorbed sodium salts. Chromium should be tested for and, if found, the corresponding weight of chromium phosphate is deducted from the weight of combined phosphates. To determine titanium gravimetrically in this precipitate it is fused with a large excess of sodium carbonate, the mass leached with water, the insoluble sodium titanate collected and dissolved in hydrochloric acid, and the titanium precipitated by Gooch's acetate method. A. R. POWELL.

## PATENTS.

**Manufacture of a highly refractory material.** M. L. FREED, Assr. to U.S.A. (U.S.P. 1,644,244, 4.10.27. Appl., 10.6.26).—A mixture containing anhydrous aluminium silicate and topaz is calcined to convert it into mullite; the product is moulded with a temporary binder, and fired at a temperature sufficient to convert it into a homogeneous mass. W. G. CAREY.

**Manufacture of magnesite refractories.** U.S. METALS REFINING CO., Asses. of A. MARKS (E.P. 271,847, 6.5.27. U.S., 6.5.26).—See U.S.P. 1,616,192; B., 1927, 189.

**Drawing glass tubes or rods.** S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (E.P. 277,807, 30.9.26).

## IX.—BUILDING MATERIALS.

**Plastic magnesia [magnesium oxychloride] cements.** L. G. STEWART (Ind. Eng. Chem., 1927, 19, 1139—1143).—Magnesium oxide for use with magnesium chloride as magnesium oxychloride cements is prepared by the calcination of magnesite or of precipitated magnesium hydroxide or carbonate; the product should not be "dead burnt," but must remain plastic and be very finely ground. Combined and adsorbed moisture should be below 5% otherwise the strength and setting time of the cement are adversely affected; free lime and calcium silicate are detrimental. Commercial magnesium chloride is used, and typical analyses of this and of various samples of plastic magnesia are given. Cement made from these materials with the addition of ground aggregate, sand, and asbestos are tested for strength, setting time, volume change, and also for weather- or water-resistance. The cement is used for building purposes on account of its high early strength, elasticity, resilience, thermal insulation, and fireproof qualities.

W. G. CAREY.

**Use of plaster of Paris as an impression material.** W. H. SODEAU and C. S. GIBSON (Brit. Dental J., 1927, reprint, 27 pp.).—Whilst dental plaster consists mainly of the hemihydrate, the material is a complex and variable mixture, and the authors' conclusions are limited to certain types actually examined. The expansion on setting is about 0.3%, but is increased up to 1%

by stirring after the mixture has appreciably stiffened or by addition of previously hydrated plaster. Expansion can, however, be reduced to a negligible amount by the use of a potassium salt as an accelerator, an alkaline substance such as borax being employed if necessary to prevent too rapid setting. The chloride, nitrate, or sulphate of potassium may be employed, the last in conjunction with borax being very suitable; it is convenient to add an appropriate dyestuff, such as Alizarin S. Data are given of tensile strengths developed by various mixtures under different conditions. S. I. LEVY.

**Importance of corrosive sublimate as a wood-impregnating material.** F. MOLL (Z. angew. Chem., 1927, 40, 1137—1140).—Mercuric chloride and aniline or sodium derivatives of dinitrophenols are superior to copper sulphate and zinc chloride as wood preservatives, both as regards penetration and fungicidal effectiveness. The supposed advantages of mixing sodium fluoride or other salts with mercuric chloride are considered, previous work in this field being summarised. In the fixation of mercuric chloride, the active product is usually mercury oxychloride and not calomel. The pre-eminence of corrosive sublimate as a wood preservative is supported by long-period practical tests. S. S. WOOLF.

**Commercial lime.** ROGERS.—See VII.

## PATENTS.

**Production of coloured effects in building materials.** G. N. WHITE (E.P. 277,389, 8.3., 26.7., and 18.8.26).—Vat dyes (and other coloured compounds capable of being vatted) in the form of dry powder, aqueous suspension, or colloidal solution, preferably after dispersion by oxidation of the leuco-compound in presence of a dispersing agent, are incorporated with building materials (cement, plaster, asbestos, lime, etc.), either by mixing dry, or in gauging water, or by subsequent impregnation. C. HOLLINS.

**Manufacture of porous siliceous objects [from clay etc.].** F. C. and F. E. KERN (E.P. 262,826, 14.12.26. U.S., 14.12.25).—Wood or other cellular or fibrous material is heated for 10 min. at approximately 325° until its resilience is destroyed and ingredients more volatile than ketones are volatilised. The product (125 pts. by wt.) is mixed with 100 pts. of clay, rendered plastic by the addition of water, formed into shapes, dried, and burnt. Reactive agents such as light porous or colloidal siliceous substances may be added as a flux or to make the mixture more plastic.

W. G. CAREY.

**Manufacture of a fibrous compound [for building purposes].** E. M. LUNDGREN (E.P. 276,709, 1.5.26).—Granulated cork, peat, or other vegetable material is artificially petrified by successive treatment with (i) calcium chloride solution and sodium silicate solution, with or without addition of lime water, (ii) calcium chloride solution and aluminium sulphate solution, or (iii) an aqueous suspension of calcium bicarbonate and lime water. The resulting products may be used as insulating or building materials. D. J. NORMAN.

**Paper bags for containing cement, lime, etc.** W. ERNST and E. LUH (MITTELBADISCHE PAPIER-MANUF. ERNST & LUH (E.P. 278,275—6, 2.5.27. Ger., 7.12.26).

**Fused cement** (E.P. 267,518).—See VII.



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

**Stainless iron and its application to chemical plant construction.** W. M. MITCHELL (Ind. Eng. Chem., 1927, 19, 1171—1175).—The true stainless steels containing 11–13% Cr and 0.3–0.4% C which require heat treatment are mainly suitable for valve seats, the wearing parts of crushers, etc., being too hard for most purposes. Mild stainless steel (11–14.5% Cr with under 0.12% C) can be obtained in all commercial forms and may be used for tanks and autoclaves. It is very suitable for valve stems, pump rods, and shafts. True stainless iron (above 16% Cr and below 0.1% C) is better suited for general purposes of fabrication. Its strength is not so great as that of the mild steels, but it is more resistant to corrosion and free from hardening tendency. Up to 0.1% Si is advantageous especially for resistance to nitric acid. Nickel-iron-chromium steels are more costly, but have the advantage of being easily welded. Chromium steel containing 25–30% Cr is useful when resistance to oxidation at very high temperatures is required. The high-carbon type is only available as castings. The proportion of chromium necessary to confer effective resistance to nitric acid is about 14%. At above 16% Cr the action of halide acids is slight. Sulphuric acid attacks all the alloys unless sufficient ferric sulphate is present in solution to inhibit the action, but "mixed acid" does not attack high-chromium alloys. Difficulties arise in both welding and rivetting except with nickel-iron-chromium alloys. Channels, angles, and seamless-drawn tubing can be successfully made of stainless iron. Sound castings are only readily obtained with high carbon content.

C. IRWIN.

**Surface decarburisation of carbon steels.** E. H. SCHULZ and W. HÜLSBRUCH (Arch. Eisenhüttenwesen, 1927, 1, 225–240; Stahl u. Eisen, 1927, 47, 1694–1695).—The decarburising action of various atmospheres on three steels containing 0.5, 0.9, and 1.3% C has been determined at 750–1050°. In all gases, except pure nitrogen and carburising gases, such as coal gas, methane, or carbon monoxide, severe decarburisation takes place at 900° or above. Pure nitrogen and methane-hydrogen mixtures are inert, but even traces of carbon dioxide or steam in these gases causes severe decarburisation to occur. A similar action ensues if very small quantities of steam or carbon dioxide are present in gases which, if pure, act as carburising agents. In hydrogen or in a technical "reducing atmosphere," decarburisation commences at 550–600° and in steam, carbon dioxide, or air at 800°; in pure oxygen or perfectly dry air no action takes place below 850°. The depth of decarburisation in a given time increases in all cases with rise of temperature and, above 850°, with increase in the carbon content. The ferrite crystallites formed on the surface by decarburisation above 750° are extraordinarily coarse and columnar in shape, the long edges being perpendicular to the surface of the metal. In cases where a coating of scale is formed, this acts as a protective layer below 850°; the heaviest layer of scale is formed in a salt bath and only slightly thinner layers in oxygen or steam.

A. R. POWELL.

**Report on metallic materials for electrical heating.** [Analysis of nickel-chromium and nickel-chromium-iron alloys.] D. HARVEY (Amer. Soc. Testing Materials, 1927, No. 20. Advance copy. 22 pp.).—In the determination of nickel with dimethylglyoxime, the residue insoluble in aqua regia, after removal of silica as silicon tetrafluoride, is fused with sodium carbonate, and a solution of the mass in hydrochloric acid is added to the principal solution. A 3% solution of dimethylglyoxime in 3% aqueous ammonia is used as the precipitant, the precipitate being dissolved in sulphuric acid and the nickel determined electrolytically. Chromium is determined by the persulphate method (permanganate being reduced by heating with sodium chloride in sulphuric acid solution), and iron by the cupferron method. For the determination of manganese the sample is dissolved in aqua regia and the solution evaporated with concentrated nitric acid. The manganese dioxide is precipitated with potassium chlorate, and dissolved in acidified ferrous sulphate solution, the excess of which is titrated with permanganate. Carbon is determined by Cain's method. The "insoluble residue" is obtained by treating the alloy (5–10 g.) with concentrated hydrochloric acid (200–400 c.c.) and nitric acid (1–2 c.c.) and removing silica as silicon tetrafluoride. The determination of silicon is conventional.

CHEMICAL ABSTRACTS.

**Causes of failure in heat-resisting alloys.** R. SUTTON (Trans. Amer. Soc. Steel Treat., 1927, 12, 221–234).—The lives of five case-hardening boxes of the 60% Ni, 20% Cr, 15% Fe type of alloy varied from 75 to over 7500 hrs. Four of the boxes containing aluminium were free from heavy scale. The principal reasons for failure were found to be strains caused by non-uniform castings, the presence of slag and other impurities in the alloy, also occluded gases caused by incomplete deoxidation. The best box consisted of uniformly distributed austenite and cementite, was non-magnetic, and had the lowest ball-hardness. It was poured at a low temperature and cooled slowly.

T. H. BURNHAM.

**Magnesium and its alloys.** J. A. GANN and A. W. WINSTON (Ind. Eng. Chem., 1927, 19, 1193–1201).—Commercial magnesium prepared by electrolysis of the dehydrated chloride contains above 99.9% Mg. It forms numerous compounds with other metals, e.g., silver, aluminium, copper, nickel, lead, tin, and zinc, with, in most cases, solid solutions as well; such alloys are hard and brittle. Their properties are usually improved by the addition of a third metal, e.g., aluminium to magnesium-copper alloy. Manganese is introduced as a corrosion inhibitor. Particulars of the composition of four such commercial alloys intended for castings and forgings are given with their physical characteristics. They have  $d$  1.74–1.84, and weight for weight are in most respects far superior to iron and steel. Metallographic specimens are etched with 2% nitric acid solution. The presence of aluminium and cadmium breaks up the magnesium-copper eutectic and gives a fine-grained structure. The metal is melted with a flux in cast steel pots, the flux covering the metal and protecting it. A mixture of magnesium chloride with sodium or potassium chloride is used, the density



of which is equal to that of the metal, so that the latter when melted floats as a ball within it. Magnesium oxide, sand, and dirt sink to the bottom. Oxidation of the metal during pouring is prevented by burning sulphur around it. Heat-treatment of certain cast alloys and before forging is desirable. The alloys can be extruded, hot-rolled, or machined. The corrosion which formerly occurred with magnesium and its alloys was mainly due to spongy metal and impure spots. Manganese is an inhibitor. Paint, varnish, or lacquer may be applied after a preliminary treatment with a hot acid phosphate solution. C. IRWIN.

**Scleron metal.** ANON. (Mitt. Materialprüf., 1927, 5, 106—107; cf. B., 1927, 168).—A light aluminium alloy called scleron metal consists of 0.35% Si, 3.14% Cu, 0.49% Fe, 0.72% Mn, 13.20% Zn, 0.077% Li, and remainder aluminium. The properties of forged bars 39 mm. in diameter, after age-hardening, are  $d$  3.0099, elastic modulus 725,000 kg./cm.<sup>2</sup>, breaking strength 4890 kg./cm.<sup>2</sup>, elongation 11.1%, and reduction in area 14.5%. Its resistance to corrosion by salt water equals that of pure aluminium. It is noticeably free from segregation and internal oxidation, and should be suitable for constructional purposes. C. J. SMITHELLS.

