

B.—APPLIED CHEMISTRY

NOV. 7 and 14, 1930.*

I.—GENERAL; PLANT; MACHINERY.

Heat losses from plant surfaces. R. M. COLLES (Trans. Ceram. Soc., 1930, 29, 182—190; cf. B., 1929, 913).—Steel cylinders were heated at 230° or at as high a temperature as the surface coating would permit, *i.e.*, at temperatures at which convection forms the major source of loss, and the rate of loss of heat was plotted against the temperature for various coatings and weather conditions. Since radiation loss varies as the difference between the fourth powers of the absolute temperatures of the object cooling and that of its surroundings, and convection loss is proportional to the 1.25th power of the difference in temperature, it is possible to separate the two losses. It is found that radiation is greatly affected by the nature of the surface and is unaffected by the weather, whilst convection is almost *vice versa*. The formulae and graphs are convenient to use, on the assumption that the surface temperature can be first estimated and the increased convection loss caused by wind and decreased by shielding is demonstrated. The shape of the object is not very important unless it is so re-entrant that pockets of still air are formed or radiation to itself takes place. A wind of 10 m.p.h. increases the convection loss to 2½—3 times that in still air. B. M. VENABLES.

Application of Osborne Reynolds' theory of heat transfer to flow through a pipe. G. I. TAYLOR (Proc. Roy. Soc., 1930, A, 129, 25—30).—Mathematical. The work of Eagle and Ferguson (B., 1930, 843) is criticised on the ground that Reynolds' theory suffers from two possible sources of error, and that even if the theory is true, the experiments were carried out under conditions to which it is not applicable. Moreover, Eagle and Ferguson's method of measuring mean temperature at any section of their pipe is inadmissible. L. L. BIRCUMSHAW.

Factors affecting the problem of smoke prevention. W. E. GIBBS (J. Inst. Fuel, 1930, 3, 361—375).—Coal smoke is formed partly by condensation (carbon particles) and partly by mechanical dispersion (ash particles). Screen analyses of particles from chain-grate stokers and powdered-fuel firing are given. The latter are the smaller, and the residual particles passing through a centrifugal separator show 97% below 75 μ . The total amount may be 6 g./m.³ of solids in such a smoke, and whilst the larger particles may be removed by gravity or centrifugal force, they do not carry the smaller particles with them. The problem of smoke prevention is that of the flocculation of the smaller particles. It is shown that with turbulent motion in gas flues there is no possibility of gravity settlement of particles of less than

500 μ diam. Centrifugal separators readily collect particles down to 50 μ . The most efficient types combine a high radius of curvature with a short transverse path for the particles. Perforated plates with staggered perforations give this condition, but the power consumption is too high for power-station practice. The efficiency of all separating methods is greatly increased by wetting the particles. Spray-washing is much more effective than bubbling, but smokes are much more difficult to wash than gases as the particles move more slowly than gas molecules. The degree of dispersion of the spray and the "wettability" of the smoke particles are both important. The latter quality may be modified by lowering the surface tension of the water or by electrical charges. The electrostatic flocculation of smokes is also discussed. The cyclone type of separator is the most satisfactory apparatus known at present, but it would be desirable for it to be supplemented by some simple method of flocculating the finer particles. C. IRWIN.

Correlation of sieving analyses. H. HEYWOOD (J. Inst. Fuel, 1930, 3, 428—432).—Results of sieving analyses depend on variations in procedure, time of sieving, and variation in aperture with the diameter of the sieve wire. Curves are obtained giving conversion factors for use in the sieving of coal dust with increase in time of sieving, weight on sieve, and slight variations in aperture. The true apertures in microns of a number of standard sieves are given. No standard exists for silk sieves, in which the aperture depends on the strength of the strands, and very high tolerances are necessary for the finer wire sieves. C. IRWIN.

Electrical determination of the dew point of flue gases. H. F. JOHNSTONE (Univ. Ill. Eng. Exp. Sta. Circ., 1929, No. 20, 22 pp.).—A method based on the variation of the electrical conductivity of the surface of a dielectric when a film is condensed on it is described. Minimal concentrations of sulphur trioxide exert a profound effect on the dew point.

CHEMICAL ABSTRACTS.

Determination of the capacity of a large vessel. F. W. J. CLENDINNEN (J. Soc. Chem. Ind. Victoria, 1930, 30, 361).—The vessel is filled to the mark with water and the chloride content of this determined before and after the addition of a relatively small measured volume of saturated brine solution by Cavanagh's electrometric titration method (A., 1927, 1045; 1928, 607). The capacity is calculated from these data.

H. I. DOWNES.

Town's gas for heat-treatment furnaces. WALTER. —See II. **Wetting of powders.** HARKINS and DAHLSTROM.—See XIII.

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

PATENTS.

Retort furnace. C. V. McINTIRE, Assr. to CONSOLIDATION COAL PRODUCTS Co. (U.S.P. 1,757,644, 6.5.30. Appl., 20.8.25).—The heat-transmitting wall is semi-cylindrical with deep circumferential corrugations and is made of easily renewable metallic sections. The coal or other material under treatment is conveyed through the trough by stirrers which oscillate through about 270°.

B. M. VENABLES.

Heating furnace [for viscous fluids, e.g., tar etc.]. H. A. DREFFEIN (U.S.P. 1,757,707, 6.5.30. Appl., 30.3.28).—To avoid local overheating of the fluid in a tubular, direct-fired heater, the burning gases are caused to pass upwards through a free space between two nests of tubes, heating them only by radiation; the partially cooled gases are then passed in contact with the tubes.

B. M. VENABLES.

[Regulation of] dry kilns. M. B. HALL, Assr. to FOXBORO Co. (U.S.P. 1,757,629, 6.5.30. Appl., 7.9.27).—Wet- and dry-bulb thermometers containing a volatile liquid are inserted in the kiln, and the vapour pressure of the thermometric liquid directly actuates valves controlling, respectively, water sprays and heating steam. The "bulb" of the dry thermometer is in the form of a long tube extending along the full length of the kiln, so that the maximum temperature at any point will be the controlling one.

B. M. VENABLES.

Heat-insulating material. G. B. PEEBLES (U.S.P. 1,757,470, 6.5.30. Appl., 8.8.28).—Sheets for building-board or insulating purposes are constructed from the precipitated waste products of the ammonia-soda process, which are dried, mixed with magnesia and sodium silicate, and pressed at about 20,000 lb./in.²

B. M. VENABLES.

Grinding mill. G. F. PETTINOS (U.S.P. 1,758,010, 13.5.30. Appl., 18.8.28).—The mill is of the disintegrator type with rigid hammers and a vertical shaft, and with a number of grinding compartments, increasing in diameter downwards, which are separated from each other by annular partitions.

B. M. VENABLES.

Attrition mill. A. SCHUCHARDT (U.S.P. 1,758,539, 13.5.30. Appl., 2.4.29).—Means are described for feeding material through apertures close to the centre of one disc of a disc grinder in which each disc is rotated by a separate motor.

B. M. VENABLES.

Disintegration of [cellular] matter. W. KOEHLER (U.S.P. 1,746,731, 11.2.30. Appl., 21.1.29).—Cellular material of any kind, e.g., oils, fats, vegetable products, or living organisms, is broken down and the material sterilised by subjecting it to the very high pressure and radiant heat produced by an explosion, the pressure being then suddenly, but not necessarily immediately, reduced to a low figure by exhausting into a vacuum chamber. For the sterilisation of milk, hydrogen and oxygen in atomic proportions would be used; for fruit juices it is better to use carbon oxysulphide and oxygen in proportions to yield sulphur dioxide and carbon dioxide.

B. M. VENABLES.

Apparatus for separating comminuted matter. R. ANDERSON (U.S.P. 1,758,422, 13.5.30. Appl., 12.6.28).—The apparatus, particularly suitable for

separating precious metals from sweepings, comprises a vertical tube with closed bottom and open top (guarded by a dust bag) and divided at an intermediate point by a collar supporting a screen underneath which is an air inlet. At the commencement of operations the material is laid on the screen, the apparatus is then assembled, and the separation effected by puffs of air.

B. M. VENABLES.

Mixer. J. JOHNSON, Assr. to TURBO-MIXER CORP. (U.S.P. 1,757,197, 6.5.30. Appl., 14.11.28).—A bladed impeller is surrounded by stationary deflecting blades which support a screen or other disintegrating means.

B. M. VENABLES.

Wet-mixing method and apparatus. F. D. PFEFFER and F. TROTTER, Assrs. to UNITED STATES GYPSUM Co. (U.S.P. 1,758,200, 13.5.30. Appl., 19.3.26).—A proportioning and mixing device is described in which the actual mixing is effected by a centrifugal disc provided with pins.

B. M. VENABLES.

Obtaining solids from liquids. A. B. JONES, Assr. to INDUSTRIAL ASSOCIATES, INC. (U.S.P. 1,758,745, 13.5.30. Appl., 5.8.26).—The liquid is subjected to spray-drying in two stages, viz., concentration and solidification, at different temperatures and in the presence of different quantities of gases. Preferably, moderately heated gas is supplied to the solidification stage and exhausted gas therefrom together with any entrained dust to the concentration stage, where an additional quantity of more highly heated gas is supplied in such a way that it meets the raw liquor spray before mingling with the already used gas; the exhaust, however, is common to both quantities.

B. M. VENABLES.

[Self-balancing] centrifugal dryer. W. V. ORR (U.S.P. 1,757,585, 6.5.30. Appl., 6.5.29).—An outer basket is rigidly attached to the shaft, and is arranged to retain some liquid, but the inner basket (containing the material) is of spherical or other bulged shape, and is permitted a limited movement on spherical seats; when it deflects owing to uneven loading it will displace liquid from the outer basket on the heavy side, thus restoring the balance.

B. M. VENABLES.

Rotating filter. E. B. GOODWIN (U.S.P. 1,758,910, 13.5.30. Appl., 24.7.28).—A hollow, truncated cone is rotated about a horizontal axis by a hollow shaft on one end of which, beyond the cone, driving means is provided, and on the other end is a feed scoop. The interior of the cone is fitted with an interrupted helix tending to carry material up to the small end. The feed apertures in the shaft are some distance within the filter, and nearer the small end is a supply of wash-liquor, the large end being closed by filter medium.

B. M. VENABLES.

Multi-disc suction filter. V. C. BENJAMIN and P. W. PRUTZMAN, Assrs. to CONTACT FILTRATION Co. (U.S.P. 1,757,355, 6.5.30. Appl., 4.1.28).—The filter discs lie in a single vertical plane, and the hollow horizontal shaft is used for the application of vacuum only. Automatic ball valves, placed in the short spokes between the discs and the shaft, serve to cut off the vacuum as a disc with nearly dry cakes reaches the top of its travel; the cakes are then scraped off from each side of the disc.

B. M. VENABLES.

Filter mat. R. STRINDBERG (U.S.P. 1,757,690, 6.5.30. Appl., 16.3.28).—A filter suitable for cleaning air or gas is constructed of a stiff wire bent zig-zag to form a frame, around which is entwined a strand of wire-cored wool or other suitable substance.

B. M. VENABLES.

Pressure distillation. N. E. LOOMIS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,757,579, 6.5.30. Appl., 16.5.22).—The vapours of, *e.g.*, oil derived from a still are passed through a horizontal drum with inclined shelves down which condensed light oils and feed liquor trickle from another drum above. In the uppermost drum the vapours are bubbled through the light and feed oils, and finally exhaust through a condenser. The top drum may have a cooling coil, and is fed with fresh oil equal in amount to that of the distillate.

B. M. VENABLES.

De-aeration and evaporation of liquid. W. S. ELLIOTT (U.S.P. 1,758,566, 13.5.30. Appl., 27.3.23).—The liquid, *e.g.*, raw feed-water, is heated by splashing over trays in the presence of steam in a vessel at a pressure and temperature corresponding to that of the steam; the liquid falls to a flash boiler, which is a similar vessel maintained at a lower pressure so that a small proportion flashes into steam and carries with it all the dissolved gases. The vapour and gases pass to a condenser-evaporator where the vapour is condensed and runs back to the flash boiler while the permanent gases only are withdrawn by an ejector or other device. The cooling-water side of the condenser is provided with a vapour space, this being maintained at a still lower pressure so that part of the cooling water is evaporated, the vapour from which is condensed in another heat exchanger, yielding distilled water and preheating the raw water. A modification is described by which the yield of distilled water may be increased by applying more heat indirectly to the condenser-evaporator.

B. M. VENABLES.

Viscosimeter and display device. W. D. SMITH (U.S.P. 1,758,677, 13.5.30. Appl., 15.6.25).—A pair of evacuated test tubes are about half filled with the two liquids to be compared, *e.g.*, fresh and used oil; midway of the tubes is a diaphragm with a small aperture through which the oil will flow in a thin stream when the tubes are inverted. Another pair of tubes to demonstrate dirt in used oil may also be mounted in the same frame.

B. M. VENABLES.

Dialysing apparatus. E. HEIBIG (U.S.P. 1,757,364, 6.5.30. Appl., 13.6.28. Holl., 2.2.27).—Cf. B.P. 329,235; B., 1930, 694.

[Pulverised fuel] furnace walls. O. NYGAARD (B.P. 313,875, 6.6.29. U.S., 18.6.28).

Refrigerators. N. V. MIDDEN-EUROPEESCHE OCTROOI-MAATS. (B.P. 319,270, 27.8.29. Switz., 19.9.28).

Absorption refrigerating systems. ELECTROLUX, LTD., Assees. of A. LENNING (B.P. 319,015, 14.9.29. U.S., 15.9.28).

Absorption machines for producing cold or heat. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 313,618, 14.6.29. Ger., 16.6.28).

Evaporator for cooling plants [refrigerators].

O. FRH. VON KOHORN (O. KOHORN & Co., MASCHINENFABR.) (B.P. 334,535, 4.6.29. Austr., 6.3.29).

Absorption process (U.S.P. 1,748,051).—See II. **Carrying out reactions under pressure** (U.S.P. 1,746,687).—See III. **Humidity-control apparatus** (U.S.P. 1,757,931).—See V.

II.—FUEL; GAS; TAR; MINERAL OILS.

Hardness, abrasability, and reactivity of coke.

R. A. MOTT (Fuel, 1930, 9, 400—411).—From a comparison of the results of shatter tests with those of an abrasion test carried out by rotating 2—3-in. coke in a smooth-walled drum under standard conditions it is concluded that the $\frac{1}{2}$ -in. shatter index can be used as a measure of the "abrasion hardness" of a coke. A good blast-furnace coke should possess a low abrasability, *e.g.*, a $\frac{1}{2}$ -in. shatter index of not less than 97, combined with a sufficiently high "impact hardness," a measure of which is given by the $1\frac{1}{2}$ -in. shatter index. The relationship between the $\frac{1}{2}$ -in., 1-in., $1\frac{1}{2}$ -in., and 2-in. shatter indices is discussed, and the "normal" relationship, based on the figures for a large number of cokes tested by the Midland Coke Research Committee, is illustrated diagrammatically. Cokes made in fast-coking (14-in.) ovens yield abnormally low 2-in. shatter indices; cokes made by blending coking coals with non-coking materials yield abnormally high 2-in. shatter indices. The standard shatter-test apparatus adopted by the Midland, Northern, and Scottish Coke Research Committees is described. The reactivity of coke has been determined in a combustibility furnace of 1 sq. ft. grate area with a 12-in. bed of coke of standard size. The coke is blown with a specified amount of air and when a steady temperature has been attained gas samples are taken at different levels; the distances above the grate at which (a) the oxygen has just disappeared (reactivity towards oxygen) and (b) the concentration of carbon monoxide reaches 20% (reactivity towards carbon dioxide) are recorded. Different cokes differ little in reactivity at high temperatures. Marked reactivity is not generally desirable in a blast-furnace coke, since it is usually accompanied by low combustion temperatures. Decrease in coke size increases reactivity and decreases the average combustion temperature.

A. B. MANNING.

Briquetting of coal slack without the use of a binder. W. SWIENTOSLAWSKI, B. ROGA, and M. CHORAZY (Fuel, 1930, 9, 421—439; cf. B., 1929, 230, 1037).—The briquettes were made in a laboratory press under controlled conditions, and their mechanical strength and combustibility were determined. Neither anthracite nor semi-bituminous coals of low volatile content could be briquetted without a binder, even under high pressures (3000 kg./cm.²). Briquettes of satisfactory mechanical strength could be obtained from coking coals either (a) by heating to the temperature of incipient plasticity, (b) by prolonged heating at temperatures at which plasticity is fully developed (390—440°), or (c) by a shorter heating at a temperature corresponding to the last stage of the plastic state (450°); the necessary pressures are of the same order as those used industrially, *e.g.*, 200—300 kg./cm.². Agglutinating gas

coals gave briquettes under conditions similar to those used for coking coals. Non-agglutinating gas coals could also be briquetted under suitable conditions; the optimum temperature lay between very narrow limits, viz., 400–420°, and the optimum duration of heating was relatively short; the minimum pressure necessary was 400 kg./cm.² The briquettes possessed considerable mechanical strength, and behaved well on combustion. They were normally resistant to atmospheric conditions, and were not more hygroscopic than other solid fuels.

A. B. MANNING.

Simultaneous production of light oils, smokeless briquettes, and coke. L. VERTU (*Chim. et Ind.*, 1930, 24, 27–30).—Pulverised anthracite or coke is mixed with heavy petroleum, briquetted by pressure, and distilled. The gas evolved is used to supply part of the heat for the distillation. In the upper part of the retort, where the temperature is about 400°, smokeless briquettes are formed, and in the lower part, at about 650°, very hard coke. The oil which distils over is lighter and more volatile than that obtained by distillation of the heavy petroleum. By fractionating the distillate about 7% distils over up to 150°, and the residue may be used as a binder for the pulverised solid and re-treated, whereby a further yield of oil (b.p. below 150°) is obtained and the residue may be used as before.

D. K. MOORE.

Mechanism of charcoal activation. M. E. BARKER (*Ind. Eng. Chem.*, 1930, 22, 926–931).—Primary charcoal, prepared from wood or other carbonaceous materials, has a true density of about 1.45, contains considerable hydrogen and oxygen, which probably act as binders for the carbon grains, and shows no characteristic X-ray diffraction pattern. When such charcoal is activated, e.g., by steam at 800°, an internal shrinkage forms numerous minute voids and capillaries with the production of a large internal and active surface. The absorptive capacity of commercial wood charcoal may thus be increased from 11 to 1480. As a result of such activation, the charcoal granules become of graphitic character having a crystalline surface and a limiting d of 2.15. Part of the occluded gases, but not all, is expelled, and the diffraction rings attributed to graphite are observed.

C. A. KING.

Wood as a source of fuel for internal combustion engines. G. DUPONT (*Chim. et Ind.*, 1930, 24, 3–19).—The production of gas in gas producers on the vehicle is advocated. The fuel used in the gas producer must be low in ash and very reactive, give rise to no corrosive gas, and the gas must carry with it practically no dust or tar. Wood and wood charcoal are found to meet these requirements, but high-temperature charcoal is too dirty and bulky and wood evolves vapours when the engine is stopped. When pine wood is distilled there is little decomposition below 275°, and at about 300° exothermic decomposition takes place. As the temperature rises the percentage of carbon monoxide evolved increases until 320° is reached, after which it decreases and hydrogen and hydrocarbons are produced. Acetic acid is formed chiefly at 280–310° and tar at 270–380°. Other woods, cellulose, and lignin behave in a similar way, except that lignin and the more ligneous woods undergo the exothermal decomposi-

tion at a somewhat higher temperature. The brown charcoal produced at the temperature at which exothermic decomposition begins (275–290°) is dense, convenient for manipulation, and gives theoretically the best thermal yield (calc. on dry wood); when gasified in the producer it gives a gas with the greatest power per m.³ of engine cylinder, and practically no vapours are evolved in the producer when the engine is stopped. These theoretical figures have been experimentally confirmed. If the wood is distilled in a bath of organic liquid, e.g., pitch, 5–10% of the pitch is absorbed by the charcoal, and this by cracking in the producer should increase the cal. value of the gas; in practice, however, such increase is negated owing to imperfect cracking, smoke formation, and channelling. Impregnation of the wood with acids, e.g., phosphoric acid, before distillation is found to facilitate the evolution of water from the cellulose, whilst the lignin is not affected, and both the yield and cal. value of the charcoal are increased. Impregnation with alkalis, e.g., soda, retards the decomposition of the cellulose.

D. K. MOORE.

Separation of the constituents of coal gas by condensation in stages. A. THAU (*Gas- u. Wasserfach*, 1930, 73, 717–721; cf. *Bronn. B.*, 1929, 766).—The plant at Mont Cenis, comprising four units, each capable of dealing with 5300 m.³ of coke-oven gas per hr., is described (cf. *Engineering*, 1930, 129, 163). The separated mixture of hydrogen and nitrogen is used for the synthesis of ammonia. The plant at Duisberg, which supplies hydrogen for the recently-erected Bergius hydrogenation plant, has a throughput of 7000 m.³ of coke-oven gas per hr., and produces therefrom 3500 m.³ of hydrogen of 95% purity. The utilisation of the residual gas is discussed. The process cannot be used economically for rendering town's gas non-poisonous by the removal of carbon monoxide.

A. B. MANNING.

Utilisation of town's gas as a fuel in heat-treatment furnaces. C. M. WALTER (*J. Inst. Fuel*, 1930, 3, 408–419).—The first regenerative, gas-fired oven furnace heated the secondary air from the working chamber, and was capable of producing temperatures up to 1250°. Better recuperation was provided in later types, but even so thermal efficiencies rarely exceeded 25%, owing to very high radiation losses. The much higher figures now obtained are due to advances in insulation. A detailed account is given of trials with a set of modern recuperator furnaces, burning up to 700 cub. ft. of gas per hr., employed for carburising gear parts at 930°. Control was either by hand or arranged automatically by means of a thermocouple working on the gas supply, together with automatic damper control operated by the same motor. The efficiency is estimated at over 50% during the heating-up period, and the gas consumption is less than half that of furnaces of older type.

C. IRWIN.

Determination of the softening points of asphalts and pitches by the Kraemer-Sarnow method. H. BURSTIN (*Petroleum*, 1930, 26, 789–791).—The softening points of 8 asphalts and 2 pitches have been determined by the Kraemer-Sarnow method and by modifications thereof in which the mercury is replaced by a small metal rod (cf. *Spilker, B.*, 1929, 383; *Holde, B.*, 1929,

584). In general, the modified methods gave lower results than the original, the differences varying from $+1.7^{\circ}$ to -3.7° . Better agreement was obtained when a brass rod, 4 mm. in diam. and weighing 5 g., was used than with the type-metal rod suggested by Spilker (*loc. cit.*). A. B. MANNING.

Cracking of petroleum oils and low-temperature coal tars. W. H. JONES (J. Inst. Fuel, 1930, 3, 376—392).—Vapour-phase processes have the disadvantages of high fuel consumption, large production of gases and carbon, heavy up-keep and operating costs, but they produce a petrol of high antiknock value. Liquid-phase processes working at 100–750 lb./in.² and 450–500° include practically all the commercial units working to-day. For each oil there is a narrow temperature range which must not be exceeded. The Gyro process, the Cross process, and the Dubbs process representative of three classes are described in detail. Cracking processes may be worked to produce either fuel oil or coke, in the latter case with a higher yield of petrol. Analyses of charging stock and products are given. Results of cracking on a laboratory scale of various American low-temperature tars are given, together with cracking results from neutral oils washed for acid, German lignite tar, and distillates therefrom. Petrol prepared by these methods requires treatment with caustic alkali and sulphuric acid to render it water-white and stable to light. C. IRWIN.

Conversion of low-temperature tar into light oil. G. KROUPA (Petroleum, 1930, 26, 791–794).—The Semo process is described (cf. Stephan, B., 1930, 173).

A. B. MANNING.

Extinction of ethylene dichloride flames with carbon dioxide. G. W. JONES and R. E. KENNEDY (Ind. Eng. Chem., 1930, 22, 963–964).—The limits of inflammability of ethylene dichloride-air mixtures at 100° were 5.8% and 15.9%. The lower limit at 22° was 6.2%. Ethylene dichloride vapour at 100° is rendered non-explosive by mixing it with at least 2.3 times its own volume of carbon dioxide, and it is recommended that at atmospheric temperature and pressure at least 2.5 vols. of carbon dioxide should be added per vol. of ethylene dichloride. C. A. KING.