**Aluminium alloy, alneon, for castings.** M. VON SCHWARZ (Z. Metallk., 1927, 19, 390—395).—The name alneon is applied to zinc-aluminium alloys containing 10—20% Zn and small proportions of nickel and copper. The alloys have  $d$  2.8—3.2, a Brinell hardness of 100—150, an elastic limit of 6.9—7.4 kg./mm.<sup>2</sup>, a tensile strength of 20—34 kg./mm.<sup>2</sup>, an elongation of 0.1—4%, and a high resistance to repeated impacts. The alloys are particularly suitable for the production of castings, owing to their small contraction on solidification and to the absence of any tendency to segregation. Like all aluminium alloys of this type alneon exhibits age-hardening properties, the maximum effect being obtained in 2—3 weeks at the ordinary temperature with alloys containing 80—87% Al, and in 2—3 months in alloys containing 88—90% Al. A. R. POWELL.

**Tarnish-resisting silver alloys.** L. JORDAN, L. H. GRENNELL, and H. K. HERSCHMAN (Tech. Papers U.S. Bur. Stand. No. 348, 1927, 21, 459—496).—The effect on the rate of tarnishing of silver in sodium polysulphide solutions of additions of the following elements singly or in pairs has been determined; aluminium, cadmium, copper, magnesium, zinc, silicon, tin, bismuth, antimony, tellurium, manganese, nickel, molybdenum, titanium, chromium, germanium, beryllium, gold, and sodium. The most resistant alloys were those containing zinc or cadmium, but their tensile strength was low, although they could readily be rolled into sheet. Addition of antimony or tin or of both to either zinc-silver or cadmium-silver alloys increased appreciably their strength, and slightly improved their resistance to tarnishing. The most satisfactory sterling silver alloys from the point of view of resistance to tarnishing, workability, and strength contained 4.5% Zn, 2% Sb, and 1% Sn, and had a tensile strength, after rolling into sheets, of 37,000 lb./in.<sup>2</sup> Addition of gold to any of the alloys did not improve the resistance to tarnishing, but the tensile strength was increased. Alloys containing 15—

20% Zn or Cd and 20% Au were highly resistant to corrosion, and of equal strength and hardness to standard silver alloys containing only copper. A. R. POWELL.

**Preparation of alloy single crystals.** H. SENG (Mitt. Materialprüf., 1927, 5, 105—106).—A modification of Bridgman's method is described in which the molten alloy is sucked up from a crucible into a tube, tapered at the lower end and surrounded by a second tube to shield it from draughts. The whole is enclosed in a furnace which controls the temperature. The tube containing the alloy is lowered through the bottom of the furnace at the rate of 30 cm. in 24 hrs. Freezing begins in the tapered portion and the metal solidifies as a single crystal. Single crystals of a series of bismuth-antimony alloys were prepared 30 cm. long and 5 mm. in diameter. Although theoretically the crystals should vary in composition along their length this could not be detected by analysis. C. J. SMITHELLS.

**Annealing metal wires.** P. SIEBE (Z. Metallk., 1927, 19, 385—389).—The rate at which wires 1.5 mm. thick of electrolytic and fire-refined copper, copper containing 0.6% Sn, brass with 67% Cu, commercial aluminium (99.3% Al), and soft iron are annealed in a nitrate salt bath at 200—800° has been determined and the results are reproduced in graphical form. With a standard time of immersion in the bath of 100 sec. electrolytic copper wires are completely annealed at 300—350°, refined copper wires at 400°, copper conductor wires (99.4% Cu) at 500°, brass wires at 450°, aluminium wires at 250—300°, and iron wires at 700°. The minimum time of annealing to obtain the maximum ductility is usually somewhat greater than that required to obtain the minimum tensile strength, especially at the lower annealing temperatures. A. R. POWELL.

**Determination of antimony in white metals etc.** H. R. FITTER (J.S.C.I., 1927, 46, 414 T).—The sample is dissolved in nitric acid and the mixed oxides of tin and antimony are collected, washed, and dissolved in a hot solution of oxalic acid. The antimony is precipitated with hydrogen sulphide, the washed precipitate is dissolved in a 10% caustic soda solution, and traces of copper and lead are filtered off. The filtrate is acidified with oxalic acid, the antimony again precipitated with hydrogen sulphide, and the precipitate oxidised with fuming nitric acid and weighed as Sb<sub>2</sub>O<sub>4</sub>.

**Preparation and application of beryllium.** K. ILLIG (Z. angew. Chem., 1927, 40, 1160—1163).

ERRATUM. B., Sept. 2, 1927, 657, col. 1, in the abstract on "Pure nickel and technical nickel," for "Mond nickel usually contains" read "a sample of Mond pellet nickel produced by the carbonyl process contained."

#### PATENTS.

**Alloy impact-tool steel.** P. A. E. ARMSTRONG (U.S.P. 1,645,213, 11.10.27. Appl., 14.11.25).—A very hard and tough steel consists of 1—2.5% W, 0.75—1.75% Cr, 0.5—0.6% Si, 0.5—0.6% Mn, 0.35—0.60% C, 0.05—0.95% Ta, the remainder being principally iron. F. G. CROSSE.

**Manufacture of magnesium and the alkaline-earth metals such as calcium by the electrolysis**



of molten chlorides. A. C. JESSUP (E.P. 256,241, 27.7.26. Fr., 29.7.25).—In the electrolytic manufacture of magnesium, the electrolyte consists of a mixture of chlorides, including magnesium chloride, more electro-positive than the latter and of such composition, *e.g.*,  $3\text{KCl}, \text{MgCl}_2$ , that the density is less than that of the molten magnesium formed. The cathode, which may be of graphite or of a metal slightly miscible with the metal formed and of high m.p., *e.g.*, chromium, tungsten, silicon, etc., or their alloys, is of such form that the metal produced flows over the cathode to maintain a clean surface "wetted" by the molten metal. The composition of the bath should be kept substantially constant.

C. A. KING.

**Improvement of magnesium alloys.** I. G. FARBERININD. A.-G. (F.P. 618,425, 2.7.26. Ger., 3.7.25).—Magnesium alloys containing 7–20% Al or 3–20% Zn or with a combined content of 3–20% (Al + Zn) together with small quantities of silicon and other metals are subjected to a sudden cooling during or immediately after casting.

A. R. POWELL.

**Treatment of lead-tin alloys derived from waste material from lead-smelting works.** M. SPEICHERT, Assec. of F. VOGEL (E.P. 253,908, 14.6.26. Ger., 17.6.25).—Residues accruing from lead-smelting processes, and containing 10–40% of impurities (chiefly copper, antimony, arsenic), are formed into electrodes and used as anodes in an acid solution of, *e.g.*, lead silicofluoride, at a suitable temperature. Lead and tin are dissolved and other impurities remain insoluble.

C. A. KING.

**Resistance alloy.** T. S. FULLER, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,645,099, 11.10.27. Appl., 25.2.27).—The alloy consists chiefly of aluminium, together with 4–10% Mn and 2–8% Zn.

F. G. CROSSE.

**Apparatus for classifying ores etc.** A. FRANCE (E.P. 264,536, 17.1.27. Belg., 18.1.26).—In a classifying apparatus in which a mixture of solid material and water is carried over a perforated surface to a discharging surface, the perforated portion is inclined at a different angle, or may be of varying angle as compared with the discharging surface.

C. A. KING.

**Apparatus for galvanic processes, *e.g.*, chrome-plating.** SIEMENS & HALSKE A.-G. (E.P. 276,610, 16.10.26. Ger., 28.8.26).—All parts of apparatus coming into contact with the electrolyte, except the electrodes, are made of aluminium or aluminium alloy.

J. S. G. THOMAS.

**Treatment of aluminium to render it weldable.** A. KIRCHHOF (E.P. 278,164, 16.9.26).—See U.S.P. 1,604,698; B., 1927, 47.

**Lead-cadmium alloys.** W. FRIEDRICH, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,645,098, 11.10.27. Appl., 12.4.24. Ger., 14.6.23).—See G.P. 405,148; B., 1925, 213.

**Electrodeposition of tin.** VULCAN DETINNING Co., Assees. of H. R. MCILHENNEY (E.P. 269,474, 11.9.26. U.S., 14.4.26).—See U.S.P. 1,597,231; B., 1926, 922.

**Chromium plating.** H. C. PIERCE, Assr. to METALS PROTECTION CORP. (U.S.P. 1,645,927, 18.10.27. Appl., 5.3.26).—See E.P. 267,080; B., 1927, 390.

**Refractory blocks [with tongue and groove] particularly for use in [roofs of] metallurgical furnaces.** L. S. LONGENECKER (E.P. 278,070, 28.6.26).

**Hardness testing device** (E.P. 277,517).—See I.

**Prevention of corrosion** (E.P. 277,417).—See XI.

## XI.—ELECTROTECHNICS.

**Electrical insulating materials.** A. GÜNTHER-SCHULZE (Z. Elektrochem., 1927, 33, 360–368).—The electrical properties of gaseous, liquid, and solid insulating materials are reviewed and their theoretical interpretation is discussed. According to the purposes for which they are required, technical insulators can be divided into four groups. Examples of each type are described. The considerable effect of impurities in diminishing insulating power is emphasised.

H. J. T. ELLINGHAM.

**Electrolytic corrosion.** BOLZINGER.—See II.

**Colour of light sources.** CUNLIFFE and FARROW.—See V.

**Conductivity of glass.** BRYSON.—See VIII.

**Materials for electrical heating.** HARVEY.—See X.

## PATENTS.

**Electric furnace.** J. A. SEEDE, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,645,074, 11.10.27. Appl., 5.6.26).—A closed-loop heating channel having refractory walls communicates at widely separated points with a similar open-loop reservoir for the charge and contains an electric winding for heating, by induction, a conducting charge in the channel.

J. S. G. THOMAS.

**Electric furnace.** V. J. CHAPMAN, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,645,091, 11.10.27. Appl., 26.3.25).—A carbonaceous electrode ends within the wall of a refractory crucible, and a metallic terminal member having substantially the same electrical conductivity as the electrode is secured in electrically conducting engagement with the electrode at a point within the wall.

J. S. G. THOMAS.

**Electric furnace.** C. STEENSTRUP, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,645,119, 11.10.27. Appl., 1.12.22).—An electric furnace comprises a container which is closed except at the bottom, a device forming a closure and seat for the container, means for moving the container relative to this device to permit introduction and removal of the charge, and means for maintaining a supply of any desired gas in the container in its closed and open conditions.

J. S. G. THOMAS.

**Electric furnace resistor.** T. A. REID, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,645,293, 11.10.27. Appl., 25.6.21).—A notched refractory bar is supported by some of a number of alined, spaced refractory members placed in certain of the walls enclosing a furnace chamber, and a second notched refractory bar is arranged in opposite parallel-spaced relation to the first bar, whilst a metallic resistor is helically wound round the two bars and supported in the notches of one of the bars.

J. S. G. THOMAS.

**[Heating elements for] electric furnaces.** BRITISH THOMSON-HOUSTON Co., LTD., Assees. of A. N. OTIS and



C. L. IPSEN (E.P. 258,276, 10.9.26. U.S., 10.9.25).—In electric furnaces of the type comprising refractory walls forming the heating chamber and apertures arranged at a level between upper and lower heating elements, elements having a downwardly-extending limb on either side are looped over supports arranged above each aperture, and sinuous heating elements are laid horizontally below.

J. S. G. THOMAS.

[Separator for] primary cell. Manufacture of primary cells. M. L. MARTUS and E. H. BECKER (U.S.P. 1,644,344 and 1,644,389, 4.10.27. Appl., [A] 29.12.26, [B] 3.1.27).—(A) A separator for use in primary cells having a copper oxide cathode, a zinc anode, and a caustic alkali electrolyte consists of cellulose treated with a solution of sodium zincate ( $d$  1.38). (B) A paper sleeve impregnated with sodium zincate solution and coated with a solution of alkali silicate is sprinkled, while still wet, with zinc dust and placed in a copper oxide-zinc cell with its metallised surface in contact with the copper oxide.

J. S. G. THOMAS.

Electric battery. A. N. HAZLEHURST (E.P. 277,798, 13.9.26).—An electric battery comprises a positive element of lead peroxide, a negative element having an active surface of copper, an electrolyte consisting of sulphuric acid or a soluble sulphate, and means, *e.g.*, a layer of heavy oil floating on the electrolyte, for preventing access of air to the copper at the free surface of the electrolyte.

J. S. G. THOMAS.

Preparation of an electron-emitting cathode. H. MILLER, Assr. to HAZELTINE CORP. (U.S.P. 1,640,710, 30.8.27. Appl., 1.3.26).—An alkaline-earth oxide or hydroxide colloiddally dispersed in methyl alcohol is treated with carbon dioxide to form a colloidal solution of the corresponding carbonate, which is coated electrolytically or otherwise on platinum wire. The coating is converted into the oxide by heating at 1300°.

T. S. WHEELER.

Method of artificially producing [increasing the radioactivity of] radioactive materials. A. GASCHLER (U.S.P. 1,644,370, 4.10.27. Appl., 4.9.24. Ger., 20.4.23).—Material submitted to a high temperature and pressure is treated with electric current at high potential.

J. S. G. THOMAS.

Production of insulating materials [from minerals containing magnesium]. W. ZIMMERMANN (E.P. 277,577, 28.4.27).—Mineral substances containing magnesium, *e.g.*, dolomite, hornblende, are fused and then torn to threads in a stream of steam or compressed air.