Smoke prevention. GIBBS. **Sieving analyses [of coal dust].** HEYWOOD.—See I. **Infusorial earth and floridin.** MARKMAN and KOVALENKO.—See VII. **"Flotation sulphur" for control of fruit diseases.** SMITH.—See XVI.

PATENTS.

Artificial fuel. J. A. WYLER, Assr. to TROJAN POWDER CO. (U.S.P. 1,752,935, 1.4.30. Appl., 9.1.28).—An infusible artificial fuel consists of a solidified mixture of nitrostarch, methyl and ethyl alcohols, and water. The addition of small quantities of formaldehyde, furfuraldehyde, etc. assists the formation of the gel.

A. B. MANNING.

Preventing or diminishing the formation of smoke in the burning of coal in open fire-places. J. WARR (B.P. 331,943, 19.4.29).—The fire grate is fitted with an enclosed retort, which may take the form of a metal box built behind the fire, and in which the coal is predistilled before being burned in the grate.

The volatile products of distillation are conveyed to a burner which heats the retort from below.

A. B. MANNING.

Production of finely-divided carbon or carbon black. G. C. LEWIS (B.P. 331,821, 4.4.29).—A gaseous mixture containing hydrocarbons is heated at a temperature, e.g., 600–850°, at which reactions occur involving the formation of aromatic hydrocarbons without the production of free carbon, and the products, after being cooled to a temperature, e.g., 250°, above that at which the vapours present would begin to condense, is fed to a burner for the production of free carbon by incomplete combustion.

A. B. MANNING.

Production of activated carbon. O. FUCHS, Assr. to HOLZVERKOHLUNGS-IND. A.-G. (U.S.P. 1,753,507, 8.4.30. Appl., 9.2.29. Ger., 13.1.28).—Carbonised material is reduced to particles of approximately uniform shape, size, and sp. gr., and is then treated with activating gases for a comparatively short period of time at about 900°.

A. B. MANNING.

Activation of charcoal. G. ENSSLE, Assr. to HOLZVERKOHLUNGS-IND. A.-G. (U.S.P. 1,753,984, 8.4.30. Appl., 6.2.29. Ger., 13.1.28).—The charcoal is treated at a suitable temperature with a mixture of gases, free from oxygen, containing at least 40% of carbon dioxide and a substantial proportion of a combustible gas. The uncondensable gases resulting from the carbonisation of wood form a suitable activating agent.

A. B. MANNING.

Activation of carbonaceous materials suspended in gases. SOC. DE RECHERCHES ET D'EXPLOIT. PÉTROLIFÈRES (B.P. 317,047, 23.7.29. Fr., 9.8.28).—The carbonaceous material, suspended in the activating gas, is passed through tubular retorts so designed that the products issuing therefrom are cooled by and simultaneously preheat the entering gas and material.

A. B. MANNING.

Apparatus for high- or low-temperature distillation of coal or lignite. H. HARDY (B.P. 317,374, 14.8.29. Belg., 14.8.28. Cf. B.P. 318,520; following abstract).—A number of oven units are arranged in a group, preferably in ring formation. Each unit consists of superimposed heating, distillation, and suction chambers. Between the units are combustion chambers having shutters by which they may be placed in communication with the heating chambers of one or both of the adjacent units; also conduits are provided whereby the hot gases from one oven unit may be conducted to another while permitting one or more intermediate units to be by-passed. By these means the temperature of each oven unit can be independently regulated. The suction chambers of each unit communicate with means for collecting and separating the tar and other by-products. The coal is carried in a removable mould having a perforated cover held in place by locking means in order to prevent swelling of the coal during distillation.

A. B. MANNING.

Low-temperature distillation of coal or lignite with production of semi-coke and valuable by-products. H. HARDY (B.P. 318,520, 3.9.29. Belg., 4.9.28).—The coal is contained in moulds which are conveyed through the distillation chamber by an

arrangement of rack bars and pinions, or similar device. The distillation chambers alternate with suction chambers and combustion chambers in such a manner that the moulds are heated only from one side, the volatile distillation products passing from the other side into the suction chambers and thence to the condensers. The heating of the distillation chambers is so arranged that the moulds pass successively through zones of gradually rising temperature until distillation is complete, and then through a cooling zone. The heat evolved in the latter is utilised for preheating the air for combustion. The moulds are charged and discharged in a separate plant. (Cf. preceding abstract.)

A. B. MANNING.

Drying, low-temperature carbonisation, distillation, or combustion of granulated or pulverulent fuels. C. GEISSEN (B.P. 310,032, 12.4.29. Ger., 20.4.28).—A layer of the material is supported on a horizontal grate and the active gases or vapours are supplied through openings in the grate which are provided with mushroom- or valve-shaped distributing devices. The shafts of the latter almost fill the openings in the grate, leaving only narrow annular passages which offer a high resistance to the flow of the gases or vapours.

A. B. MANNING.

Working of chamber ovens for production of gas and coke. C. OTTO & Co., G.M.B.H. (B.P. 318,935, 12.9.29. Ger., 12.9.28).—The ovens are arranged in groups and adjustable closing devices for each group are provided in the common gas and air supplies in such a manner that each group can be worked at a different carbonising period. By this means it is possible to vary the gas output over a considerable range without putting any of the groups of ovens out of operation.

A. B. MANNING.

Manufacture of liquid products from coal or carbonaceous materials by heating under pressure in the presence of hydrogen, or gases or vapours containing hydrogen. N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 314,859, 31.5.29. Holl., 3.7.28).—“Luxmass,” the alkaline material containing iron oxide obtained as a by-product from bauxite in the preparation of aluminium oxide, is used as a catalyst in the hydrogenation process. It is found to be much improved for this purpose if it is first repeatedly extracted with hot water, and then dried at 120°.

T. A. SMITH.

Destructive hydrogenation of coal, oils, etc. C. F. R. HARRISON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 331,817, 2.4.29).—The vapours originating from the destructive hydrogenation of carbonaceous materials in the liquid phase are superheated and are then passed, together with hydrogen, over a hydrogenating catalyst at a temperature below that prevailing in the superheating zone. Cracking catalysts, e.g., iron coated with zinc, but not hydrogenating catalysts, may be used in the superheating zone.

A. B. MANNING.

Recovery of the products of destructive hydrogenation and cracking processes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 331,916, 11.4.29).—The processes of condensing the vapours and scrubbing the gases leaving the reaction vessel are carried out

simultaneously by introducing the washing oil into the gaseous and vaporous products before they enter the condensers.

A. B. MANNING.

Gas producers. SOUTH METROPOLITAN GAS CO., and C. C. CARPENTER (B.P. 332,921, 30.4.29. Cf. B.P. 305,764; B., 1929, 313).—Loss of fuel during clinking is diminished by substituting for the natural draught a suction so controlled that the draught is kept above the minimum necessary for avoiding dangerous escape of hot gas through the clinking doors. The substituted draught is preferably equal to or only slightly less than the normal draught during the non-clinking period. It is also preferable to apply the suction at a level above the normal off-take of the producer in such a manner that the gases are drawn through a bed of freshly charged coke; with such an arrangement a diminished suction may be applied also during the non-clinking period.

A. B. MANNING.

Removal of sulphur compounds from gases. W. J. HUFF, L. LOGAN, and O. W. LUSBY (B.P. 305,026, 25.1.29. U.S., 28.1.28).—The gases are passed over a purifying material comprising at least one metal of group VI or VII in intimate admixture with a metal of another group, e.g., copper (80%) and chromium (20%), or copper (80%), uranium (10%), and chromium (10%). The treatment is carried out above 250°, and removes hydrogen sulphide and organic sulphur. The purifying material is revived by the action of an oxidising gas.

A. B. MANNING.

Ammonia-recovery process [from coke-oven gas]. J. BECKER, Assr. to KOPPERS CO. (U.S.P. 1,747,616, 18.2.30. Appl., 2.8.22).—The gas is scrubbed with hot wash-liquor, consisting of aqueous condensate from the coke ovens, to remove the fixed ammonium compounds, and is then passed up a cooler in counter-current to cold wash-liquor, which takes up part of the free ammonia. The hot wash-liquor is conducted to the lower part of an ammonia still, where it is mixed with milk of lime and distilled; the cold liquor undergoes separate distillation in the upper part. The combined vapours together with the gas from the cooler pass to a saturator, where the ammonia is recovered. Means are provided for separating the tar. The process considerably reduces the amount of wash-liquor to be evaporated and more effectively utilises the remainder.

W. J. WRIGHT.

Manufacture of acetylene from hydrocarbons or mixtures containing them in the electric arc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 332,917, 24.4.29).—Acetylene is produced in 4–8% yield in one operation, without appreciable deposition of carbon, when methane is passed through an electric arc of at least 50 kw., the ratio of cub. m. of gas per hr. to the kw. power of the arc being between 0.6 and 1.6. A direct or an alternating current of 500–2000 periods is employed.

C. HOLLINS.

Treatment of hydrocarbons. N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 334,032, 30.7.29. Holl., 23.10.28).—Blown asphalt is prepared in an apparatus consisting of a separating vessel and a heating coil through which the asphalt may be circulated. Air is blown through a meter into the bottom of the separating

vessel and volatile products are removed. Air connexions are also made to the heating coil so that this may be blown through if necessary. The vapours from the separator are condensed, the liquid being used as fuel. The permanent gases are also used as fuel.

T. A. SMITH.

Manufacture of liquid fuels. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,550, 11.3.29).—Coal, tars, or mineral oils, or their products by distillation, destructive hydrogenation, or cracking, and preferably the middle oil fraction of the same, are subjected to the action of solvents which separate the initial materials into portions richer and poorer in hydrogen; the latter, after removal of the solvent, is subjected to destructive hydrogenation under pressure either alone, or with other carbonaceous substances, or even with a portion of the fraction rich in hydrogen, thereby producing a motor fuel of low b.p. Suitable solvents are liquid sulphur dioxide or ammonia, used, if desired, with cyclohexane or cyclohexanol and methyl alcohol.

H. S. GARLICK.

Natural-gas liquefaction. H. DUMARS, Assr. to BOWEN-DUMARS POWER CORP., and H. F. CUNTZ (U.S.P. 1,747,761, 18.2.30. Appl., 8.9.22).—Natural gas, at well temperature and pressure, is admitted to an expansion engine where the temperature and pressure are reduced, but not to such an extent that liquefaction takes place. The cooled gas is passed into heat-exchange relation with another portion of the gas, at well temperature and pressure, which after precooling is admitted to a second expansion engine where liquefaction of at least a portion of the gas takes place.

H. S. GARLICK.

Absorption process [for natural gas]. P. D. BARTON, Assr. to UNITED OIL CO. (U.S.P. 1,748,051, 25.2.30. Appl., 4.5.26).—Natural gas is passed into contact with a colloid solution composed of water and a colloid capable of absorbing gasoline, whereby the gasoline is absorbed and subsequently separated by stratification. The process is applicable to any mixed fluids and to absorbing medium capable of forming an emulsion selectively with one of them.

H. S. GARLICK.

Oil-cracking process. A. SCHWARZ, Assr. to PETROLEUM AND PRODUCTS CORP. (U.S.P. 1,756,887, 29.4.30. Appl., 25.2.24).—Petroleum oil is distilled at cracking temperature and under superatmospheric pressure in a cracking zone and the vapours are passed under successively decreasing pressures into successive masses of subdivided solid particles (*e.g.*, iron wool) wherein a portion of the vapours are condensed and held in minutely subdivided form over an extended area, while the rest makes intimate contact with the condensate. Hot residue from the cracking zone is passed countercurrent to the flow of vapours, and fresh oil may be introduced into the last of the series of the condensing system.

H. S. GARLICK.

Cracking of hydrocarbons. E. C. HERTHEL, Assr. to SINCLAIR REFINING CO. (U.S.P. 1,755,600—1, 22.4.30. Appl., [A] 30.6.25, [B] 27.7.27).—(A) In conjunction with an oil-cracking pressure still is a heat-insulated reflux tower of baffle construction into which fresh oil is introduced one or two baffles below the top of the tower. Initially the oil flows downwardly in one or more com-

pact streams which are not broken up and sprayed or distributed through the vapours until the oil is part way down the tower, thus avoiding difficulties due to entrainment of constituents of fresh oil in the vapour escaping from the vapour end of the tower. (B) Vapours from a pressure distillation are passed into a reflux condenser into which a light oil, which is substantially vaporised completely, under the prevailing conditions, is introduced, in direct contact with the vapours at a series of points so distributed that the proportion supplied at the part nearest the vapour inlet to the reflux condenser is less than the proportion supplied at a further point in the path of vapour travel.

H. S. GARLICK.

Cracking of [hydrocarbon] oils. C. P. DUBBS (B.P. 332,913, 27.3.29).—The oil is heated in a cracking zone to cracking temperature and passed directly into an adjacent reaction or separating zone under lower pressure where cracking is completed and the treated oil separated into vapours, which are dephlegmated, and an unvaporised liquid residue, which is removed without coming into contact with either reflux condensate or with fresh oil. To obtain a liquid residue suitable for fuel oil, the polymerisation of the unvaporised residue is checked (a) by controlling the reduction of pressure in the reaction zone with consequential temperature reduction therein; (b) by introducing as a cooling medium, into direct contact with the oil, subsequent to the heating but prior to its expansion, an oil of lower temperature than that of the oil being cracked; (c) by regulating the speed of withdrawal of the residue and so maintaining a controlled liquid level; and (d) by dissipating the remaining heat from the residue immediately after its withdrawal in such a way that the amount of sludge or coke formed in the residual oil is normally less than 6%.

H. S. GARLICK.

Cracking of hydrocarbons. G. EGLOFF and J. C. MORRELL, Assrs. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,748,065, 25.2.30. Appl., 28.6.26).—Oil is subjected to cracking temperature and pressure and the cracked vapours are removed and collected. The residue from the expansion chamber, containing carbonaceous and pitchy material, is passed continuously through a colloid mill and passed again either through the same or another cracking coil.

H. S. GARLICK.

Manufacture of hydrocarbons which are saturated with hydrogen. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,511, 2.4. and 14.10.29).—Crude hydrocarbon mixtures are treated with hydrogen, or gases containing or supplying hydrogen, under ordinary or increased pressure and at temperatures above 200° (preferably 300–360°), in the presence of catalysts preferably deposited on or applied to carriers such as Florida or fuller's earth, silica gel, etc. The catalysts consist of solid oxides or sulphides of the metals of groups II–VII or compounds of these metals capable of supplying such oxides or sulphides, preferably those of group VI or mixtures thereof, again preferably in conjunction with metals of group VIII or with heavy metals of group I, *e.g.*, silver, gold, or suitable compounds thereof. Examples are oxides or sulphides of molybdenum or tungsten and nickel or cobalt. The process is

continued until not only any sulphur or oxygen compounds present in the materials under treatment, but also nitrogen and halogen compounds as well as resinifying agents, are rendered non-injurious, without the occurrence of any appreciable splitting up of the hydrocarbons themselves. When treating polynuclear compounds the conditions should be such that in addition to purification of the hydrocarbons a hydrogenation of some, but not all, of the double linkings in the molecule takes place. The purified hydrocarbons thus obtained are hydrogenated in the presence of hydrogenating catalysts activated with metals of groups II—VII, or compounds thereof, not already contained in the catalyst of the first stage. H. S. GARLICK.

Pyrolysis of unsaturated hydrocarbons. IMPERIAL CHEM. INDUSTRIES, LTD., T. S. WHEELER, and J. MCAULAY (B.P. 332,998, 4.6.29).—Olefines are passed at high space velocity (*e.g.*, 100 or 6400 vols. per min. through unit vol. of tube) through a tube heated above 1000° (*e.g.*, at 1050° or 1100°) for conversion into light oil. C. HOLLINS.

Converting high-boiling mineral oils containing unsaturated compounds into lower-boiling hydrocarbons with simultaneous decoloration. N.V. MIJNBOUW- EN CULTUURMAATS. "BOETON" (B.P. 333,553, 9.4.29. Holl., 16.1.29).—Crude oil containing unsaturated compounds is treated outside the still at temperatures above 60° (preferably at 70—100°) with concentrated aqueous solutions, liquid at ordinary temperatures, containing at least 58% of one or more solid hygroscopic chlorides of heavy metals, *e.g.*, ferric chloride solution of *d* 1.6; the product after removal of the salt solution is neutralised if necessary prior to distillation. H. S. GARLICK.

Desulphurisation of liquid hydrocarbons. STADTBERGER HÜTTE A.-G. (B.P. 332,910, 26.2.29).—The liquids are completely dehydrated and freed from a portion of their sulphur content by known methods and then introduced into the bottom of a tubular receptacle (fitted with heating coil and stirrer) charged with finely-divided copper, independent of any support, that has been prepared by precipitating metallic copper from its salt solutions by means of iron or other suitable base metal and subsequently heating it above 100° in a reducing atmosphere. A filtering device to retain any copper particles is disposed in front of the outlet. H. S. GARLICK.

Production of hydrocarbons of low b.p. from those of higher b.p. H. W. STRONG, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 331,816, 2.4.29).—The vapours of middle oils are passed with hydrogen, under a total pressure of at least 20 atm. and at 400—500°, first over a contact mass consisting of iron coated with zinc, and then over a porous contact mass consisting of ferric oxide. A. B. MANNING.

Apparatus for removing carbonaceous deposits from oil apparatus. L. C. HUFF, ASSR. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,756,027—8, 29.4.30. Appl., 22.7.25. Renewed 2.2.29).—(A) A single, continuous, flexible element, *e.g.*, chain or cable, is suspended in such a way from spaced points at the top of the enlarged reaction chamber of an oil-cracking apparatus that its

withdrawal causes disruption of the deposited carbon. (B) Separate flexible elements may be attached diagonally to the chain described in (A) and so arranged that the system may be withdrawn as a whole, or part may be withdrawn through the top and the rest through the bottom of the reaction chamber. H. S. GARLICK.

Treatment of acid sludge and fuel product obtained therefrom. R. A. HALLORAN, W. N. DAVIS, and G. A. DAVIDSON, ASSRS. to STANDARD OIL Co. OF CALIFORNIA (U.S.P. 1,745,692, 4.2.30. Appl., 6.9.23).—The acid sludge obtained in the treatment of petroleum oils, with or without the addition of fuel oil, is mixed with dirty dilute acid in a closed retort and heated to hydrolysing temperature under a pressure exceeding 50 lb. until separation into acid and a liquid fuel is obtained. H. S. GARLICK.

Manufacture of lubricating oils. Refining of hydrocarbon oils [for production of lubricating oils]. E. C. HERTHEL and H. L. PELZER, ASSRS. to SINCLAIR REFINING Co. (U.S.P. 1,756,153 and 1,756,156, 29.4.30. Appl., 16.6.28).—The constituents of raw stock suitable as components of lubricating oil are vaporised therefrom and the vapours bubbled through a series of hot, alkaline, liquid oil baths, the vapours being introduced beneath the surface of each of the baths. The oil baths are maintained alkaline by supplying a caustic alkali to the treating bath through which the vapours last pass and overflowing the liquid from the bath to the preceding one and so on until discharged from the last one. Vapours of the desired lubricating oil are taken off from the last hot oil bath and condensed out of contact with the liquid flowing countercurrent to the vapours through the series of hot oil baths. H. S. GARLICK.

Lubricant. F. L. KOETHEN, ASSRS. to ACHESON GRAPHITE Co. (U.S.P. 1,758,446, 13.5.30. Appl., 10.11.26).—A water-free lubricating compound comprises a fine suspension of an iron soap in a mineral oil. H. S. GARLICK.

[Lubricant for] treatment of drill-stem joints. DE R. FRIZELL, ASSR. to RICHFIELD OIL Co. OF CALIFORNIA (U.S.P. 1,758,598, 13.5.30. Appl., 7.6.27).—A water-insoluble grease consists of a calcium soap and a mineral lubricating oil containing suspended, comminuted, spherical, metallic particles, *e.g.*, 60% of lime soap cup-grease and 40% of air-floated zinc dust. H. S. GARLICK.

Resolution of oil emulsions and reagents therefor. F. H. PENN (U.S.P. 1,758,802, 13.5.30. Appl., 28.12.25. Renewed 20.4.28).—The emulsion is treated with 0.02—0.1% of a reagent consisting of an acid derivative of a mineral oil, *e.g.*, a sulphonated paraffin oil (2 pts.), and a fatty acid glyceride, *e.g.*, cotton-seed, olive, or castor oils (1 pt.), with or without a trace of phenol. H. S. GARLICK.

Motor fuel. W. GAUS, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,775,674, 16.9.30. Appl., 7.5.26. Ger., 8.5.25).—See B.P. 251,969; B., 1927, 645.

Refining of low-boiling hydrocarbons. J. METZGER, A. KREUTZER, and T. HELLTHALER, ASSRS. to A. RIEBECK'SCHE MONTANWERKE A.-G. (U.S.P. 1,776,039,

16.9.30. Appl., 30.4.26. Ger., 10.6.25).—See G.P. 439,608; B., 1927, 695.

Production of solid lubricants. W. PUNGS and E. FRESE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,776,193, 16.9.30. Appl., 25.4.29. Ger., 9.5.28).—See B.P. 317,630; B., 1929, 862.

[Air-blast] extraction of dust from minerals such as coal. S. HUNTER (B.P. 334,792, 21.11.29).

Pulverised-fuel burners. BABCOCK & WILCOX, LTD. From FULLER LEHIGH CO. (B.P. 334,755, 14.10.29).

Retort furnace (U.S.P. 1,757,644). Heating furnace for tar etc. (U.S.P. 1,757,707). Pressure distillation (U.S.P. 1,757,579). Viscosimeter (U.S.P. 1,758,677).—See I. **Polymerisation process (U.S.P. 1,746,168). Polymerised diolefines (B.P. 333,872 and 333,894). Organic compounds by dehydrogenation (B.P. 333,975). Oxidation of organic products (B.P. 309,498).**—See III. **Ammonia oxidation (U.S.P. 1,748,646).**—See VII. **Waterproof concrete (U.S.P. 1,744,869). Asphalt emulsions (B.P. 332,591). Pavement materials (B.P. 333,937). Paving blocks (B.P. 333,047). Road-making materials (B.P. 332,897). Wood-impregnating product (B.P. 310,804—5).**—See IX. **Rubber compounding (U.S.P. 1,743,433).**—See XIV.

III.—ORGANIC INTERMEDIATES.

Flash points of mixed solvents. F. D. SNELL (Ind. Eng. Chem., 1930, 22, 893—894).—Mixtures of carbon tetrachloride with acetone and with toluene were prepared, representing commercially occurring cases of mixtures of non-inflammable solvents with inflammable solvents of higher and lower volatility, respectively. The flash points of the mixtures as prepared and after keeping until 50% evaporation had occurred were determined, the acetone mixtures showing a rise and the toluene mixtures a fall in flash point. The significance of these results with reference to "flame-proofed" mixtures sold as fabric cleaners, larvicides, etc. is discussed. S. S. WOOLF.

PATENTS.

Manufacture of chlorine and bromine derivatives of compounds of the acetylene series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 333,946, 25.5.29).—Chlorine or bromine is introduced into an acetylenic compound by direct substitution by action of an aqueous hypochlorite or hypobromite, preferably in presence of excess of free alkali. The preparation of dichloroacetylene, m.p. -64° to -68° , b.p. 32° ; dibromoacetylene; dibromodiacetylene, m.p. $52-53^{\circ}$ (decomp.); bromopropiolic acid, m.p. $84-86^{\circ}$; chloropropiolic acid, m.p. $69-70^{\circ}$; 1-bromomethinylcyclohexan-1-ol, m.p. $55.5-56^{\circ}$, b.p. $111-112^{\circ}/11-12$ mm.; and bromomethinyl-diethylcarbinol, m.p. $16-18^{\circ}$, b.p. $84-85^{\circ}/13-14$ mm., is described. C. HOLLINS.

Photochemical process [of polymerisation]. H. S. TAYLOR, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,746,168, 4.2.30. Appl., 28.5.28).—Polymerisable compounds, e.g., olefines, vinyl compounds, resinous or rubber-forming compounds, etc., are prefer-

ably mixed or maintained in contact with a metallic vapour in the presence of the liquid or solid phase of the metal (e.g., mercury) and subjected to the resonance radiation of that metal. H. S. GARLICK.