J. S. G. THOMAS.

Photometers [for electric incandescence lamps]. GEN. ELECTRIC CO., LTD., N. R. CAMPBELL, and C. G. EDEN (E.P. 277,467, 27.7.26).—Electric lamps of a similar type, even if of different powers, running at equal efficiencies, emit light of the same colour. Photoelectrically active metals vary in their sensitivity to different coloured light, *e.g.*, rubidium is relatively more sensitive to red than blue, compared with sodium. A photometer for determining the voltage at which electric lamps give a standard efficiency is constructed to make use of the above phenomena.

B. M. VENABLES.

Electric [induction] furnace. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of M. UNGER (E.P. 263,857,

30.12.26. U.S., 31.12.25).—See U.S.P. 1,626,437; B., 1927, 450.

High-frequency electric furnace. F. GERTH, Assr. to C. LORENZ A.-G. (U.S.P. 1,645,526, 18.10.27. Appl., 24.3.26. Ger., 30.3.25).—See E.P. 249,843; B., 1926, 985.

Prevention of corrosion [due to stray currents]. E. CUMBERLAND (E.P. 277,417, 15.6.26).

Apparatus for analysis (E.P. 253,090).—See I.

Carbon granules for telephone transmitters (E.P. 277,129).—See II.

Magnesium (E.P. 256,241). Lead-tin alloys (E.P. 253,908). Resistance alloy (U.S.P. 1,645,099).

Apparatus for galvanic processes (E.P. 276,610).—See X.

## XII.—FATS; OILS; WAXES.

Setting point or titre [as an indication of the purity of fats]? M. DITTMER (Chem. Umschau, 1927, 34, 258—259).—Polemical. Whilst the setting point of a fat and the titre of the fatty acid prepared from it are both determined in the same way, the probable error in the former determination is larger owing to the greater tendency of the fat to form supercooled solutions, and the result varies considerably with different forms of apparatus. The titre is therefore the more suitable criterion, but in those cases where the fat is to be used directly the setting point should be determined, using Shukoff's apparatus.

W. J. POWELL.

Saponification of butter fat for determining the Reichert-Meissl value. G. SPITZER and W. F. EPPLÉ (J. Dairy Sci., 1927, 10, 193—194).—Potassium hydroxide is added directly to the previously heated fat, and saponification is completed in an electric oven in 30—40 min.

CHEMICAL ABSTRACTS.

Detection of blubber. J. DAVIDSOHN (Chem. Umschau, 1927, 34, 260; cf. Tsujimoto, B., 1926, 1020; 1927, 754).—Tsujimoto's test for the recognition of blubber, using iodine trichloride, gave negative results with all samples tested.

W. J. POWELL.

Mechanism of the sulphuric acid splitting of vegetable oils and distillation of the resulting fatty acids. A. BAGH (Chem. Umschau, 1927, 34, 271—273).—A critical investigation has been carried out on the behaviour of cotton seed oil, a cotton seed oil-bone grease mixture, and the fatty acids obtained from them by various means, when examined in the Mackey apparatus. The mixed liquid fatty acids obtained from the sulphuric acid splitting of an 80% cotton seed oil-20% bone grease mixture gave a temperature of 99.5° after 2 hrs., the corresponding acids obtained by a potassium hydroxide saponification and subsequent liberation with sulphuric acid gave 147° after  $\frac{3}{4}$  hr., whilst pure cotton seed oil gave 109° at 1 hr. Quantities of the semi-liquid acids from the sulphuric acid splitting of the foregoing mixture were examined after (1) steam distillation during 1½ hrs., (2) heating at 200—210° for 12 hrs. in the presence of carbon dioxide and then distilling, (3) heating at 270—280° as in (2), and (4) heating with superheated steam at 280—290° for 35 hrs. and then distilling. The temperatures attained in the Mackey apparatus after 1 hr. were 144°, 152°, 98°, and 97°, respectively. It is concluded that



when a fatty acid mixture containing linoleic acid is heated at 280° in an atmosphere of carbon dioxide or steam, isomerisation occurs giving a form much more resistant to oxidation. This is apparent in the Mackey tests not only in the moderate temperatures attained, but in the lengthened period necessary for the temperature rise.

E. HOLMES.

**Mackey test [for oils].** H. VON NABELL (Chem. Umschau, 1927, 34, 273–275).—Considerable errors may be introduced into the Mackey test by the use of metallic wire-gauze cylinders, which, in contact with the oil under test, give rise, particularly at the higher temperatures, to metallic soaps which have a marked accelerating effect on the self-heating properties of the oils. In an example, a sample of olein tested in an iron-gauze cylinder attained a temperature of 100° in 1 hr. 5 min., and ignited in 1 hr. 50 min.; an identical specimen, when tested in a copper cylinder reached 97° in 2 hrs., whilst a test carried out in a buckram cylinder gave less than 90° in 2 hrs. Buckram is, therefore, recommended as a suitable material for the purpose, or, failing that, one of the noble metals, as copper or silver.

E. HOLMES.

**Various bromine-iodine values of oils and fats.** W. VAUBEL (Z. angew. Chem., 1927, 40, 1143–1145).—The primary, secondary, and tertiary bromine-iodine values (cf. B., 1927, 49) of a number of vegetable and animal oils, fats, and waxes are tabulated and the main points of interest arising therefrom are discussed. As evidence of the selective nature of the reaction, secondary and tertiary values are shown by paraffin and glycerol, but not by benzene. In general, oils show increased values on saponification. Ring-splitting and oxidation processes are in the main responsible for tertiary values and partially for the secondary values, actual "additive" bromine corresponding to the primary value, e.g., olive oil, or the secondary value, e.g., linseed oil, or lying between the two. In the case of linseed oil, the three values are analysed in terms of the components of the oil, as stated by Eibner and Schmidinger. S. S. WOOLF.

**Determination of iodine-bromine numbers with potassium bromate and arsenite solution.** L. W. WINKLER (Arch. Pharm., 1927, 265, 554–560).—In this determination it is an advantage to use approximately 0.25*N*-arsenious acid solution in place of the less stable 0.5*N*-sodium arsenite recommended in the Deutsches Arzneibuch. The arsenious acid solution may be accurately titrated against the potassium bromate solution alone, or in the presence of iodine as an indicator. If specially purified carbon tetrachloride is used there is no loss of bromine, either from the vapour phase or by leaving the reaction mixture for 24 hrs., and it is unnecessary to moisten the stopper of the flask with phosphoric acid. With ordinary carbon tetrachloride loss of bromine occurs. There is no necessity to use indigo-carmin as indicator, and the time of the reaction can be conveniently shortened to  $\frac{1}{2}$  hr. It is then necessary to correct the limiting numbers given in the Arzneibuch to agree with this time of reaction (cf. Schulek, A, 1921, ii, 603). Good results are recorded with 12 unsaturated oils, including castor oil. The latter is an excellent substance for checking new test solutions; with a time

of reaction of  $\frac{1}{2}$  hr. the iodine-bromine number must be  $84.5 \pm 0.5$ . During the determination daylight must be rigidly excluded, and the amount of fat taken must be sufficient to react with approximately half of the bromine liberated.

S. COFFEY.

**Gossypol content and chemical composition of cottonseeds during certain periods of development.** W. D. GALLUP (J. Agric. Res., 1927, 34, 987–992; cf. Schwartze and Alsberg, B., 1924, 22).—Analyses of cottonseeds at different stages of development are reported. The percentage of gossypol present increased rapidly from the time the boll was mature until it opened, and continued to increase slowly for some time afterwards. No correlation was found between the formation of oil and the formation of gossypol.

C. T. GIMINGHAM.

**Determination of free alkali in soap.** J. DAVID-SOHN (Chem. Umschau, 1927, 34, 260).—In the determination of free alkali in soft soaps and in bar soap containing much water, the titration of the alcoholic solution with 0.1*N*-alcoholic hydrochloric acid is often uncertain owing to the return of the red colour of the phenolphthalein after the end-point is reached. This may be avoided by adding anhydrous sodium sulphate (4–6 g.) to the alcoholic soap solution (3.5 g. in 50–70 c.c. of 95% alcohol, previously neutralised), decanting, and washing the sodium sulphate with absolute alcohol, and then carrying out the titration.

W. J. POWELL.

**Rubber seed oil.** DITMAR.—See XIV.

PATENTS.

**Separation of substances of dissimilar volatilities [e.g., refining fats].** E. WECKER (E.P. 277,085, 7.6.26. Addn. to E.P. 213,267; cf. E.P. 235,792; B., 1925, 680).—The refining process described in the earlier patents is carried out in suitable apparatus, details of reaction chambers and scrubbing, heat-exchanging, and atomising devices being given.

S. S. WOOLF.

**Reduction of auto-oxidation of oils and fats.** A. RECHBERG G.M.B.H., G. BRAUN G.M.B.H., and H. OESTERMANN (E.P. 254,303, 21.6.26. Ger., 24.6.25).—The auto-oxidation of oils and fats used for greasing wool is reduced or prevented by the addition of catalyst poisons ("paralysts"), e.g., sulphur, arsenic sulphide. [Statutory ref. to E.P. 256,654; B., 1926, 887.]

S. S. WOOLF.

**Fixing agent for perfumes etc.** (G.P. 441,630).—See XX.

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

**Physical factors influencing the properties of paint pigments.** A. DE WAELE (J. Oil and Colour Chem. Assoc., 1927, 10, 232–249).—The specific absorptions of polar and non-polar media by pigments of various types and dispersions are tabulated, and these and typical photomicrographs are discussed with reference to the three types of solid-liquid dispersions previously postulated by the author, i.e., colloidal dispersion, equilibrium, and flocculation. Permanently dispersed systems behave virtually as true fluids up to certain limiting volume concentrations, whilst flocculated systems show the effects of interference between dispersed particles, the stress-strain relationship being curvilinear and the



system as a whole showing static rigidity at close packing. The fundamental characteristics of such systems are bound up with the resistance set up by transverse orientation of liquid molecules from particle to particle.

S. S. WOOLF.

**Application of vat colours.** WILSON.—See VI.

#### PATENTS.

**Method of imparting a decorative finish to articles.** H. E. B. and J. H. GREENE (E.P. 276,864, 8.11.26).—Articles are dipped into paint, the medium of which contains a highly volatile solvent, *e.g.*, cellulose lacquers, and withdrawn slowly at an optimum rate. After superfluous paint has drained off, the article is revolved and reversed periodically until the film is set, the formation of folds or wrinkles being thus avoided. "Marbled" effects may be obtained by applying another paint of similar nature but different colour by dipping or otherwise at an intermediate stage in the drying of the first coat, and continuing the reversing and revolving process.

S. S. WOOLF.

**Manufacture of pigments, impregnating compounds, etc.** R. M. GERLACH (E.P. 277,083, 7.6.26).—Heavy-metal salts of weak acids, *e.g.*, acetates, formates, ferro- or ferri-cyanides, silicofluorides of lead, mercury, and copper, are combined with the acidic components of resins, oils, or fats, *e.g.*, abietic acid, naphthenic acid, higher fatty acids, in the cold or with the application of slight heat, producing a complex compound which dissolves in excess of the acid solvent. Increased disinfectant, preservative, and fungicidal properties are claimed.

S. S. WOOLF.

**Manufacture of iron oxide.** H. C. STEWART, Assr. to WESTMORELAND CHEM. & COLOR CO. (U.S.P. 1,642,975, 20.9.27. Appl., 3.5.23).—Dry copperas is fed into a furnace with a blast of air and burnt therein to oxide of iron.

D. J. NORMAN.

**Waterproofing composition.** A. ASHBY (E.P. 277,051, 5.2.27).—Waterproofing compositions consisting of mixtures of turpentine or turpentine substitute, linseed or other drying oil, and 6–12% of a wax (preferably a mineral wax) are claimed. Small amounts of goldsize and/or driers may be added.

S. S. WOOLF.

**Condensation-product [air-drying] varnish.** A. L. BROWN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,640,562, 30.8.27. Appl., 29.3.24).—A mixture of cresol, trioxymethylene, and tung oil is heated first at 100–120° and then, with addition of a drier, *e.g.*, cobalt linoleate, at 200°. The product is thinned with turpentine.

T. S. WHEELER.

**Manufacture of condensation products of crotonaldehyde.** I. G. FARBENIND. A.-G., Assees. of W. KROPP (U.S.P. 1,640,899, 30.8.27. Appl., 5.3.26. Ger., 14.5.25).—The process claimed in G.P. 372,855 (B., 1923, 1140 A) is modified as regards its application to crotonaldehyde by omission of a condensing agent.