Manufacture of polymerisation products of diolefines. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,894, 16.5.29).—Rubber substitutes are obtained by polymerising butadienes at 70° in presence of hydrazine derivatives, e.g., $\alpha\alpha'$ -hydrazoisobutyric acid, ethyl hydrazinedicarboxylate, phthalhydrazide, *s*-benzoylacethydrazide, benzhydrazide, or hydrazine hydrazinecarboxylate. C. HOLLINS.

Manufacture of soluble polymerisation products from diolefines. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,872, 15.3.29).—Diolefines (butadiene, isoprene) are treated at $60-100^{\circ}$ with alkali metals dispersed in an organic solvent (decahydronaphthalene, xylene). The products are viscous or tenacious masses, only slightly elastic, and soluble in all proportions in hydrocarbon or ester solvents. C. HOLLINS.

Concentration of aqueous acetic or formic acid. IMPERIAL CHEM. INDUSTRIES, LTD., and F. D. LEICESTER (B.P. 332,983, 17.5.29).—Acetic acid is selectively adsorbed when the vapour of the dilute acid is passed over active carbon at $120-130^{\circ}$, and may be recovered in concentrated form (96%) by heating the carbon to 200° . For formic acid the adsorption occurs at $100-110^{\circ}$. Reduction of pressure assists the recovery of either acid. The selective adsorption may also be accomplished with dilute liquid acids without heat. C. HOLLINS.

Manufacture of glacial acetic acid from dilute acetic acid. I. G. FARBENIND. A.-G. (B.P. 312,173, 21.5.29. Ger., 18.5.28).—Dilute acetic acid is cooled, with vigorous agitation, to -26° (eutectic point of acetic acid–water), the ice crystals are removed, and the concentrated acid (60%) is treated with a substance which forms a double compound from which acetic acid may be regenerated, e.g., carbamide $[\text{CO}(\text{NH}_2)_2 \cdot 2\text{AcOH}]$, eutectic point -17° or potassium acetate $(\text{AcOK} \cdot 2\text{AcOH})$, eutectic point -24° . The double compound is frozen out and glacial acetic acid is distilled from the separated product in a vacuum. Preferably the amount of substance added should be less than sufficient for combination with all the acetic acid present, and the mother-liquors are returned to the process. C. HOLLINS.

Carrying out ester condensations. [Synthesis of acetoacetic esters.] DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 311,707, 8.5.29. Ger., 14.5.28).—A 94% yield of ethyl acetoacetate is obtained from ethyl acetate and sodium ethoxide by distilling off as completely as possible during the reaction the alcohol formed. The process is applicable to esters of methyl, ethyl, and propyl alcohols. C. HOLLINS.

Manufacture of stable polymerisation products from vinyl esters. I. G. FARBENIND. A.-G. (B.P. 319,682, 29.5.29. Ger., 26.9.28).—Unchanged vinyl ester, acetaldehyde, acetic acid, etc. are removed and a stable polymeride is obtained by prolonged (several

hours') contact of the polymeride, in the form of raspings, clippings, spun threads, or thin sheets, with water.

C. HOLLINS.

Manufacture of acrylyl chloride. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,079, 14.9.29).— β -Chloropropionyl chloride gives acrylyl chloride in 80–85% yield when the vapour is led over a suitable catalyst (barium chloride, broken clay, alumina) heated at or above the b.p. of the starting material, *e.g.*, 280–300°. The chloropropionyl chloride may, if desired, be distilled through a column charged with the contact material.

C. HOLLINS.

Manufacture of trisubstituted thioureas [thiocarbamides]. I. G. FARBENIND. A.-G. (B.P. 314,542, 27.6.29. Ger., 30.6.28).—Alkaline carbamates derived from primary amines are heated in water or alcohol with a salt of a secondary amine. Dimethylamine methyl-dithiocarbamate yields trimethylthiocarbamide, and from dimethylamine phenyldithiocarbamate *N*-phenyl-*N'*-dimethylthiocarbamide is prepared.

C. HOLLINS.

Manufacture of organic compounds by dehydrogenation. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,975, 5.6.29).—For the dehydrogenation of hydrogenated aromatic hydrocarbons, petroleum fractions, etc., a mixed catalyst is used comprising cobalt, iron, or metals of groups V, VI, and VII, or compounds of these, in admixture with one another or with other substances, the mixed catalyst being pretreated above 200° with hydrides or other compounds of solid non-metals of groups V, VI, and VII. The added substances are preferably lead, tin, zinc, cadmium, or their compounds, or solid oxides of group V non-metals or the corresponding acids or salts. *cyclo*Hexane is converted largely into benzene when the vapour is led at 480° over active charcoal which has been impregnated with ammonium molybdate, lead nitrate, and phosphoric acid and pretreated with hydrogen selenide at 300°. Other catalysts mentioned are: ammonium tungstate, cobalt nitrate, and antimonie acid on active charcoal, treated with hydrogen sulphide at 350°; ammonium vanadate, cobalt, nitrate, and phosphoric acid on active charcoal, treated with hydrogen and carbon disulphide at 350°; uranium oxide and cobalt oxide, treated with hydrogen and carbon disulphide at 350°. Petroleum fractions may be enriched in aromatics, and pentane converted into pentene by the use of such catalysts.

C. HOLLINS.

Manufacture of organic products. [Oxidation by air or oxygen at high pressures.] D. FUTACCHI (B.P. 309,498, 10.4.29. Fr., 11.4.28).—The oxidation of methyl alcohol to glycol, of ethyl and methyl alcohols to propylene glycol, of petroleum to alcohols, aldehydes, and ketones, of paraffins to fatty acids and amino-fatty acids (in presence of ammonia), etc., is carried out by treating the starting material in liquid form or in solution in an inert liquid with air or oxygen at pressures of 150–800 kg./cm.² below 500° in presence or absence of catalysts. *E.g.*, air at 450 kg./cm.² is forced through two similar tubes in series, the second of which contains methyl alcohol heated under pressure to 180°; the first tube provides a safety chamber into which the

liquid flows when the air pressure is released in the event of overheating. The product consists of glycol 40%, methyl alcohol 5%, glycerol 20%, and a viscous mixture of carbohydrates.

C. HOLLINS.

Oxidation of benzene hydrocarbons. I. G. FARBENIND. A.-G. (B.P. 318,550, 3.9.29. Ger., 5.9.28).—The process of B.P. 309,005 (B., 1929, 917) is extended to tetrahydronaphthalenes. The production of α -keto-tetrahydronaphthalene, b.p. 130–135°/15 mm. (with 20% of the α -hydroxy-compound), of α -ketoethyltetrahydronaphthalene (semicarbazone, m.p. 223°) from *ar*-ethyltetrahydronaphthalene, and of *ar*-chloro- α -keto-tetrahydronaphthalene, b.p. 160°/20 mm. (semicarbazone, m.p. 219°), is described.

C. HOLLINS.

Manufacture of polymerisation products of styrene. I. G. FARBENIND. A.-G. (B.P. 307,936, 15.3.29. Ger., 16.3.28).—The polymerisation of styrene is effected in aqueous emulsion in presence of an emulsifying agent or colloidal solution at temperatures not substantially above 60° and without superatmospheric pressure. Styrene is stirred, *e.g.*, with 1.3 pts. of milk for 10 days at 60° to give a resinous polymeride which is elastic at 100°.

C. HOLLINS.

Manufacture of aldehydes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 334,009, 4.7.29).—The yields of aldehydes by Gattermann's method from carbon monoxide and aromatic hydrocarbons under pressure are increased by adding titanium chloride to the aluminium chloride. The action of chlorine on a titaniferous bauxite and carbonaceous matter gives a suitable catalyst. The preparation of benzaldehyde from benzene at 30–35° and 60 atm., *p*-tolualdehyde from toluene at 35–40° and 60 atm., and *p*-chlorobenzaldehyde from chlorobenzene at 60° and 50 atm., is described.

C. HOLLINS.

Manufacture of arylaminophenolcarboxylic acids. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 333,783, 28.9.29).—The alkali-metal salt of a *m*-hydroxydiphenylamine is heated with carbon dioxide under pressure, *e.g.*, at 150–170° and 5–10 atm. for 16 hrs. The following 4-arylaminoalicyclic acids are described: anilino-, m.p. 180–181°; *o*-toluidino-, m.p. 180–182°; *m*-toluidino-, m.p. 176–177°; *p*-toluidino-, m.p. 185–186°; *o*-anisidino-, m.p. 199–200°; *p*-anisidino-, m.p. 176–177°; *o*-chloroanilino-, m.p. 198°; *m*-chloroanilino-, m.p. 189–190°; *p*-chloroanilino-, m.p. 188–190°; *m*-4-xyldino-, m.p. 175°; *p*-xyldino-, m.p. 175–176°; 6-methoxy-*m*-toluidino-, m.p. 165°; 5-chloro-*o*-toluidino-, m.p. 180–181°; 4-chloro-*o*-toluidino-, m.p. 208–210°; 6-chloro-*m*-toluidino-, m.p. 195–197°; 2-chloro-*p*-toluidino-, m.p. 190°; 2,4-dichloroanilino-, m.p. 215°; 3,4-dichloroanilino-, m.p. 199°; 4,5-dichloro-*o*-toluidino-, m.p. 203°; *p*-phenoxyanilino-, m.p. 189°.

C. HOLLINS.

Manufacture of 2-chlorobenzthiazoles. I. G. FARBENIND. A.-G. (B.P. 310,815, 30.4.29. Ger., 30.4.28).—A 2-thiol- or 2-hydroxy-benzthiazole is treated with phosphoryl chloride or thiophosphoryl chloride (PSCl₃) or with phosphorus pentachloride diluted with either of these. The preparation of 2-chlorobenzthiazole, b.p. 248°, and its 5-methyl (m.p. 40–47°, b.p. 266° or

132°/15 mm.) and 6-nitro- (m.p. 190°) derivatives is described. 6-Nitro-2-anilinothiazole has m.p. 247°.

C. HOLLINS.

Preparation of thymol. RHEINISCHE KAMPFER-FABR. G.M.B.H. (B.P. 312,907, 29.4.29. Ger., 1.6.28. Addn. to B.P. 293,753; B., 1928, 837).—In place of propylene in the process of the prior patent propyl or isopropyl alcohol or ester or other compound yielding propylene is employed, preferably in presence of catalysts effecting the conversion into propylene. *E.g.*, *m*-cresol is heated in an autoclave at 350° with isopropyl alcohol, water is distilled off, and the heating repeated with more alcohol; thymol equivalent to 60–70% of the unrecovered *m*-cresol is obtained. If propyl chloride is used as a source of propylene, zinc oxide or other acid absorbent is added.

C. HOLLINS.

Manufacture of C-alkylated phenols. SCHERING-KAHLBAUM A.-G. (B.P. 309,865, 15.4.29. Ger., 16.4.28. Addn. to B.P. 254,753 and 274,439; B., 1928, 740; 1929, 164).—The fission of dihydroxydiphenylmethanes by hydrogen in presence of catalysts is facilitated and nuclear hydrogenation avoided by diluting the hydrogen with 33% of inert gas, preferably steam. The steam is advantageously generated *in situ*, by using the catalyst in the form of oxide. *E.g.*, 4:4'-dihydroxydiphenylpropane is converted by hydrogen at 200° and 20–25 atm. pressure in presence of mixed carbonates or hydroxides of nickel and copper (with traces of cobalt and iron) into *p*-hydroxyisopropylbenzene and phenol.

C. HOLLINS.

Manufacture of condensation products from aldehydes and phenols [mothproofing compounds].

A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 333,561, 11.5.29).—Mothproofing compounds which are not dyes are obtained by condensing the hydrol (etc.) prepared from an aldehyde (non-hydroxylated, if aromatic) and 1 mol. of a *p*-halogenated phenol or naphthol having a free *o*-position and no salt-forming group, with a second mol. of the same or a similar phenol or naphthol, preferably in presence of sulphuric acid, hydrochloric and acetic acids, or zinc chloride. Examples are: benzaldehyde-*o*-sulphonic acid with 2 successive mols. of 2:4-dichlorophenol, or 2:3-dichlorophenol and *p*-chlorophenol, or 2:4:5-trichlorophenol and 2:4-dichlorophenol; formaldehyde with 2:4-dichlorophenol and *p*-chlorophenol.