T. S. WHEELER.

**Waste acid from pigment manufacture** (E.P. 277,769).—See VII.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Preserved latex.** V and VI. **Preserving latex with ammonia.** O. DE VRIES and N. BEUMÉE-NIEUWLAND

(Comm. Proefstat. Rubber, Buitenzorg, 1927, [17 and 18], 289–324, 325–346; Arch. Rubbercultuur, 1927, 11, [8]).—V. To maintain latex in good condition in open tanks for 24 hrs. on the plantation the addition of 0.07% of anhydrous ammonia is sufficient. The decrease in alkalinity observed earlier in stored latex preserved with a sufficient proportion of ammonia is found to be due to loss by evaporation and not to any chemical or bacterial action; if the proportion of ammonia, however, is insufficient, the bacterial decomposition which leads to coagulation also leads to the formation of acids which cause a rapid decrease in the alkalinity. Spontaneous coagulation in ammoniated latex appears to be due to a decomposition of different type from that in non-preserved latex, the clotting in the former case occurring at a lower degree of acidity. The deposit which forms in freshly-ammoniated latex consists mainly of water, ammonium magnesium phosphate, and rubber, with smaller proportions of other mineral matter, protein, dirt, etc.

VI. The alteration in the rate of vulcanisation of the rubber in preserved latex on storage is not accompanied by any corresponding change in the alkalinity or in the nitrogen content of the rubber; the viscosity and plasticity remain almost constant and show a decrease only in old ammoniated latex. Rubber from ammoniated latex shows normal behaviour with respect to a slight decrease in viscosity and plasticity on storage; the tensile strength and slope remain constant, whilst the rate of vulcanisation gradually decreases and approaches the value for the control from unpreserved latex. With increase in the age of the ammoniated latex up to six months, the rate of vulcanisation and viscosity of the rubber decrease whilst its plasticity is constant, and its condition remains good even after 1½ years.

D. F. TWISS.

**Isolation of the natural oxidation inhibitors of crude *Hevea* rubber.** H. A. BRUNSON, L. B. SEBRELL, and W. W. VOGT (Ind. Eng. Chem., 1927, 19, 1187–1191).—Two substances of marked anti-oxidant character have been separated from the unsaponifiable portion of the acetone extract from raw rubber. These are high-boiling viscous oils of composition  $C_{27}H_{42}O_3$  and  $C_{20}H_{30}O$ ; they are apparently sterols and are present to the extent of 0.08% and 0.16% of the rubber, respectively. There are also present *n*-octadecyl alcohol, a ketone  $C_{15}H_{24}O$ , and a hydrocarbon  $C_{15}H_{24}$ , but these are without protective action. The aqueous extract of the acetone-soluble material also contains an anti-oxidant, probably a mixture of phenolic and ketonic substances.

D. F. TWISS.

**Micrographic studies of vulcanised rubber, showing the evolution of the free sulphur.** REGNAUD (Chim. et Ind., 1927, 18, 93–96).—Micrographic examination of various samples of vulcanised rubber at progressive intervals after vulcanisation reveals generally a succession of changes, *viz.*, flocculation, coalescence, and crystallisation, in the residual free sulphur; the crystals in the interior tend finally to disappear as crystallisation proceeds at the surface. The observations are believed to support a colloidal theory of vulcanisation.

D. F. TWISS.



**Mould on rubber.** O. DE VRIES (Comm. Proefstat. Rubber, Buitenzorg, 1927, [16], 262–283; Arch. Rubbercultuur, 1927, 11, [8]).—The prolonged and stimulated growth of mould on raw smoked sheet rubber, *e.g.*, for several years, causes an actual loss of rubber hydrocarbon. Even after two years when the loss amounts to 20%, however, the rubber after cleaning and washing shows normal properties and composition, but continuous mould growth for five years, accompanied by a loss of over 30%, causes depreciation in viscosity, slope, and tensile strength, and a decrease in the rate of vulcanisation. The nitrogen content of the various samples indicates that the nitrogenous constituents are not consumed by the mould.

D. F. TWISS.

**Ageing properties of rubber coagulated with formic acid.** R. RIEBL (Comm. Proefstat. Rubber, Buitenzorg, 1927, [20], 354–359; Arch. Rubbercultuur, 1927, 11, [8]).—Storage experiments over 2 and, in one case, 3 years give a definite indication that with formic acid as coagulant, the rubber obtained is comparable in properties with that produced with acetic acid.

D. F. TWISS.

**Comparative experiments with gas black and lamp black [in rubber].** P. SCHIDROWITZ (India-rubber J., 1927, 74, 569–572).—Comparison of three samples of American gas black and three of European lamp black in standard mixtures of two types containing rubber (100 pts. by wt.) and black (36 pts.) with sulphur (4 pts.), zinc oxide (10 pts.), and diphenylguanidine (1 pt.), and with sulphur (5 pts.) and litharge (30 pts.), respectively, show that after vulcanisation the tensile product (tensile strength  $\times$  elongation at break), energy of resilience, and resistance to abrasion are greater for the gas black samples. The differences persist after artificial ageing.

D. F. TWISS.

**Rubber seed oil and its industrial application.** R. DITMAR (Caoutchouc et Gutta-Percha, 1927, 24, 13,725–13,728).—After a review of earlier work on the oil present in the seeds of *Hevea brasiliensis*, this oil is shown to be more fluid than colza oil. Details are given for its conversion into light and dark “rubber substitutes.” These, as well as the oil itself, are effective as softeners for raw rubber during the milling and mixing operations.

D. F. TWISS.

**Determination of sulphur in manufactured rubber.** L. GRAFFE (Caoutchouc et Gutta-Percha, 1927, 24, 13,722–13,723).—The sample (0.25 g.) in small pieces is treated with 5 c.c. of nitric acid (*d* 1.52), the reaction being initiated by cautious warming on a sand bath and subsequent removal. After the first vigorous action the heating is continued for  $\frac{1}{2}$  hr. A small quantity of a mixture of potassium chlorate and sodium nitrate (1:1) totalling 5 g. is introduced, and heating is continued until the disappearance of the chlorine. The reaction mixture is then transferred to a porcelain crucible with the aid of a few c.c. of nitric acid and heated on a water-bath with the occasional addition of portions of chlorate-nitrate mixture until no further reaction results. When at least 2.5 g. of the oxidising mixture have been added, the product is evaporated to complete dryness and the crucible cautiously heated over a flame

until the oxidation process is complete. After introducing the remainder of the chlorate-nitrate mixture, the crucible is heated strongly until the evolution of oxygen ceases. The contents are then dissolved in water, acidified with hydrochloric acid, filtered if necessary, and treated with barium chloride in the usual way.

D. F. TWISS.

**Constitution of caoutchouc.** R. PUMMERER (Z. angew. Chem., 1927, 40, 1168–1172).—A review.

**Rubber filter-press frames etc.** FRITZ and CLARK, JUN.—See I.

## PATENTS.

**Process for accelerating the vulcanisation of rubber.** W. SCOTT, Assr. to RUBBER SERVICE LABORATORIES Co. (U.S.P. 1,639,903, 23.8.27. Appl., 14.8.24).—The product obtained by the action of a primary arylamine, *e.g.*, aniline (1–3 mols.), on an aliphatic aldehyde, *e.g.*, acetaldehyde (1–3 mols.), is treated at below 30° with 2% of concentrated hydrochloric acid, then neutralised, and heated with formaldehyde or acetaldehyde.

T. S. WHEELER.

**Manufacture of aldehyde reaction products [accelerators] of the aldehyde derivative of a Schiff's base.** C. O. NORTH, Assr. to RUBBER SERVICE LABORATORIES Co. (U.S.P. 1,639,957, 23.8.27. Appl., 25.3.25).—The product obtained by the action of a primary arylamine, *e.g.*, aniline (2 mols.), on an aliphatic aldehyde, *e.g.*, acetaldehyde (3 mols.), is heated with formaldehyde or acetaldehyde at 95° under increased pressure for 4 hrs., and then under reduced pressure.

T. S. WHEELER.

**Preparation of plastics containing rubber.** J. MCGAVACK, Assr. to REVERE RUBBER Co. (U.S.P. 1,640,363–4, 30.8.27. Appl., [A] 3.7.23; [B] 24.3.25).—(A) Pale crêpe rubber dissolved in carbon tetrachloride is heated with sulphuric acid and an aliphatic aldehyde, *e.g.*, formaldehyde, with or without addition of phenol, at 100° for 2.5 hrs. (B) Vulcanised rubber dispersed in benzene is treated with chlorine, and then heated with phenol and formaldehyde. Substitutes for hard rubber are thus obtained.

T. S. WHEELER.

**Machine for mixing rubber and like material.** FARREL & MACHINE Co. (E.P. 271,075, 9.5.27. U.S., 17.5.26).

## XV.—LEATHER; GLUE.

**Hydrolysis of pelt in acid media.** G. GRASSER and S. TAGUCHI (Le Cuir, 1927, 426–428).—Pieces of pelt (1–10 g.) were immersed in 100 c.c. of solutions of hydrochloric, nitric, sulphuric, phosphoric, formic, oxalic, lactic, mono- and tri-chloroacetic, tartaric, citric, acetic, boric, and butyric acids of various strengths, and boiled under reflux. *N*- and 0.1*N*-hydrochloric, *N*- and 0.1*N*-nitric, and *N*-sulphuric acids hydrolysed the pelt completely in 1, 7, 1.3, 17, and 3.7 hrs., respectively; 0.1*N*- and 0.01*N*-sulphuric, *N*-phosphoric, 2*N*-formic, *N*-oxalic, lactic, mono- and tri-chloroacetic, tartaric, and citric acids hydrolysed the pelt completely after prolonged boiling. The hydrolytic action of the acids depends on their  $p_H$  value, and they can be arranged in the following order:—hydrochloric, nitric, sulphuric, oxalic, phosphoric, tartaric, citric, lactic, boric, butyric, and acetic acids. The first five exercise



strong hydrolytic action, tartaric, citric, and lactic acids are much feeblers, whilst the remaining acids have very little effect.

D. WOODROFFE.

**Measurement of the adhesive strength of glue.** C. E. LANYON (Ind. Eng. Chem., 1927, 19, 1191—1193).—Briquettes were made by mixing 400 g. of sand with 60 g. of a 20% solution of the glue, filling into moulds, and drying for 6 weeks in air at 25° and 60% relative humidity. The adhesive strength of the glue was measured by determining the breaking strength of the briquette by means of a Riehle cement-testing machine. The mean of a number of tests made by two investigators on the same glue differed only by 2%. No relationship was found between viscosity, tensile strength, and briquette strength, but on varying the acidity a maximum briquette strength was obtained with a glue solution of  $p_H$  7.5. Wide differences were observed in the adhesive strength of glues of the same grade from different manufacturers, and hence the viscosity or jelly strength alone is useless for testing the suitability of glues for joints in the abrasive industry.

F. R. ENNOS.

#### PATENTS.

**Method of degreasing skins.** C. A. BROWN and G. J. ESSELEN, JUN., ASSRS. to MANUFACTURING IMPROVEMENT CORP. (U.S.P. 1,640,478, 30.8.27. Appl., 25.7.24).—Sheep skins are impregnated with an aqueous emulsion of petroleum and an olive oil soap, then immersed in warm brine, and finally pressed.

T. S. WHEELER.

**Ornamentation of leather** (E.P. 277,091).—See V.

### XVI.—AGRICULTURE.

**[Exchangeable bases in soil.]** A. M. SMITH (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1927, B, 8—9).—Treatment of soil with calcium chloride largely increased the proportion of exchangeable calcium at the expense of the other bases. An equimolecular solution of sodium and potassium chlorides increased the amount of exchangeable potassium to a much greater extent than the exchangeable sodium.

A. G. POLLARD.

**Exchangeable hydrogen and soil reaction.** C. J. SCHOLLENBERGER (Science, 1927, 65, 552—553).—*N*-Ammonium acetate solution at  $p_H$  7.0 (prepared from ammonia and acetic acid, the reaction being controlled with a quinhydrone electrode) is preferred to ammonium chloride for extracting the exchangeable bases of soils. A method of electrometric titration is described.

A. A. ELDRIDGE.

**Sorption of nitrates in soils.** K. FLEROV (Kolloid-Z., 1927, 43, 81—84).—From the work of previous investigators it appears that nitrates are not adsorbed by soils. The author shows that this conclusion is due to neglect of differentiation between "true" and "apparent" adsorption. Experiments were carried out on the adsorption of potassium nitrate by gels of silica, ferric oxide, and alumina. Positive adsorption was observed in all cases, and the adsorptive power was greater the fresher and more rich in water the gel. Although the formation of insoluble basic ferric and aluminium nitrates may play some part, this is considered to be of a secondary nature. The true nature of the adsorption is the partition of the nitrate between the dispersion medium and the solid phase.

E. S. HEDGES.

**Absorption of nitrogen oxides in an aqueous suspension of phosphate rock.** V. N. MORRIS (Ind. Eng. Chem., 1927, 19, 1143—1147).—It was found that a suspension of ground Florida pebble phosphate in water or nitric acid absorbed oxides of nitrogen more effectively than water or nitric acid alone. The unabsorbed oxides of nitrogen were reduced to nitrogen with Devarda's alloy and the total nitrogen in the exit gases was determined. 50 g. of rock to 100 c.c. of 40% nitric acid gave the best absorption; excess of phosphate was injurious. Trials with calcium nitrate and with monocalcium phosphate produced a similar effect, the absorption-concentration curves showing maxima in each case. The dissolution of phosphate is slow under these conditions, and a large excess of acid or long time is necessary to bring it to completion. The mixture of calcium nitrate and monocalcium phosphate eventually obtained is rather hygroscopic for use as a fertiliser.

C. IRWIN.

**Effect of applications of [calcium] cyanamide on the nitrate content of field soils.** F. E. ALLISON (J. Agric. Res., 1927, 34, 657—662).—Experiments are reported which show that calcium cyanamide greatly retards nitrification in soils, the amount of nitrates present in soils receiving cyanamide being below that in untreated soils, even after 61 days' incubation. The results account, at least partly, for the poor results obtained with cyanamide as a fertiliser for cotton.

C. T. GIMINGHAM.

**Sodium hydroxide versus sodium carbonate [in soils].** P. S. BURGESS (Science, 1927, 65, 445—446).—Few black alkali soils contain sodium carbonate under field conditions, the alkalinity being due to sodium hydroxide derived from hydrolysis of sodium zeolite.

A. A. ELDRIDGE.

**Determination of soil reaction by means of the quinhydrone electrode.** E. BILLMANN and S. TOVBORG-JENSEN (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1927, B, 236—274).—Comparison of  $p_H$  determinations by means of the hydrogen electrode and by the quinhydrone electrode showed agreement to within  $p_H$  0.1 in the majority of cases, the greatest difference observed being 0.2. The differences in values obtained with duplicate soil samples were from 0.1 to 0.3. Potential differences with the quinhydrone electrode were attained instantaneously, and there was no evidence of the presence in soil extracts of substances affecting the components of quinhydrone. No appreciable difference resulted whether the electrode was immersed in the soil-water paste or in the clear liquid above. The effect of changes in the soil: water ratio on the  $p_H$  values obtained is most marked in the case of soils with  $p_H$  less than 6.5, greater dilution causing higher values to be recorded. In many cases  $p_H$  values of fresh and of air-dried samples are substantially the same. Boiled or aerated water should be used in preparing soil suspensions, as the presence of carbon dioxide causes irregularities in the method. Nitrates, in the concentrations usually found in soils, do not appreciably affect the quinhydrone electrode. The latter cannot be utilised for determining  $p_H$  values greater than 8.5.

A. G. POLLARD.

**Quinhydrone electrode applied to the determina-**



tion of  $p_H$  values of soils. C. BRIOUX and J. PIEN (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1927, B, 5—7).—Using 1 : 5 soil-water suspensions,  $p_H$  values determined with the hydrogen electrode increase with the period of preliminary agitation. With the quinhydrone electrode maximum values are reached in a few minutes, and are markedly higher than those obtained with the hydrogen electrode. The  $p_H$  values of centrifuged soil extracts obtained with the quinhydrone electrode increase with the period of preliminary shaking, reaching a maximum after 20—44 hrs. The figures thus obtained are practically identical with those for soil suspensions determined by the hydrogen electrode.

A. G. POLLARD.

**Rapid determination of soil moisture by alcohol.** G. J. BOUYOUCOS (Science, 1927, 65, 375; cf. *ibid.*, 1926, 64, 651).—Manipulative details of the hydrometer method are given.

A. A. ELDRIDGE.

**Relation of potash removed by crops to active, total, acid-soluble, and acid-insoluble potash of the soil.** G. S. FRAPS (Texas Agric. Exp. Sta. Bull., 1927, No. 355, 33 pp.).—Methods are given for the determination of active, total, and acid-soluble potash in soil, and the relation between the form of the potash and the amount removed by crops is determined.

CHEMICAL ABSTRACTS.

**Action of calcium carbonate and phosphorite on the composition of soil solutions and aqueous extracts from soils.** D. DRUZHININ (Nauk. Agron. Zhur., 1925, 2, 629—644).—The accumulation of nitrates in unfertilised soils is ordinarily not accompanied by a decrease in the ratio of calcium (equivalent) to the total acid (equivalents); in limed soils and soils treated with phosphorite the calcium equivalent was approximately equal to the sum of the anions. Adsorbed calcium is the most important neutralising agent of the nitric acid.

CHEMICAL ABSTRACTS.

**Combined fraction of soil solution. Negative adsorption of electrolytes by soil.** A. V. TROFIMOV (Nauk. Agron. Zhur., 1925, 2, 613—628).—Calcium nitrate and chloride, and certain alkali chlorides, when added to black soils, loam, or podsol soils, become irregularly distributed in the soil solutions, being least concentrated in the surface soil solution as a result of negative adsorption. The degree of non-uniformity could be expressed by the adsorption isotherm.

CHEMICAL ABSTRACTS.

**Saline county soils. Marion County soils.** R. S. SMITH, E. A. NORTON, E. E. DE TURK, F. C. BAUER, and L. H. SMITH. **Will County soils.** R. S. SMITH, O. J. ELLIS, E. E. DE TURK, F. C. BAUER, and L. H. SMITH (Ill. Agric. Exp. Sta., Soil Rep., 1926, No. 33, 53 pp.; No. 34, 66 pp.; No. 35, 61 pp.).

**Bordeaux mixture.** HOLLAND and others.—See VII.

PATENTS.

**Drying of root-crops, grain, etc.** B. J. OWEN (E.P. 277,434, 18.6.26).—The material to be dried is piled between two concentric cylindrical structures, the inner one forming an internal air-distributing chamber. Heated air is delivered either into this internal chamber or into an external chamber formed by means of a removable air-tight casing, capable of being fixed round

the outer cylinder. The chambers and supply-pipes are fitted with closing slides or shutters, and are so arranged that the direction in which the air is passed through the mass may be reversed at intervals or at any particular stage of the operation.

C. T. GIMMINGHAM.

**[Plant] disinfectant.** R. LIESKE, W. SCHULEMANN, and W. BONRATH, Assrs. to WINTHROP CHEMICAL CO. (U.S.P. 1,640,901, 30.8.27. Appl., 28.1.25).—An organic mercury compound, e.g., mercuribenzoic acid, is mixed with an alkali phenoxide in presence of water to give solutions of value as plant disinfectants.

T. S. WHEELER

## XVII.—SUGARS; STARCHES; GUMS.

**Discoloration of sugars of different qualities at high temperatures in absence and in presence of other substances.** O. SPENGLER and F. TÖDT (Z. Ver. deut. Zucker-Ind., 1927, 623—640).—Comparison of samples of white sugar in respect of their liability to discoloration when melted, e.g., for confectionery, can be satisfactorily made by heating 5 g. lots in test-tubes in an oil bath. With samples ranging in ash content from 0.0026 to 0.03%, the authors obtained very pronounced differences by raising the temperature from 100° to 167° and cooling again to 100° in a total period of 110 min. A study of the effects of moistening with 0.2 c.c. of various solutions before heating indicates that neutral salts and betaine are not responsible for discoloration, solutions of these substances producing no more effect than distilled water. A sample slightly discoloured by one heating is darkened much more by a second heating, so that the effect, as suggested by Lunden, is due more to decomposition products of the sugar than to foreign matters. Moistening with sodium hydroxide or mineral acid may cause strong discoloration, and with acid the colour depends very strikingly on the relative amounts of sugar and water present. Sugar (5 g.) moistened with 0.2 c.c. of 0.1N-hydrochloric acid melts below 100° to a clear, greenish yellow, partially inverted mass which darkens on continued heating and eventually becomes thoroughly caramelised. An inferior white sugar, which by itself cannot be melted without discoloration, will, if thus moistened with acid, yield a much lighter melt, which is partly-inverted, however, and therefore hygroscopic. If 5 g. of sugar are heated in a water-bath with 0.2, 0.6, 2, and 5 c.c. of 0.1N-hydrochloric acid, the maximum colour is produced by 0.6 c.c. These curious results with acid are attributed to changes in the activity of the acid, resulting from the abstraction of water by the sucrose and by the inversion process.

J. H. LANE.

**Influence of hyposulphites on [beet] syrups.** J. ZAMARON (Bull. Assoc. Chim. Sucr., 1927, 44, 426—428).—In laboratory tests on beet syrups of about 65° Brix, treated with hyposulphite and then heated to 95° before filtration, no improvement in colour was perceptible with less than 1 g. of hyposulphite per litre, and the purity of the syrups was practically unchanged. Sulphited factory syrups treated with 40 g. of hyposulphite per hectolitre at 70° and then heated to 95° before filtration underwent a slight amount of inversion although alkaline.

J. H. LANE.



**Fermentation of beet molasses.** CLAASSEN.—See XVIII.

#### PATENTS

**Purification of sugar juice.** P. J. T. MORIZOT (F.P. 609,731, 21.1.26).—The juice, after treatment with lime, is treated with carbon dioxide and phosphoric acid or a soluble monobasic phosphate. L. A. COLES.

**Manufacture of starch.** F. B. DEHN. From PENICK & FORD, LTD., INC. (E.P. 277,400, 19.4.26).—In the production of starch from corn, the overflow water from the gluten settlers instead of being rejected is used again—the minor portion for steeping the grain, and the major portion, after being sterilised by means of the steam obtained by concentrating the steep waters, for effecting further separation of the insoluble ingredients of the grain. F. R. ENNOS.

**Manufacture of starch.** E. C. R. MARKS. From CORN PRODUCTS REFINING CO. (E.P. 277,572, 8.4.27).—Successive quantities of maize are treated continuously by steeping and disintegrating the grain in the presence of sulphur dioxide, removing the germ, bran, and fibre, tabling the starch, separating the water from the gluten, and twice washing and filtering the starch with water containing sulphur dioxide to remove the soluble material. The water separated from the gluten and that from the starch filters is returned to the process for re-use, the former being used partly for steeping and partly in the germ and coarse slop separation, and the latter, after heating, in the fine slop separation. F. R. ENNOS.

**Treatment of potatoes in the production of starch.** MASCHINENBAU-ANSTALT HUMBOLDT (G.P. 441,911, 20.2.25).—Potatoes are treated in disc mills open to the whole circumference of the discs. The cells are completely broken down, and the starch may be easily washed out. B. FULLMAN.

**Manufacture of large-grained dextrose.** W. B. NEWKIRK, ASSR. to INTERNAT. PATENTS DEVELOPMENT CO. (U.S.P. 1,640,717, 30.8.27. Appl., 23.10.24).—See U.S.P. 1,521,830; B., 1925, 183.

### XVIII.—FERMENTATION INDUSTRIES.

**Fermentation of [beet] molasses by the aeration process of yeast manufacture.** H. CLAASSEN (Z. Ver. deut. Zucker-Ind., 1927, 607—622).—Preliminary experiments to ascertain the losses of alcohol by evaporation during the aeration of 0.2—0.7% aqueous solutions of alcohol at temperatures of 25—31° showed losses ranging from 1.32 g. to 3.68 g. of alcohol per cub. m. of air. The worts for the fermentation experiments were made by diluting beet molasses to 20° Balling, heating to 80—90°, treating with 1% of sulphuric acid and 4% of superphosphate, and keeping the mixture for several hours to clarify. Only 5—10% of the total sucrose was inverted by this treatment, but inversion was practically complete within 4 hrs. of the addition of the yeast. In a series of small-scale experiments with 6-litre portions of wort, fermented with 30 g. of yeast, and differently aerated with 20—80 litres of air per min., the yeast crop increased with the intensity of aeration up to 60 litres per min., but 80 litres per min. produced no further increase. The total amount of nitrogen assimilated was the same in all cases, and the nitrogen content of the

yeast formed was, therefore, in inverse ratio to the amount of the crop. In baking and keeping qualities the more intensely aerated yeasts were inferior to the others. Addition of 5 g. of ammonium sulphate to the wort, combined with aeration at 60 litres per min., did not improve the amount or quality of the yeast crop, although it increased the nitrogen content. A large-scale fermentation was carried out with wort from 3000 kg. of molasses, 120 kg. of superphosphate, and an extract of 525 kg. of malt germs. The amount of pitching yeast was 360 kg. (77% of moisture) and the duration of fermentation 11 hrs. Aeration was started at 3000 cub. m./hr., increasing soon to 5000 cub. m., and falling again to 2000—3000 cub. m. for the last 2 hrs. The crop of commercial pressed yeast was 1875 kg., representing 1733 kg. of new yeast containing 28.71 kg. of nitrogen or 48.6% of the original total nitrogen of the wort. Of the total sugar of the molasses, equivalent to 1575 kg. of invert sugar, 52.2% was accounted for as carbon dioxide, 26.8% as alcohol (including 6.2% lost by evaporation), 14.2% as non-protein matters of the new yeast, and the balance, 6.8%, as other undetermined metabolic products. The fermented wort contained unfermentable reducing substances equivalent to 1.4% of the original sugar. J. H. LANE.