C. HOLLINS.

Manufacture of sulphonated condensation products [from phenols and halogenated benzyl halides]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 332,960, 1.5.29. Addn. to B.P. 320,056; B., 1930, 315).—The process of the prior patent, especially when used with polyhalogenated aralkyl halides, is conducted in presence of zinc chloride. The condensation of phenol with trichlorobenzyl chloride and of *p*-chlorophenol with *o*-chlorobenzyl chloride, followed by sulphonation of the products, is described.

C. HOLLINS.

Manufacture of condensation products from aliphatic aldehydes and bases of the naphthalene series [anti-agers]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 333,941, 24.5.29).—Odourless, finely-divided products, useful as anti-oxidants, are

obtained by condensing aliphatic aldehydes with amines of the naphthalene series in alcohol in presence of acid. *E.g.*, α -naphthylamine and commercial aldol in boiling ethyl alcohol containing a little formic acid yield on precipitation with water a fine powdery resin, m.p. 210°. The product from 1:5-naphthylenediamine and aldol has m.p. 200°; the 1:8-diamine gives a similar product, m.p. 250°.

C. HOLLINS.

Manufacture of derivatives of naphthalene [acyl-naphthalenes and 4-substituted α -naphthoic acids].

J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,666—7, 4.6.29).—(A) Naphthalene, or a 1-aralkyl-naphthalene having a free 4-position, is condensed with an acid chloride in presence of aluminium chloride etc., and the products are oxidised if desired. 1-Benzyl-naphthalene and benzoyl chloride yield phenyl 4-benzyl- α -naphthyl ketone, m.p. 110°, which is oxidised by nitric acid to 1:4-dibenzoylnaphthalene, m.p. 106°. 1-*p*-Chlorobenzyl-naphthalene, b.p. 176–177°/0.2 mm., is similarly converted into phenyl 4-*p*-chlorobenzyl- α -naphthyl ketone, m.p. 123°, and 1-benzoyl-4-*p*-chlorobenzoylnaphthalene, m.p. 118. 4-Benzyl- α -naphthyl methyl ketone, m.p. 78°, from benzyl-naphthalene and acetyl chloride, gives on oxidation 4-benzoyl- α -naphthoic acid, m.p. 184°. (B) 4-Alkyl- α -naphthyl ketones are oxidised, *e.g.*, with nitric acid or alkaline hypochlorite. Phenyl 4-methyl- α -naphthyl ketone gives 4-benzoyl- α -naphthoic acid, m.p. 184°; 4-methyl- α -naphthyl methyl ketone gives 4-methyl- α -naphthoic acid, m.p. 165°, or, with more oxidant, naphthalene-1:4-dicarboxylic acid.

C. HOLLINS.

Manufacture of hydroxy-1':8'-naphthoyle-naphthiminazoles and sulphonic acids thereof.

I. G. FARBENIND. A.-G. (B.P. 316,143, 22.7.29. Ger., 23.7.28).—The naphthiminazoles obtained by condensing 1:8-naphthalic anhydride with 1:2-naphthylenediaminesulphonic acids are fused with alkali. The compound from the diamine-5-sulphonic acid gives 6-hydroxy-1':8'-naphthoyle-naphthiminazole (annexed formula), m.p. 323°. The 8-hydroxy-compound, m.p. 350°, and the 6:8- and 9:7-hydroxy-sulphonic acid are obtained from the iminazoles derived from 1:2-naphthylenediamine-7-sulphonic, -5:7- and -6:8-disulphonic acids, only

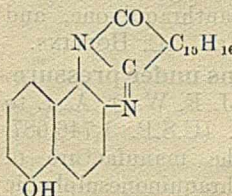
the α -sulphonic groups in the disulphonic acids being hydrolysed. The products have pharmaceutical application.

C. HOLLINS.

Manufacture of derivatives of the naphthoyle-naphthiminazole series [from naphthalenetetracarboxylic acid and *o*-diamines].

I. G. FARBENIND. A.-G. (B.P. 334,111, 22.10.29. Ger., 22.10.28).—The bisiminazoles obtained by condensing 1:4:5:8-naphthalenetetracarboxylic anhydride with *o*-diamines (*o*-phenylenediamine, 3:4-diaminophenetole, 1:2-naphthylenediamine) are nitrated and reduced (with sodium sulphide or in the vat). The iminazoles from the three diamines mentioned yield blue-grey to black, violet-brown, and olive-green vat dyes, respectively.

C. HOLLINS.



Manufacture of derivatives of quinoline. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 332,911, 25.3.29).—Arylamines are condensed with esters of malonic acids in a high-boiling solvent (nitrobenzene, excess of the malonic ester), the alcohol formed being distilled off during the reaction. 2:4-Dihydroxyquinolines from α -naphthylamine and *m*-toluidine are described.

C. HOLLINS.

Manufacture of derivatives of benzanthrone. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,568, 15.2.29).—Halogen, nitro-, or hydroxyl substituents introduced by known methods into a 3-alkoxy-benzanthrone enter position 2. The preparation of the following compounds is described: 2-nitro-1-methoxybenzanthrone, m.p. 237°, by nitration with nitric acid in nitrobenzene at 45–50°; 2-bromo-1-methoxybenzanthrone, m.p. 186–188°; 2-chloro-1-ethoxybenzanthrone, m.p. 140–141°; 2-chloro-1-methoxybenzanthrone, m.p. 183–185°; 2:3-dihydroxybenzanthrone, m.p. 335° (dimethyl ether, m.p. 156–158°), from 3-methoxybenzanthrone by oxidation with manganese dioxide and sulphuric acid at 5–10°, followed by reduction with bisulphite; 9-chloro-2-nitro-3-methoxybenzanthrone, m.p. 288–290°, from 9-chloro-3-methoxybenzanthrone; 2:9-dinitro-3-methoxybenzanthrone, m.p. 290–291°, from 9-nitro-3-methoxybenzanthrone; 11-chloro-2:9-dinitro-3-methoxybenzanthrone, m.p. 285°, and the more soluble 2-mononitro-compound, m.p. 232–235°, from 11-chloro-1-methoxybenzanthrone.

C. HOLLINS.

Production of intermediates for vat dyes. D. A. W. FAIRWEATHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 332,907, 23.1.29).—Monosulphuric esters of leucoanthraquinones are obtained by removal of one ester group from a disulphuric leuco-ester by means of zinc and aqueous sodium hydroxide or of hydrochloric acid. Alternatively, a mixed sulphuric and organic ester of the leuco-compound may be hydrolysed to the monosulphuric ester, *e.g.*, with dilute sodium hydroxide solution. In the examples the starting materials are anthraquinone, 1- or 2-acetamidoanthraquinone, and 2-chloroanthraquinone.

C. HOLLINS.

Carrying out chemical reactions under pressure. [Manufacture of anthrarufin.] J. F. WAIT, ASSR. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,746,687, 11.2.30. Appl., 18.12.24).—In the manufacture of anthrarufin by hydrolysis of anthraquinonesulphonic acid in an autoclave under pressure, the rise in pressure and temperature is obtained by means of injected steam.

B. M. VENABLES.

Pyrolysis of unsaturated hydrocarbons (B.P. 332,998).—See II. **Leuco-esters of aminoanthraquinones etc.** (B.P. 333,507).—See IV. **Wetting agents etc.** (B.P. 310,941).—**Reserving agents for wool** (B.P. 333,559). **Colouring of higher fatty acids** (B.P. 309,148).—See VI. **Sodium salicylate** (U.S.P. 1,755,362).—See VII. **Methyl α -hydroxyethyl ketone and diacetyl** (B.P. 315,264).—See XVIII.

IV.—DYESTUFFS.

PATENTS.

Manufacture of dyes containing metals. Soc. CHEM. IND. IN BASLE (B.P. 311,385, 10.5.29. Switz.,

10.5.28).—The prechromed dyes of B.P. 301,772 (B., 1929, 165) are treated with solutions of salts of copper, cobalt, nickel, tungsten, uranium, aluminium, tin, titanium, or molybdenum, whereby a second metal is introduced. The chromium compound of nitro-1:2:4-aminonaphtholsulphonic acid \rightarrow β -naphthol gives two-metal complexes with copper sulphate, tungsten trioxide in sodium acetate, sodium uranate, cobaltous chloride, nickel sulphate.

C. HOLLINS.

Manufacture of [thioindigo] vat dyes. I. G. FARBENIND. A.-G. (B.P. 309,379, 8.4.29. Ger., 7.4.28).—Violet-blue to blue vat dyes are obtained by condensing substituted thioindoxyl with 5- or 7-alkoxyisatin α -halides etc. which contain further substituents. Examples are: 7-methoxy-4-methylisatin α -chloride with 5-chloro-7-methylthioindoxyl; 5-chloro-7-methoxy-4-methylisatin α -chloride with 5-chloro-7-methyl-5-chloro-4:7-dimethyl-, 5:7-dichloro-4-methyl-, 7-chloro-5-methyl-, 5-chloro-, or 5:6:7-trichloro-thioindoxyl; 7-chloro-5-methoxyisatin α -chloride with 5:6:7-trichlorothioindoxyl; 4-chloro-7-methoxyisatin α -chloride with 5-chloro-, 5:7-dichloro-, 5:6:7-trichloro-, 5:7-dichloro-4-methyl-, or 5-chloro-4:7-dimethyl-thioindoxyl.

C. HOLLINS.

Production of dry powders of anthraquinone acetate silk dyes. J. S. WILSON, E. G. BECKETT, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 333,236, 2.2.29).—Insoluble anthraquinone dyes for acetate silk are mixed with an alkali soap and dried below 50°. The state of dispersion of the dyes is thereby preserved.

C. HOLLINS.

Preparation of water-soluble dyes of the anthraquinone series. CHEM. FABR. VORM. SANDOZ (B.P. 333,188, 4.5.29. Ger., 27.11.28).—A 2-bromo-1-amino-4-*p*-acetamidoanilinoanthraquinone is heated with a sulphite, water, and phenol at 140°, whereby the bromine is replaced by a sulphonic group without hydrolysis of the acetamido-group. The treatment of the *p*-acetamido- and *p*-acetmethylamido-compounds is described.

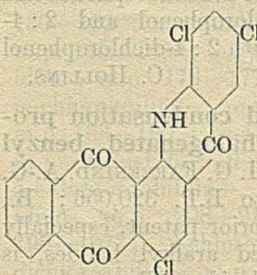
C. HOLLINS.

Manufacture of dianthraquinonylamines of the anthraquinoneacridone series [vat dyes]. I. G.

FARBENIND. A.-G. (B.P. 311,283, 7.5.29. Ger., 8.5.28).—An anthraquinone-1:2-acridone, halogenated in the 4-position of the anthraquinone residue as well as in the benzene residue, *e.g.*, the trichloro-compound having the annexed formula, is condensed with an aminoanthraquinone, *e.g.*, 1-amino-4- or -5-benzamidoanthraquinone, or α -aminoanthraquinone, to give grey vat dyes.

C. HOLLINS.

Manufacture of condensation products of the anthraquinoneacridone series. I. G. FARBENIND. A.-G. (B.P. 314,899, 3.7.29. Addn. to B.P. 311,283; preceding).—The process of the prior patent is extended to the condensation of the halogenated anthraquinone-acridones there specified with halogenated arylamines of the benzene or naphthalene series; *e.g.*, 2:5- and



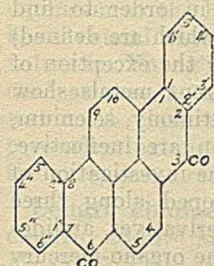
3 : 5-dichloro- and 3 : 4 : 5-trichloro-anilines give with trichloroanthraquinoneacridone bluish-green vat dyes.

C. HOLLINS.

Manufacture of green vat dyes of the [di]benzanthrone series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,762, 4.9.29).—Pure dimethoxydibenzanthrone obtained according to B.P. 181,304 (B., 1922, 582 A) is chlorinated with sulphuryl chloride in nitrobenzene at 80–90° in presence of ferric chloride, and, if desired, an acid-fixing agent (calcium oxide, magnesia). The products are brilliant green vat dyes.

C. HOLLINS.

Manufacture of vat dyes [dibenzpyrenequinones] containing halogens. I. G. FARBENIND A.-G., and J. Y. JOHNSON (B.P. 333,165, 22.4.29. Addn. to B.P. 287,050 and 287,845; B., 1929, 590, 591).—1 : 2 : 7 : 8-



Dibenzpyrene-3 : 6-quinones (other than iodo-derivatives) are halogenated in organic media (nitrobenzene) or in aqueous suspension or in absence of solvents, preferably in presence of halogen carriers (iron, iodine). The products dye redder shades than those of the prior patents, and are often pure red. The halogens probably enter positions 3', 4', 6' and 3'', 4'', 6'' (annexed formula) instead of at 5' and

5''. The preparation of chloro-, bromo-, chlorobromo-, dichloro-, and dichlorodibromo-derivatives is described.