**Bacterial infection of beers.** C. G. MATTHEWS and G. C. MATTHEWS (J. Inst. Brew., 1927, 33, 502—506).—A deficiency of carbon dioxide in beers and stouts at any time during fermentation and the use of malt of poor quality in their manufacture predisposes such beers and stouts to infection. A case is described where a bottled beer, already so predisposed to adverse change, was attacked by ropiness, the cause of which was traced to the combined effect of the presence of *Pediococcus* in the finings and in the colouring solution. C. RANKEN.

**Adaptation of the bactericidal action of chloroform to the preparation of bacterins.** H. BUNYEA (J. Agric. Res., 1927, 34, 623—630).—Experiments are reported on the use of chloroform as a sterilising agent in the preparation of bacterins. A broth culture of *Staphylococcus aureus* is sterilised by the passage of air containing chloroform vapour until the liquid has absorbed about 4.5% of chloroform, provided that the process is so retarded as to use that amount of chloroform in 2½ hrs. C. T. GIMMINGHAM.

**Synthetic methyl alcohol and ammonia from butyl fermentation gases.** J. C. WOODRUFF (Ind. Eng. Chem., 1927, 19, 1147—1150).—The waste gases from the butyl alcohol-acetone fermentation of maize, which are very constant in composition at 60% of carbon dioxide and 40% of hydrogen, are stripped of their solvent vapour content (55% of acetone and 22.5% each of butyl and ethyl alcohols) by passing through cooled activated coconut-shell charcoal. After removal of the carbon dioxide, the hydrogen is mixed with air to give a 3:1 hydrogen-nitrogen mixture after burning the oxygen to water vapour, and this mixture is then passed to the high-pressure converter. The yield of ammonia is equal to that obtained from the purest electrolytic hydrogen. The high-pressure ammonia plant has been adapted to the production of methyl alcohol, for which purpose it is necessary to remove only a portion of the



carbon dioxide content of the fermenter gas, the reaction employed being  $\text{CO}_2 + 3\text{H}_2 = \text{CH}_3\cdot\text{OH} + \text{H}_2\text{O}$ . Investigations are in progress for the development of a suitable process for supplying carbon monoxide in order to make use of the reaction  $\text{CO} + 2\text{H}_2 = \text{CH}_3\cdot\text{OH}$ , whereby the methyl alcohol output for a given compressor capacity may be increased 33%. F. R. ENNOS.

**Production of lactic acid by fermentation of wood sugar remaining after alcoholic fermentation.**

E. A. MARTEN, E. C. SHERRARD, W. H. PETERSON, and E. B. FRED (Ind. Eng. Chem., 1927, 19, 1162—1165).—In the production of ethyl alcohol by the fermentation of sugars that result from the acid hydrolysis of wood, only a portion of the total sugar is acted upon by yeast. The residual sugars, consisting largely of pentoses, form about 35% of the total sugar from soft woods and 65% of that from hard woods. By the addition of suitable bacteria together with an excess of calcium carbonate and a nitrogenous material, *e.g.*, yeast, malt sprouts, dried blood, etc., a part of the residue may be made to undergo a second fermentation process, which is complete at 27° in about 10 days, with the formation of a mixture of lactic and acetic acids. The proportions of these acids obtained depend on the kind of sugars present in the liquor, and this varies with the nature of the wood and the extent of the previous alcoholic fermentation. The ratio of lactic to acetic acid is higher for soft woods (average 7.1:1) than for hard woods (average 3:1). Small-scale experiments indicate a yield, in addition to the alcohol, of 12 lb. of acetic acid and 84 lb. of lactic acid per ton of soft wood, the figures for hard wood being 32 lb. and 97 lb. respectively.

F. R. ENNOS.

**PATENTS.**

**Manufacture of yeast.** INTERNAT. YEAST CO., LTD. From FLEISCHMANN CO. (E.P. 277,476, 24.8.26).—A fermenter is charged with wort of a gravity of about 2° Balling containing yeast nutrients sufficient to produce about half the weight of the pitching yeast. The wort is aerated during the fermentation, which is continued at 31—33° until the yeast content increases to about one and a half times the original yeast concentration. At that stage approximately one third of the wort is withdrawn and the yeast separated. The operation is repeated after adding to the fermenter sufficient water and nutrients to replace that withdrawn.

C. RANKEN.

**Simultaneous dehydration and purification of alcohol.** SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (F.P. 617,042, 3.6.26. Belg., 4.6.25).—Acids and esters contained in the alcohol are neutralised and hydrolysed, respectively, in the dehydrating column, or salts or organic acids formed in the process are withdrawn at the base of an auxiliary column.

L. A. COLES.

**XIX.—FOODS.**

**Rôle of phosphates in bread making.** R. A. BARACKMAN and C. H. BAILEY (Cereal Ch.m., 1927, 4, 400—410).—A survey of the work done on the investigation of the effect of phosphates on the colloidal properties of dough, and on yeast activity and reproduction. 0.2—0.5% of acid calcium phosphate

improves the quality of a dough as is shown by the general improvement in baking quality, but further addition tends to impair it. None of the methods so far proposed gives a satisfactory measure of this improving effect. The addition of phosphate results in an increased rate of gas production, not by an increase in the number of functioning yeast cells, but because of the greater activity of each cell in fermentation.

F. R. ENNOS.

**Interpretation of [bread] baking tests.** L. W. HAAS (Cereal Chem., 1927, 4, 389—394).—The method for the production of the test loaves approaches actual baking practice as nearly as possible. Loaves are made from dough fermented for different times in order to gauge the correct fermentation period. A record is kept of the behaviour of the dough during fermentation in order to make any necessary adjustment in the absorption. The important points to consider in judging the test loaves, which are compared with those made from a standard flour, are measured loaf volume, oven spring, shape, bloom, grain, texture, colour of crumb, flavour, and odour.

F. E. ENNOS.

**Determination of fat content of flour and milling stocks.**

C. W. HERD (Cereal Chem., 1927, 4, 370—376).—Less fat is extracted by ether from completely dried flour than from that which is moist or partly dried. The first hour's drying, which removes at least 80% of the moisture, does not affect the amount of fat extractable by ether, but after the removal of the last 1—2% of moisture the amount extracted diminishes with the time of heating. On heating the extracted fat at 98° it becomes darker and less mobile, its refractive index changes from 1.4830 to 1.4890, and it becomes practically insoluble in the ordinary organic solvents. The low extractability of fat from partly dried flour appears to be due not to loss by "steam distillation" or to the formation of an insoluble oxidation product, but rather to polymerisation of the unsaturated acids of the fat. The change in the fat is not responsible for the gain in weight of a flour on heating after it has been dried out, since this occurs both in the extracted and in the non-extracted flour, but it probably accounts for the darkening in colour. A suggested method for the determination of fat in flour consists in drying for 1 hr. at 100°, extracting in a Soxhlet apparatus for 8 hrs. with anhydrous ether, filtering, evaporating to dryness, and drying the fat for 2 hrs. at 100°.

F. R. ENNOS.

**Relationship between various factors and the ash constituents of milk.** R. O. DAVIES and A. L. PROVAN (Welsh J. Agric., 1927, 3, 236—249).—A close relationship was observed between the concentration of calcium and the yield of milk; the chlorine content varied widely, and the amounts of phosphorus and potassium were related to the activity of the glands. Low-protein winter rations should be adjusted to the same phosphorus content as the balanced ration. At the commencement of grazing there was a temporary increase in the concentration of phosphorus and protein.

CHEMICAL ABSTRACTS.

**Buffers of milk and buffer value.** J. H. BUCHANAN and E. E. PETERSON (J. Dairy Sci., 1927, 10, 224—231).—The calculated buffer value of milk varies with the



$p_H$  range; its average value at  $p_H$  8.5–9 is 0.0067. Casein has little effect as a buffer in the region of its isoelectric point, and the phosphates are least among the important buffers of milk. CHEMICAL ABSTRACTS.

**Comparison of the Babcock, Gerber, and Roesse-Gottlieb methods for determining fat in milk and cream.** A. C. DAHLBERG, G. E. HOLM, and H. C. TROY (New York Agric. Exp. Sta. Tech. Bull. 122, 1926, 13–32).—The Babcock and Gerber methods are equally and sufficiently accurate; the Roesse-Gottlieb method is variable. CHEMICAL ABSTRACTS.

**Acid and fat content of [technical] caseins.** J. MARCUSSE and M. PICARD (Mitt. Materialprüf., 1927, 5, 122).—The standard methods of Lunge and Berl, Höpfner and Jaudas, and Ulex for determining the acid content of technical casein give discordant results, and the following is recommended. Casein (5 g.) is mixed into a paste with 5 c.c. of water, and, after 15 min., is ground with quartz sand, extracted with ether, and the extract titrated against phenolphthalein with alcoholic potash solution. A low value is obtained if the treatment with water is omitted. Calculating the acid content as lactic acid is inaccurate, as the free fatty acids, which have 3 times the mol. wt., vary in amount. If the ether solution is extracted 3–4 times with water the lactic acid can be determined by titrating the aqueous solution, and the fatty acids by titrating the ether solution, assuming a mean mol. wt. of 280. The ether solution is now extracted twice with water to remove the soaps, the ether distilled off, and the residue of neutral fats weighed. Analyses of 5 samples of casein gave lactic acid 1.6–3.9%, fatty acids 0.8–4%, and neutral fats 0.8–4.2%. C. J. SMITHELLS.

**Effect of ethylene on the composition and colour of fruits.** E. M. CHACE and C. G. CHURCH (Ind. Eng. Chem., 1927, 19, 1135–1139; cf. B., 1924, 149, 439, 726).—During the colouring of citrus fruits by ethylene (1 pt. of the gas in 5000 pts. of air) there is no apparent change in the composition, as shown by the sucrose content and acidity of the edible portion. Ethylene in a concentration of 1 in 100,000 is a much more effective colouring agent for lemons than 1 pt. of propylene in 5000. In the case of dates no changes are observed which could not be brought about by heat alone. The action of ethylene on persimmons is to accelerate the colouring both inside and outside, to remove the astringency, and to soften the fruit. No satisfactory indications of colouring have been obtained by the ethylene treatment of bananas, tomatoes, pomegranates, and avocados. F. R. ENNOS.

**Preservation of fruits in sulphurous acid solutions.** W. V. CRUESS and A. H. EL NOUTY (Fruit Prod. J. Amer. Vinegar Ind., 1927, 6, No. 11, 18–20).—Corrosion of the container takes place if canned maraschino cherries contain more than 25 mg. of sulphur dioxide per kg. For the preservation of fruits in sulphur dioxide solution, more than 1500 mg. per litre must be present initially. CHEMICAL ABSTRACTS.

**Variations in the chemical composition of cabbage and sauerkraut.** W. H. PETERSON, E. B. FRED, and

J. A. VILJOEN (Canner, 1925, 61, No. 4, 19–21).—Variations according to the variety and maturity of cabbage are: sugar, 3–4.2; total nitrogen, 0.15–0.24; soluble nitrogen, 0.06–0.16; calcium, 0.034–0.057%. With increasing maturity the sugar decreases and the nitrogen increases. Values for sauerkraut are: moisture, 89.6–91.5; sugar, 0.09–0.77; titratable acid as lactic, 1.44–1.93; volatile acid as acetic, 0.28–0.42; non-volatile acid as lactic, 1.13–1.52; ethyl alcohol, 0.29–0.61%. The amino-nitrogen content of the sauerkraut is much higher than that of the cabbage. CHEMICAL ABSTRACTS.

**Determination of total sulphur in [animal] tissues and in foods.** L. LEMATTE, G. BOINOT, and E. KEHANE (Ann. Chim. analyt., 1927, [ii], 9, 296–297).—Ballard's method of evaporation with sodium carbonate, calcination, and extraction of the residue with acidulated water, the sulphur being finally precipitated as sulphate and weighed, gave low values when applied to lentil starch. Accurate results were, however, obtained by a method in which the material was digested with a mixture of perchloric acid and concentrated nitric acid. The oxidation of the organic sulphur is very difficult, and the use of a less concentrated nitric acid may give very low results. G. A. ELLIOTT.

**Crude fibre in food.** E. J. MAGERS (J. Amer. Dietet. Assoc., 1925, 1, 73–77).—Determinations were made of crude fibre and moisture in 23 foods in various conditions. Different samples of the same food showed wide variations. CHEMICAL ABSTRACTS.

**Phosphates in baking powders.** HERD.—See VII.

#### PATENTS.

**Manufacture of nutritional beverages and food products.** F. H. PECK (E.P. 254,724, 30.6.26. U.S., 1.7.25).—A beverage rich in active vitamins is made by a modification of the process of making beer, in which materials containing soluble vitamins, e.g., lucerne, clover bran, vegetables, etc., are added towards the end of the boiling stage, the brew being exposed to ultra-violet rays subsequent to the stages of sterilising and of fermentation. F. R. ENNOS.

**Pectin preparations and manufacture of preserves and jellies.** DOUGLAS PECTIN CORP., Assees. of H. G. LOESCH (E.P. 262,736, 3.11.26. U.S., 10.12.25).—2–10% of powdered pectin is suspended in a saturated sugar solution containing a suitable amount of organic acid, e.g., tartaric or lactic acid, with the addition of flavouring and colouring materials if desired, whereby the pectin is wetted but not dissolved. For use, the above mixture is diluted with hot water or fruit juice to dissolve the pectin, and more sugar is added so that the liquid contains sufficient pectin, acid, and sugar to ensure jelly formation. F. R. ENNOS.

**Vessel for mixing chocolate etc.** H. W. HICKLEY, Assr. to BAKER PERKINS CO., INC. (U.S.P. 1,645,990, 18.10.27. Appl., 19.11.23. U.K., 21.11.22).—See E.P. 203,587; B., 1923, 1054 A.

**Mixing machines for chocolate or other material.** A. SONSTHAGEN (E.P. 278,152, 2.9.26).



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

Contact poisons, especially in the catalytic form-aldehyde process. A. SIEGL (Chem.-Ztg., 1927, 51, 782—783).—Loss of activity of the metallic catalyst used in the oxidation of methyl alcohol to formaldehyde is frequently due to traces of some contact poison in the air, and is detected most readily by control analyses of the issuing gases, being shown by a steady increase in the total percentage of the gaseous carbon compounds (carbon monoxide and dioxide, and methane). Halogen compounds are particularly effective poisons, producing marked loss of activity even when present in the air in such small quantities that their presence cannot readily be detected analytically. If possible, the use of purer air drawn from the open at a distance from the works is recommended, and will soon repay the cost of the necessary air conduit.

A. B. MANNING.

Occurrence of  $\alpha$ -propylene glycol. K. SCHUTT (Oesterr. Chem.-Ztg., 1927, 30, 170—171).—When crude sugar is fermented in the presence of sodium sulphite, glycerol is produced together with a little trimethylene glycol, the latter passing over first when the mixture is vacuum-distilled by means of superheated steam (Connstein and Lüdecke, A., 1919, i, 463). A sample of these first runnings has been converted into the diacetates and carefully fractionated. Approximately 14% consists of  $\alpha$ -propylene glycol diacetate, m.p. 185—187.5°, which gives rise to the  $\alpha$ -dibromide, m.p. 141—143.6°.

E. HOLMES.

Determination of mercury salicylate and lactate by a mercurimetric method. A. JONESCO-MATIU and C. BORDEIANU (Bul. Soc. Chim. România, 1927, 9, 11—16).—The salicylate is oxidised by heating with a mixture of 1 pt. of nitric acid and 2 pts. of concentrated sulphuric acid until the solution is clear. After cooling and diluting, 10% potassium permanganate solution is added until a faint colour persists, followed by a small excess of sodium nitroprussiate. The solution is then titrated with standardised 0.1N-sodium chloride until the muddy colour disappears. Determinations on the pure salts give results ranging from 99.2% to 100.45%. When oxidising the lactate heating is unnecessary, and results of the order 99.49—99.91% are obtained.

F. S. HAWKINS.

Commercial applications of chlorophyll derivatives. F. M. SCHERTZ (Ind. Eng. Chem., 1927, 19, 1152—1153).—Zinc and copper phaeophytins, obtained by substitution of these metals for hydrogen in the derivative resulting from the treatment of chlorophyll with acid, are more stable than the natural chlorophyll. The copper compound is unaffected by acids or alkalis and retains its bright green colour on exposure to light or after being stored in various oils for as long as two years. It is used in foods, soaps, etc. both to hide and to give colour. A water-soluble product, a soapy mass containing a copper salt of chlorophyllin, and alcohol- and oil-soluble products, consisting of fat and wax mixed with copper phaeophytin, are used for colouring purposes. Chlorophyll and its derivatives in the form of phaeophytins exhibit marked therapeutic properties and are used in increasing amount in pharmaceutical preparations.

F. R. ENNOS.

Determination of atropine in pills. O. EHRLSMANN (Arch. Pharm., 1927, 265, 547—549).—Quantities of atropine too small to be determined chemically were determined by the mydriasis produced in cats. Four animals were selected and their sensitiveness to atropine was determined; this varied from 0.00018 to 0.00027 mg. Pills containing known small amounts of atropine were extracted with either warm water or ether, which removed the alkaloid. The latter was separated, dissolved in water, and the aqueous solution diluted until a drop (27 mg.) no longer produced mydriasis. The amount of atropine extracted was then determined from the volume of solution. The results obtained were never below 60%. The ethereal and aqueous extracts gave identical results.

S. COFFEY.

Isolation of nicotine from *Nicotiana attenuata*, Torr. J. F. COUCH (Amer. J. Pharm., 1927, 99, 519—523).—The isolation and chemical and pharmacological identification of nicotine from *N. attenuata* is described. Determination of the total alkaloid, calculated as nicotine, by Young's method (B., 1927, 123) gave the following amounts in the dry samples: leaves 1.45%, stems 0.48%, roots 0.25%. Non-volatile alkaloids were absent. The lethal dose of nicotine injected intraperitoneally in guinea pigs is 17—20 mg./kg.

E. H. SHARPLES.

Chemical test of nicotine dusts. R. M. HIXON and C. J. DRAKE (Iowa State Coll. J. Sci., 1927, 1, 373—377).—Thatcher and Streeter's method (New York Agric. Exp. Sta. Bull., 1923, No. 501, 3) is untrustworthy, since it measures the rate of evolution of nicotine vapour plus the rate of decomposition of the nicotine. With bentonite as a carrier, 23—86% of the nicotine was unaccountably lost in 24—48 hrs.; a residual oil may be oxynicotine. The concentration of nicotine vapour produced is approximately proportional to the nicotine content of the dust except with magnesium carbonate as a basis, when evaporation is hindered. Nicotine is more toxic than hydrogen cyanide.

CHEMICAL ABSTRACTS.

Ergot oil. H. MATTHES and P. SCHÜTZ (Arch. Pharm., 1927, 265, 541—546).—The oil from *Secale cornutum* of German origin had  $d_{20}^{20}$  0.9210,  $n_D^{20}$  1.4694,  $n_D^{40}$  1.4623, iodine value 70.09, saponif. value 195.4,  $[\alpha]_D^{25} + 10.5^\circ$ , unsaponifiable matter 1.01%. The mixed fatty acids, consisting of 28.79% of solid and 70.24% of liquid acids, had m.p. 40.5—41°,  $n_D^{40}$  1.4550, iodine value 70.46,  $[\alpha]_D^{25} + 2.74^\circ$ , mol. wt. 281.7, acetyl value 69.82 (acetyl value of ethyl esters 73). The solid fatty acids consisted of palmitic acid and higher homologues. The liquid acids, consisting of 50% of hydroxyoleic, 45% of oleic, and 5% of linoleic acids, on bromination and treatment with light petroleum, b.p. 30—60°, furnished an insoluble, liquid dibromohydroxyoleic acid, which afforded hydroxyoleic acid,  $n_D^{20}$  1.4721,  $n_D^{40}$  1.4684, mol. wt. 298.6, iodine value 85.6, acetyl value 142.9, on reduction with platinised zinc. The petroleum solution of bromo-acids contained only linoleic acid tetrabromide, m.p. 114°, and dibromo-oleic acid. Another sample of oil of Austrian origin gave,  $d_{20}^{20}$  0.9170,  $n_D^{20}$  1.4710,  $n_D^{40}$  1.4642,  $[\alpha]_D^{25} + 10.6^\circ$ , iodine value 66.6, unsaponifiable matter 1.037%. The fatty acid mixture contained 27.1% of solid and 71.2% of liquid acids, the latter furnishing the hydroxyoleic acid with an acetyl value of 144.6.

S. COFFEY.



**Genus *Mentha*. XIII. Oil of *Mentha piperita*, L., produced in 1924.** S. M. GORDON (Amer. J. Pharm., 1927, 99, 524—530).—An oil from *Mentha piperita*, L., had  $d^{25}_D$  0.9088,  $n$  1.465,  $\alpha$  —13.25°, acid value nil, ester value 29.22, ester value after acetylation 154.7, esters 10.32%, total menthol 49.52%, combined menthol 6.46%, free menthol 43.10%. Only 13% of the free menthol could be separated from the oil by freezing. The isolation of *d*-menthone, *l*-menthone, *d*-pulegone, *l*- $\alpha$ -pinene, and a terpinene is described. Phellandrene and limonene were absent.

E. H. SHARPLES.

**Properties and constituents of an oil extracted from the seeds of *Digitalis purpurea*.** I. S. MEL-LANOFF (Amer. J. Pharm., 1927, 99, 549).—The amber-coloured, semi-drying oil from the seeds of *D. purpurea* has  $n^{20}_D$  1.4755,  $d^{15.5}_D$  0.9231, acid value 9.30, saponif. value 207.5, ester value 198.2, unsaponifiable matter 6.12%, soluble fatty acids 1.66%, insoluble fatty acids 90.0%, hydroxy-acids nil, liquid acids 75.8% ( $n^{20}_D$  1.4670), solid acids 9.2% ( $n^{20}_D$  1.4685), and iodine value (Hanus) 127.9.

E. H. SHARPLES.

**Methyl alcohol by fermentation.** WOODRUFF. **Lactic and acetic acids by fermentation.** MARTEN and others.—See XVIII.

#### PATENTS.

**Concentration of dilute aqueous formaldehyde solutions.** M. MUELLER, Assee. of A. ZIMMERLI (Can. P. 264,342, 10.9.24).—The dilute solution is heated under reflux until equilibrium is reached between formaldehyde, its polymerides, and hydrates, when the mixture is fractionated.

B. FULLMAN.

**Manufacture of alcohols.** R. B. MACMULLIN and R. E. GEGENHEIMER, Assrs. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,641,544, 6.9.27. Appl., 27.8.24).—An alkyl, e.g., methyl, chloride is passed with steam (1 mol.) over calcium hydroxide at 400° and a pressure of 13 atm., the time of contact being about 40 sec.

T. S. WHEELER.

**Manufacture of alkyl sulphates.** R. H. MCKEE (U.S.P. 1,641,005, 30.8.27. Appl., 21.5.23. Renewed 13.7.27).—An aliphatic alcohol, e.g., ethyl alcohol, is treated with sulphuryl chloride (1 mol.) at below 30°, excess of chloride being removed by distillation.

T. S. WHEELER.

**Nitration process and nitrating mixture.** G. B. TAYLOR and A. S. RICHARDSON, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,640,737, 30.8.27. Appl., 15.7.22).—The solution of nitric and nitrosylsulphuric acids in sulphuric acid, obtained by treatment of sulphuric acid with nitrogen peroxide, can be employed to nitrate organic compounds, e.g., benzene. The spent acid, consisting chiefly of nitrosylsulphuric and sulphuric acids, is treated with steam or sulphur dioxide to form nitric oxide, which is re-oxidised to the peroxide.

T. S. WHEELER.

**Production of tertiary nitriles.** I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (E.P. 253,950, 22.6.26. Ger., 22.6.25).—Nitriles of the type  $\text{CHR}_3\cdot\text{CN}$  are alkylated etc. by means of sodium and an alkylating agent.  $\alpha$ -Ethyl-*n*-

butyronitrile,  $\text{CHET}_2\cdot\text{CN}$ , is treated with sodium or potassium in dry ether, benzene, xylene, etc. and the product is warmed with allyl bromide to form  $\alpha$ -diethyl- $\Delta^7$ -*n*-pentenonitrile, b.p. 78°/9 mm., which may be hydrolysed to the amide, m.p. 74°. Benzyl bromide similarly gives  $\alpha$ -benzyl- $\alpha$ -ethyl-*n*-butyronitrile, b.p. 120—122°/1 mm. (amide, m.p. 75°). C. HOLLINS.

**Increasing the yield of civetone, starting from civet.** M. NAEF and F. FIRMENICH (M. NAEF & CIE.) (E.P. 267,893, 20.1.27. Switz., 17.3.26).—The civetol which accompanies civetone in civet may be oxidised to civetone by usual oxidation methods. E.g., civet is hydrolysed with alkali and the neutral oil, with or without previously removing civetone as semicarbazone, is oxidised with chromic acid, or catalytically over a metal catalyst at 250—300° in vacuo. C. HOLLINS.

**Manufacture of aqueous emulsions of paraffin for use as medicines.** E. GLÜCKSMANN (E.P. 255,456, 13.7.26. Ger., 14.7.25).—Stable, stiff emulsions of paraffin in water are formed in the presence of vegetable gums and algæ as joint emulsifying agents. E.g., a 70% emulsion is formed by the addition of 70 kg. of paraffin to a solution containing 1 kg. of agar-agar, 25 kg. of water, 0.5—1 kg. of gum arabic swelled in water, 6—8 kg. of sugar, and 1 kg. of swelled gelatin.