C. HOLLINS.

Production of dyes and dye intermediates [azo dyes from diazotised leuco-esters of aminoanthraquinones or amino-vat dyes]. D. A. W. FAIRWEATHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 333,507, 8.2.29).—The sulphuric esters of leucoaminoanthraquinones or leuco-vat dyes containing amino-groups are diazotised by treatment with sodium nitrite and acid below 5°. The diazo compounds may be coupled with coupling components to give azo dyes still containing the leuco-ester residue. Examples are the following (the amine being used in the form of its leuco-ester): β -aminoanthraquinone \rightarrow β -naphthol, 2 : 3-hydroxynaphthoic 5-chloro-*o*-toluidide, anilide, etc.; 1 : 5-amino-naphthoic acid, phenol, resorcinol, acetoacetic toluidide; 3-chloro-2-aminoanthraquinone \rightarrow β -naphthol, carbonyl-J-acid, γ -acid, cresidine \rightarrow phenyl-J-acid, H-acid, *m*-phenylenediamine \leftarrow 4-nitro-*o*-aminophenol, 2 : 3-hydroxynaphthoic *p*-anisidine, β -naphthylamide, etc.; 2 : 6-diaminoanthraquinone, or α -aminoanthraquinone, or 1-chloro-2-aminoanthraquinone, or 5 : 5'-dichloro-7 : 7'-diaminothioindigo \rightarrow 2 : 3-hydroxynaphthoic 5-chloro-*o*-toluidide (cf. also B.P. 333,506; B., 1930, 1024).

C. HOLLINS.

Azo dyes. IMPERIAL CHEM. INDUSTRIES, LTD., and R. BRIGHTMAN (B.P. 333,513, 11.4.29. Addn. to B.P. 330,607; B., 1930, 897).—In place of 3 : 4'-diaminoazobenzene in the process of the prior patent there is used a 3-aminobenzene-1-azo-4'-aminonaphthalene. Examples are: *m*-nitroaniline \rightarrow α -naphthylamine \rightarrow salicylic acid, reduced, \rightarrow γ -acid (deep brown on viscose silk); 4-nitro-*o*-toluidine \rightarrow Cleve acid \rightarrow β -naphthol, reduced,

\rightarrow 2S-acid (blue); 4-nitro-*o*-anisidine \rightarrow Cleve acid \rightarrow *o*-cresotic acid, reduced, \rightarrow benzoyl- γ -acid (brown).

C. HOLLINS.

Manufacture of monoazo dyes for wool. I. G. FARBENIND. A.-G. (B.P. 311,708, 10.5.29. Ger., 14.5.28).—*p*-Nitroaniline-*o*-sulphonic acid is diazotised and coupled with an alkylated naphthylaminesulphonic acid in which the alkyl group contains more than two carbon atoms, e.g., isobutyl-2-naphthylamine-7-sulphonic acid for a blue-violet fast to washing, fulling, and light. [Stat. ref.]

C. HOLLINS.

Manufacture of substantive *o*-carboxyazo dyes containing copper. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 333,573, 10.5.29).—Azo dyes, obtained by coupling tetrazotised benzidine-3 : 3'-dicarboxylic acid with arylated or aroylated amino-naphtholsulphonic acids in which the aryl or aroyl residue carries a solubilising group, are converted into their copper complex compounds. The coupling may be made in the presence of a coppering agent (except cuprammonium compounds), or the latter may be added to the dye bath. Examples of coupling components are: *p*-5-hydroxy-7-sulpho-2-naphthylaminophenoxyacetic acid (violet-blue), the corresponding phenylthioacetic acid (blue), 5-sulpho-*o*-anisyl-J-acid (violet), ω -sulpho-*p*-tolyl-J-acid, *m*-carboxyphenyl-J-acid (blue-violet), *o*-carboxybenzoyl-J-acid (violet), *p*-5-hydroxy-7-sulpho-2-naphthylaminophenylacetic acid (violet-blue), the aminophenoxyacetic acids containing residues of J- and γ -acids (reddish-grey), 2-*p*-sulphophenoxyphenyl-J-acid (bluish-violet), *p*- β - γ -dihydroxypropoxyphenyl-J-acid (reddish-blue). C. HOLLINS.

Manufacture of dyes [of the azophthalein series]. J. R. GEIGY A.-G. (B.P. 312,330, 24.5.29. Ger., 24.5.28).—Azo couplings of resorcinol, especially with diazotised *o*-aminophenols, are condensed with 4-dialkylamino-2-hydroxybenzoyl-*o*-benzoic acids in sulphuric acid at 70° to give azophthaleins. Examples are: 4-dimethylamino-2-hydroxybenzoyl-*o*-benzoic acid with 3-amino-*o*-cresol-5-sulphonic acid \rightarrow resorcinol (reddish-blue on wool, blue on chroming) or with 4-chloro-*o*-aminophenol-6-sulphonic acid \rightarrow resorcinol (similar), or with anthranilic acid \rightarrow resorcinol (chromed brown-red on wool); 4-diethylamino-2-hydroxybenzoyl-*o*-benzoic acid with 1 : 2 : 4-aminonaphtholsulphonic acid \rightarrow resorcinol (greenish-blue). C. HOLLINS.

Manufacture of water-insoluble azo dyes on the fibre [ice colours]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 332,940, 28.3.29).—Whilst 2 : 4-dihydroxyquinoline gives valueless dyeings when coupled on the fibre, the presence in the quinoline molecule of further fused rings or of arylazo or arylazimino-substituents, or the direct linking together of two 2 : 4-dihydroxyquinolyl residues, confers improved affinity for the fibre, and such quinoline derivatives give useful ice colours. Examples are: 2 : 2' : 4 : 4'-dihydroxy-6 : 6'-diquinolyl with diazotised 1-aminoanthraquinone (brown-red) or *o*-aminodiphenyl ether (orange-yellow); 2 : 4-dihydroxy- α -naphthaquinoline with diazotised 2 : 5-dichloroaniline (brown-orange) or aminoazotoluene (yellow-red); 1 : 3-dihydroxy- β -naphthaquinoline, m.p. 345°, with diazotised 5-nitro-*o*-toluidine (red-orange) or

1-aminoanthraquinone (red-orange); 2:4-dihydroxy- $\beta\beta$ -naphthaquinoline, m.p. 355°, with diazotised 2:5-dichloroaniline (orange-yellow); 2-(2:4-dihydroxy-6-quinolyl)benztriazole, m.p. 330°, with diazotised 4:6-dichloro-*m*-toluidine (red-yellow) or 4-chloro-*o*-anisidine (yellow-orange); 2:4-dihydroxy-6-quinolineazo-2-chlorobenzene, m.p. 345° (prepared from *o*-chloroaniline \rightarrow anthranilic acid and ethyl malonate), with diazotised 4:6-dichloro-*m*-toluidine (golden-yellow) or 2:5-dichloroaniline (pure yellow). C. HOLLINS.

Manufacture of azo dyes insoluble in water [ice colours and pigments]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 332,932, 21.3.29. Cf. B.P. 331,247; B., 1930, 942).—2:3-Hydroxynaphthoic 4-chloro-2:5-diethoxyanilide is coupled with a non-carboxylated and nonsulphonated diazo or tetrazo compound in substance or on the fibre. The dyeings are fast to light and to bucking with 0.2% sodium hydroxide for 6 hrs. without pressure. Examples of diazo components are 2:5-dichloroaniline (orange-brown), 4-chloro-*o*-toluidine (yellow-red), 2:5-dichloro-*p*-toluidine (brick-red), 4:6-dichloro-*m*-toluidine (brown-red). C. HOLLINS.

Manufacture of vat dyes and intermediates derived from pyranthrone. A. SHEPHERDSON and A. J. HAILWOOD, ASSTS. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,776,200, 16.9.30. Appl., 5.5.27. U.K., 7.7.26).—See B.P. 278,102; B., 1927, 901.

Chromium compounds of azo dyes. H. KÄMMERER and K. HOLZACH, ASSTS. to GEN. ANILINE WORKS, INC. (U.S.P. 1,775,644, 16.9.30. Appl., 17.2.28. Ger., 6.8.25).—See B.P. 297,722; B., 1928, 888.

Naphthoylenediaryliminazole derivatives (B.P. 334,111).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Silk-soaking process. I. Effect of soap and other alkali on silk sericin. R. HART. **II. Soap and oil as soaking agents.** R. HART and G. W. SEARELL (Ind. Eng. Chem., 1930, 22, 980—983, 984—985).—I. On soaking raw silk in a solution of soap there is a marked decrease in the concentration of fatty material and particularly of alkali in the solution, which is due to absorption by the silk and to interaction between the silk sericin and the alkali of the soap. At the same time a large amount of free fatty acid is liberated, which is found both in the treated silk and in the spent liquor. The quantitative distribution of the ingredients after soaking is tabulated. It appears that a small but constant fraction of the sericin is capable of reacting with an excess of alkali in the cold, its combining weight being probably of the order of that of fatty acids.

II. The composition of the liquor and soaked silk was examined after soaking raw silk in an emulsion of neat's-foot oil and soap; the fatty matter absorbed by the silk consisted of almost equal parts of free fatty acid and soap. F. R. ENNOS.

Study of silk fibroin in the dispersed state. M. HARRIS and T. B. JOHNSON (Ind. Eng. Chem., 1930, 22, 965—967).—Colloidal suspensions of silk fibroin, prepared by grinding in the pebble mill, extracting with

water, and removing the larger particles by centrifuging, contain no amino-nitrogen, are immune to bacterial attack, are digested by trypsin, and have $[\alpha]_D^{20} -43.98^\circ$. When the protein is colloiddally dissolved by means of concentrated aqueous solutions of neutral salts (50% lithium bromide or 70% calcium thiocyanate) and subsequent dialysis to remove the salts, a partial hydrolysis independent of temperature occurs, as is shown by the conversion of about 5% of the total nitrogen into the amino-form, the power of the fibroin to support bacterial growth, and a change of the rotation towards dextro-rotation. F. R. ENNOS.

Inhibitory action of certain substances on the growth of mould fungi [on cotton goods]. R. G. FARGHER, L. D. GALLOWAY, and M. E. PROBERT (J. Text. Inst., 1930, 21, T245—260; cf. Morris, B., 1927, 470).—Many inorganic and organic substances have been prepared and examined in order to find an ideal antiseptic (the properties of which are defined) for use in the cotton industry. With the exception of those of thallium, the salts of different metals show no outstanding toxicity. Arsenic, antimony, selenium, and thallium in organic combination are ineffective, whilst boron is somewhat better. The investigation of organic compounds has been developed along three lines of attack: acetanilide and its derivatives, anilides of acids other than acetic acid, and the organo-mercury compounds. The introduction of substituents into acetanilide leads to a fall in toxicity which is slight for methyl (*o* or *p*), chlorine (*p*), or nitro- (*p*), and marked for hydroxyl (*p*) or *N*-methyl. The toxicities of acet- β -naphthylamide and 8-acetamidoquinoline are similar to that of acetanilide, whilst acet- α -naphthylamide, 5-acetamidoquinoline, and 2-acetamidopyridine are relatively toxic. Acet-*o*-toluidide, acet- α -naphthylamide, and 5-acetamidoquinoline, which contain a carbon substituent *ortho* to the acetamido-group, are less toxic, respectively, than acet-*p*-toluidide, acet- β -naphthylamide, and 8-acetamidoquinoline. Combination of aniline with other acids yields only one promising substance, salicylanilide, but this is as toxic as thallium carbonate, fulfils all the conditions for the ideal antiseptic (except for its slight solubility), and its use is patented under the name "Shirlan." It is a poor bactericide, but appears to be non-toxic to plant life. Introduction of mercury into the nucleus of phenol gives rise to two monosubstituted derivatives, and the more soluble of these, tested as the *o*-chloromercuriphenol, is seven times as toxic as mercuric chloride, whilst the less soluble isomeride is only as toxic as the latter. The order of decreasing toxicity with substituent groups is given, and it is found that solutions of these mercury compounds are much more stable to metals than are the inorganic mercury salts. The outstandingly toxic substances found are salicylanilide, *o*-chloromercuriphenol, *p*-acetoxymercuriacetanilide, thallium carbonate, *p*-nitrophenol, and trichlorophenol. The organisms and methods of testing employed, the preparation of salicyl derivatives by means of salicyl chloride and salol, and of the mercury derivatives of phenol, the mercuriation of aniline and *p*-toluidine, and the chemical characteristics of many of the compounds prepared are described. B. P. RIDGE.