B. FULLMAN.

**Production of complex aurothiosulphate compounds.** E. and J. KEIDING (U.S.P. 1,640,775, 30.8.27. Appl., 3.5.26. Den., 7.11.25).—Aurous bromide or iodide dissolved in aqueous-alcoholic sodium bromide or iodide solution is treated with sodium thiosulphate to form sodium aurothiosulphate, which is precipitated by addition of alcohol.

T. S. WHEELER.

**Manufacture of solutions of bismuth salts of arylarsinic acids.** A. HAYTHORNTHWAIT, and MAY & BAKER LTD. (E.P. 277,774, 16.7.26).—The insoluble bismuth arylarsinate is converted into a soluble double salt by addition of an alkaline base (ammonia, piperazine, etc.) and the solution is stabilised by means of an organic salt such as a tartrate or citrate; or the bismuth arylarsinate may be dissolved in tartaric or citric acid solutions with addition of the alkaline base. The bismuth arylarsinate may be formed *in situ*, e.g., from a bismuthyl-tartrate or -citrate and a soluble arylarsinate, and the alkaline base and stabilising salt then added. A mixture of bismuth 3-acetamido-4-hydroxybenzene-arsinate with 1 pt. of piperazine hydrate, 2 pts. of sodium potassium tartrate, and 5 pts. of water, nearly neutralised by addition of 0.35 pt. of 80% acetic acid, and made up to 100 pts. with water, gives a solution which can be sterilised at 110°.

C. HOLLINS.

**Manufacture of new formyl derivative of 2-[hydr]oxy-4-aminophenylarsinic acid [4-form-amido-2-hydroxybenzene-arsinic acid] and salts thereof.** ÉTABL. POULENC FRÈRES, and E. FOURNEAU (E.P. 277,586, 7.6.27. Fr. 29.10.26).—4-Amino-2-hydroxybenzene-arsinic acid is boiled with 98% formic acid for 3 hrs., giving 4-formamido-2-hydroxybenzene-arsinic acid (sodium and ammonium salts described).

C. HOLLINS.



**Anti-spasmodics.** F. LEUCHS, Assr. to WINTHROP CHEMICAL Co., INC. (U.S.P. 1,640,506, 30.8.27. Appl., 16.12.25. Ger., 24.3.25).—*o*-Methylbenzyl carbamate, m.p. 86°, *p*-methylbenzyl carbamate, m.p. 122°, and  $\alpha$ -phenyl-*n*-propyl carbamate, m.p. 79°, are readily prepared by the usual methods. T. S. WHEELER.

**Fixing agent for perfumes, volatile solvents, etc.** J. D. RIEDEL A.-G. (G.P. 441,630, 21.1.26).—The material consists of the product obtained by the saponification with alkali hydroxides of wholly or partially hydrogenated or polymerised sperm oil or the like.

L. A. COLES.

**Manufacture of the active principle of lobelia.** H. WIELAND, Assr. to C. H. BOEHRINGER SOHN CHEM. FABR. (Re-issue 16,772, 18.10.27, of U.S.P. 1,505,181, 19.8.24).—See B., 1925, 26.

**Manufacture of calcium salts of inositol-phosphoric acid.** A. GAMS and M. GIRARD, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,645,233, 11.10.27. Appl., 9.4.23).—See E.P. 218,014; B., 1924, 731.

**Manufacture of organic compounds (E.P. 277,394).**—See III.

**cycloTrimethylenearylpyrazolones (E.P. 260,577 and 263,773).**—See IV.

**Acetic acid mother liquors (E.P. 266,684).**—See V.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Action of potassium permanganate on the photographic plate, and exceptions to the law of reciprocity.** J. HRDLÍČKA (Compt. rend., 1927, 185, 711—713).—An acid solution of potassium permanganate (0.2%) has been used to remove the sensitised grains of silver from an exposed and developed photographic plate. The rate of dissolution varied with the density of the plate. Treatment with the solution before exposure and development produced a decrease in the sensitiveness of the plate and in gamma, but pre-development tended to neutralise its effect. Treatment between exposure and development completely destroyed the latent image. A neutral solution, however, enabled those grains to be developed which had been rendered non-developable by the initial exposure. The acid solution is considered to destroy the silver sulphide "germs" (Sheppard), the latent image being formed partly from metallic silver. For the emulsion studied, gamma decreased with an increase in the illumination.

J. GRANT.

**Mathematical methods of frequency analysis of size of particles. II. Application to silver bromide precipitates.** R. P. LOVELAND and A. P. H. TRIVELLI (J. Franklin Inst., 1927, 204, 377—389).—Thirty-three precipitates of silver bromide have been prepared which varied from each other only in two carefully-controlled factors. Size-frequency determinations of single layer plates made from these precipitates were made and analysed by the graphical method previously described (cf. B., 1927, 688). From these determina-

tions it is concluded that in a chemical precipitation of silver halide the whole distribution of particles is not governed by a single-frequency law. W. E. DOWNEY.

**Colour of light sources and reflecting power of fabric.** CUNLIFFE and FARROW.—See V.

## PATENTS.

**Manufacture of photographic silver halide emulsions.** I. G. FARBENIND. A.-G., Asses. of A.-G. F. ANILIN-FABR. (E.P. 259,926, 16.8.26. Ger., 14.10.25. Addn. to E.P. 258,237; B., 1927, 861).—Decomposition products of animal or vegetable proteins, prepared by acid hydrolysis, are added to a silver halide emulsion or to its constituents in order to bring about an increase in sensitiveness. Sensitiveness can be increased 30 times by adding a solution of products obtained by hydrolysing gelatin or other protein with about 1% nitric acid, the dissolved matter in the solution amounting to only about 2.5% of the amount of gelatin in the emulsion. W. CLARK.

**Protective layers for photographic films.** I. G. FARBENIND. A.-G. (E.P. 270,347, 2.5.27. Ger., 1.5.26).—Cellulose ether esters or mixed esters are used for the production of protective layers on the backs of photographic films. Cellulose acetate phosphate, applied in solution in  $\beta$ -methoxyethyl acetate, protects against electric discharges; ethylcellulose stearate, applied in alcohol-benzene-acetone solution, diminishes sliding friction in cinematograph films; cellulose acetate nitrate (2—3% N), applied in alcohol-benzene-acetone solution with a little monoacetin, prevents accumulation of electrostatic charges. C. HOLLINS.

**Manufacture of a shadow-producing agent.** B. RAPP (U.S.P. 1,644,446, 4.10.27. Appl., 17.5.26. Ger., 19.1.25).—Excess of moisture is removed from freshly-precipitated barium sulphate, and finely-divided sugar is mixed intimately therewith, the mixture is then spread in a thin layer, dried at 70—100°, and finely powdered by passing through a sieve. Such a preparation may consist of 100 pts. of barium sulphate, 12.5 pts. of cane sugar, and gum tragacanth. W. G. CAREY.

**Photographic copying by the reflection method.** A. MILLER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,645,112, 11.10.27. Appl., 26.3.26. Ger., 14.11.24).—See E.P. 243,023; B., 1926, 997.

## XXII.—EXPLOSIVES; MATCHES.

**Sensitivities of detonating compounds to frictional impact, impact, and heat.** C. A. TAYLOR and W. H. RINKENBACH (J. Franklin Inst., 1927, 204, 369—376).—The frictional impact necessary to detonate various compounds has been determined by means of a pendulum friction machine. The frictional impact, impact, and heat necessary to explode twenty various detonating compounds have been determined. The results show that there does not exist a direct relationship between the values for different substances by any one test. It is concluded, however, that sensitivity



is not specific towards any one form of physical influence.

W. E. DOWNEY.

#### PATENTS.

**Acceleration of gelatinisation of cellulose nitrate.** R. C. MORAN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,640,712, 30.8.27. Appl., 19.6.24).—Gelatinisation of nitrocellulose by nitroglycerin is accelerated by addition of a small proportion of an aliphatic alcohol or glycol.

T. S. WHEELER.

**Explosive.** P. R. DE WILDE, and Soc. Suisse des Explosifs (Swiss P. 117,568, 12.9.25).—An explosive, in the form of an oil (which can be used for the preparation of dynamite etc.), is obtained by the nitration of a mixture of glycerol and ethylene glycol.

B. FULLMAN.

**Safety explosives.** A. B. M. GUILHON (F.P. 612,790, 15.3.26).—To chlorate explosives is added a polypicrate, obtained by suitable combination of picric acid with the carbonates of lead, calcium, copper, and magnesium.

B. FULLMAN.

**Explosives containing hygroscopic materials.** E. BESSON (F.P. 617,285, 23.10.25).—The particles of the explosive are covered with a thin layer of a dry, pulverulent or colloidal mineral substance, *e.g.*, kaolin, water-glass, etc.

B. FULLMAN.

### XXIII.—SANITATION; WATER PURIFICATION.

**Dissolved oxygen absorption test.** II. E. A. COOPER and W. H. READ (J.S.C.I., 1927, 46, 413—414 *τ*; cf. B., 1927, 381).—The presence of small amounts of sodium permolybdate considerably increases the rate of dissolved oxygen absorption by sewage effluents. The oxygen absorption in two days in presence of the permolybdate tends to be of the same order as that observed in five days in absence of this catalyst. The preliminary results suggest the possibility of completing the Winkler dissolved oxygen absorption test in 48 hrs., instead of 5 days.

**Water softening as practised at Oberlin, Ohio.** W. H. CHAPIN (Ind. Eng. Chem., 1927, 19, 1182—1187).—The water is softened by the lime-soda process, and methods of calculation of the amounts of chemicals required are given and reproduced in the form of graphs. After-precipitation of colloidal suspensions or of supersaturated solutions caused considerable trouble in the sand filters and in the mains even after 18 hrs.' settlement and filtration. The trouble was somewhat lessened by the addition of slightly basic alum (2% excess of  $\text{Al}_2\text{O}_3$ ) at the rate of 30 pts./million. Later it was found that storage for about 4 weeks renders the water stable and prevents incrustation of pipes etc. even without filtration, the time factor and not re-carbonation being responsible. Long storage is much more effective than sand filtration for preventing after-precipitation.

W. G. CAREY.

**Determination of sulphuric acid in drinking water by use of benzidine.** F. RASCHIG (Z. angew. Chem., 1927, 40, 864).—Haase (B., 1927, 798) is mistaken

in condemning Raschig's method (A., 1906, ii, 306) for determining sulphuric acid in such small proportions as are present in drinking water. Provided the prescription is exactly followed (the benzidine solution should contain 40 g. of benzidine per litre) and the quantity of water taken is adjusted to the sulphuric acid present, the results obtained are accurate.

#### PATENTS.

**Production of disinfecting agents.** A. BOEHRINGER (C. H. BOEHRINGER SOHN) (E.P. 253,918, 16.6.26. Ger., 20.6.25).—Lactates or alkalis are added to lactic acid until the  $p_{\text{H}}$  value is between 3.25 and 4.2, or lactates are treated with acid, *e.g.*, sulphuric acid, to give the requisite hydrogen-ion concentration. Nutrient media such as peptone or carbohydrates, odoriferous material such as pine oil, or cultures of lactic acid bacteria may be added.

W. G. CAREY.

**Volumetric displacement apparatus for controlling the supply of gas for chlorinating water etc.** B. BRAMWELL (E.P. 277,869, 16.3.27).—A displacement vessel closed at the top and open at the bottom is contained in a closed water vessel and carries a U-shaped vent tube, one end of which projects outside the vessel, the other end being enlarged to give equal and positive displacements of gas without bubbling. The U-tube is so disposed that the bottom is above the lower edge of the displacement vessel.

W. G. CAREY.

**Prevention of boiler-scale formation.** LA SUVAPO (Soc. ANON.) (F.P. 617,870, 6.11.25).—To the boiler feed-water are added a cerium salt susceptible to oxidation, tannin, and an alkali, *e.g.*, a mixture of 3% of cerous sulphate, 37% of tannin extract (*d* 1.2), and 60% of sodium carbonate.

B. FULLMAN.

**Method of diminishing the internal rusting of hot-water apparatus.** C. HÜLSMEYER (Swiss P. 117,715, 7.7.25).—The entrapment of air in the feed water to boilers and other hot-water apparatus is rendered harmless by passing the water through a vessel packed with manganese steel wool, which removes the dissolved oxygen.

A. R. POWELL.

**Softening of water by base exchange.** K. MORAWE (Swiss P. 117,570, 26.2.25. Ger., 25.3.24).—The water is treated with a moulded, alkaline, base-exchange softening material capable of regeneration. The treatment is such that the water only acts on the outer layers of the pieces, which may then be regenerated in a shortened time.

B. FULLMAN.

**Utilisation of zeolites [for water-softening].** W. H. GREEN, Assr. to GENERAL ZEOLITE Co. (U.S.P. 1,644,469, 4.10.27. Appl., 8.6.23).—Hard water is passed continuously upwards through a portion of a bed of zeolite, and a regenerating solution is passed intermittently downwards through another portion of the zeolite, and is washed out, after which particles of the bed are transferred from each portion to the other.

W. G. CAREY.

**Impregnating compounds** (E.P. 277,083).—See XIII.