Swelling of cotton hairs in water and in air at various relative humidities. G. E. COLLINS (J. Text. Inst., 1930, 21, T311—315).—A close parallelism exists between the extent of the dimensional changes of cotton hairs and the amount of absorption between the temperature limits 20° and 100°. No evidence of increased swelling at temperatures up to 200° has been obtained.

B. P. RIDGE.

Extensibility of cotton hairs. G. E. COLLINS (J. Text. Inst., 1930, 21, T316—324).—The extension and recovery of single hairs from a sample of cotton previously treated with sodium hydroxide solution to smooth out the convolutions have been observed at various humidities from zero to 100%. Extensibility increases considerably with increase in humidity, and is about 8—10 times as great at saturation as at zero humidity. The hair makes a more complete recovery from a given extension at the higher humidity. On the whole, fine hairs extend rather less than coarse ones. Absolute values for Young's modulus range from 6×10^{10} dynes/cm.² at zero humidity to 0.6×10^{10} dynes/cm.² in water. A close correlation is obtained between the percentage extension imposed at a given humidity and the subsequent recovery.

B. P. RIDGE.

Length changes of cotton hairs in solutions of caustic soda. M. A. CALVERT (J. Text. Inst., 1930, 21, T293—308).—The shrinkage of single cotton hairs, not previously submitted to a swelling treatment, when immersed in 26.8% sodium hydroxide solutions has been investigated under various conditions for raw, scoured, chloroform-extracted, and mechanically treated (rubbed) hairs. The results obtained are expressed as curves showing the relation between percentage contraction and concentration of the immersion liquid. Scoured hairs show a maximum "free" shrinkage (*i.e.*, shrinkage without loading) near to 13% concentration, whilst the shrinkage of raw hairs under these conditions is much less, and the peak of 13% concentration is absent. Restricted shrinkage (shrinkage under continuous load) for scoured hairs gives curves similar to those for raw cotton hairs under free shrinkage. The restricted shrinkage of hairs extracted with chloroform is not increased by the removal of fats and waxes, whilst the severity of the scouring treatment has no effect on free shrinkage, since the latter is as great for water-boiled hairs as for those scoured with caustic soda solution under pressure. Mechanical treatment of the hairs in such a way as to remove the cuticle increases free shrinkage, which then shows a maximum near to 11% sodium hydroxide concentration. In this respect the cuticle-free hairs resemble bast fibres, which have no cuticle, and show a pronounced maximum for concentrations of 8.8—11%. The effect of removing the cuticle of the cotton hair is also reflected by marked increase in the width of the hairs on treatment with the sodium hydroxide solutions as compared with the corresponding width increases for raw and soda-boiled material. The apparatus and technique employed in making the observations are described.

B. P. RIDGE.

Determination of "soundness" in wool and cloth. C. RIMINGTON (J. Text. Inst., 1930, 21, T237—244).—The mechanism of the chemical reaction between

tyrosine and Pauly's reagent (diazobenzenesulphonic acid), which forms the basis of the method elaborated, is discussed. A weighed sample of the wool is treated with the reagent under standardised conditions, dissolved in 10% sodium hydroxide solution, and the colour intensity of the resulting liquid is matched against that of a 0.1% solution of the dye New Acid Brown S. The degree of damage of the sample is expressed numerically in such a way that 100 units of damage represent the extent of unsoundness present when 0.1 g. of the wool in question, after the above treatment, yields a solution the colour intensity of which is exactly equal to that of the standard solution of dye. The factors which influence the method are examined, and a simplified procedure for use in works is described.

B. P. RIDGE.

Mechanism of the deformation of fibres. K. ECKLING and O. KRATKY (Naturwiss., 1930, 18, 461—464).—The mechanism of the process of deformation of a cellulose ester thread and of the chemical changes which are involved during its conversion into cellulose are discussed, having regard to changes in the lattice arrangement, slip of the molecules parallel to their axis but without orientation, irreversible changes of the degree of dispersion, and the conditions governing the extension of a system of rod-shaped bodies immersed in a viscous medium. Threads subjected to but slight tension do not on hydrolysis of the ester exhibit any marked increase of the orientation of the particles, but threads which have been extended by a considerable force exhibit markedly greater orientation after hydrolysis; the cause of this phenomenon is, however, obscure.

H. F. GILLBE.

Cooking process. II. Cooking wood with sodium carbonate. S. I. ARONOVSKY and R. A. GORTNER (Ind. Eng. Chem., 1930, 22, 941—945; cf. B., 1930, 552).—Aspen sawdust was cooked with 20% and 40% sodium carbonate solutions (dry weight) at 170° and 186° for 2 and 12 hrs. under 100—150 lb. steam pressure. Comparison of the composition of the resulting residues and black liquors with that of those obtained when water alone was used showed that sodium carbonate cannot be regarded as an inert ingredient in a cooking liquor. The residual woods obtained after the sodium carbonate treatment were lighter in colour, softer, and contained more cellulose and less α -cellulose, whilst the liquors after cooking were richer in total organic matter, lignin, and volatile acids than the corresponding fractions obtained by cooking with water; the more stable pentosans associated with the cellulose were not removed by the sodium carbonate cook.

F. R. ENNOS.

Effect of fine division on the solubility of cellulose. D. F. J. LYNCH (Ind. Eng. Chem., 1930, 22, 952—953).—The solubility of cellulose, *e.g.*, peanut-hull pulp, cottonwood pulp, etc., in cold concentrated (17.5%) sodium hydroxide solution (α -cellulose test) is not materially affected by the state of division, but in hot dilute (7.14%) sodium hydroxide (nitrate's alkali-soluble determination) a very fine degree of division increases the solubility, although the mere cutting of fibres into short lengths has little effect.

F. R. ENNOS.

Relationship between α -cellulose content and potassium hydroxide solubility of certain

degraded celluloses. H. LEB. GRAY, C. J. STAUD, and J. T. FUSS (Ind. Eng. Chem., 1930, 22, 1018—1020).—Determinations were made of the α -cellulose content and solubility in hot 10% potassium hydroxide solution of a series of oxidised celluloses and hydrocelluloses prepared by the action of acid potassium permanganate and hydrochloric acid solutions, respectively. Down to 75% of α -cellulose the elliptical curves showing the relationship between the α -cellulose content (x) and the potassium hydroxide solubility (y) are identical for both series of degraded celluloses, and are expressed by $y^2 = 697.2 + 45x - 0.526x^2$.

F. R. ENNOS.

Viscosity and strength of the jelly and film of acetylcellulose. T. ARAKI and M. KUSAGAWA (J. Soc. Chem. Ind., Japan, 1930, 33, 267B).—The jelly strength of the reversible gel of acetylcellulose (8 varieties) and benzyl alcohol, and the viscosity of dopes prepared from the former, were measured. The jelly strength of 10% gel at 10° became nearly constant after 5–6 hrs. cooling, and the values of various samples from the same manufacturers were inversely proportional to the viscosity. The quality of the acetylcellulose cannot be evaluated exactly from the tensile strength (6–7 kg./mm.²) of films obtained by drying the cellulose solutions on a glass plate.

E. LEWKOWITSH.

Determination of the viscosity of cuprammonium solutions of cellulose. J. TANKARD and J. GRAHAM (J. Text. Inst., 1930, 21, T260—266).—Sufficient cellulosic material to give a 1, 1.5, or 2% solution (according to the extent of chemical modification of the cellulose) is placed in a glass tube, 28 cm. long and 1 cm. internal diam., which is closed at one end and contains a weight formed from a short piece of steel rod. Cuprammonium solution containing copper 15 g., ammonia 240 g., and nitrite <0.5 g. per litre is admitted and the tube is closed with a special stopper and slowly rotated end-over-end for several hours. The measurement of viscosity is carried out at constant temperature (25°) by the falling-sphere method, using steel balls $\frac{3}{16}$ in. in diam., and the following advantages are claimed: accurate determinations of viscosity can be made over a wide range; Ladenburg's correction for wall effect is applicable, thus obviating the necessity for calibrating each tube; small variations in the diameter of the tube are unimportant; duplicate measurements can be made on each solution; and the special stopper ensures that the volume of solution for any one tube is constant in all experiments. Methods previously employed for the determination of the viscosity of solutions of cellulose in cuprammonium are discussed.

B. P. RIDGE.

Recovery of viscose spinning bath. I. Rapid analysis of sodium sulphate in the spinning bath. K. TANEMURA and S. MIYOSHI. II. **Sp. heat of Glauber's salt.** K. TANEMURA (J. Soc. Chem. Ind. Japan, 1930, 33, 323—324 B, 324—325 B).—I. The temperature at which sodium sulphate begins to crystallise out is observed, its connexion with the sodium sulphate content having been previously determined and graphed.

II. The sp. heat of sodium sulphate decahydrate is calculated from vapour pressure measurement and other data to be 0.42.

C. W. GIBBY.

Higher orientation in cellulose materials. I. K. HESS and C. TROGUS (Naturwiss., 1930, 18, 437—441).—The orientation of the cellulose molecules in a variety of substances such as paper, cellophane, vulcanised fibre, and presspahn has been studied by means of X-ray photographs. The alterations of the mechanical properties of such materials by calendering and analogous processes are correlated with the changes of molecular orientation.

H. F. GILLBE.

Higher orientations in cellulose materials. II. X-Ray diagram of paper. K. HESS and C. TROGUS (Z. physikal. Chem., 1930, B, 9, 169—172; cf. preceding abstract).—From the X-ray examination of paper made from ordinary and mercerised cellulose and of kaolin it has been shown that in paper cellulose has a higher degree of orientation than in the form of fibres, and that when kaolin is used as a filler it, too, is oriented.

R. CUTHILL.

Automatic recording waterproof tester. A. L. HODGES (Phil. Mag., 1930, [vii], 10, 327—328).—A device, simple in principle, is described which accurately records the time taken by water to seep through various fabrics and papers.

W. GOOD.

Fine structure of artificial silks. HALL.—See VI. **Fermentation of cornstalks.** BORUFF and BUSWELL.—See XVIII. **Decomposition of photographic films.** OLSEN and others.—See XXI.

PATENTS.

[Shaped] bodies from fibrous materials. INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS GES. (B.P. 312,178, 21.5.29. Ger., 21.5.28).—In the manufacture of billiard or skittle balls etc., strips of woven fibrous material are compressed together with artificial resin, e.g., phenol-formaldehyde condensation products, and subjected to heat and pressure.

H. ROYAL-DAWSON.

Humidity-control apparatus [for fabrics]. H. A. BRITAIN, Assr. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,757,931, 6.5.30. Appl., 17.7.25).—Samples of fabric are brought to a moisture content of 6% or other definite figure by suspending them in a current of air that has been bubbled through sulphuric acid of dilution requisite to give the necessary relative humidity, irrespective of the original condition of the air.

B. M. VENABLES.

Manufacture of cellulose derivatives. BRIT. CELANESE, LTD. (B.P. 315,278, 10.7.29. U.S., 10.7.28).—Cellulosic material is treated with an alkylating or aralkylating agent (ethyl sulphate) in the presence or absence of a small quantity of an alkaline material (sodium carbonate or hydroxide) for several hours at 0–45°, and afterwards with caustic soda in the solid form or in concentrated solution, the temperature being maintained at 30–60° until the cellulose ether is formed.

F. R. ENNOS.

Manufacture of silk from acylcellulose. I. G. FARBENIND. A.-G. (B.P. 312,671, 29.5.29. Ger., 30.5.28. Addn. to B.P. 233,342; B., 1925, 985).—The material is spun into a salt solution (ammonium nitrate), to which is added an inorganic salt (ammonium thiocyanate, zinc chloride) having a swelling or solvent action on the

acylcellulose, together with sugar and a small quantity of a solvent (acetone), if desired. F. R. ENNOS.

Production of filaments, threads, yarns, ribbons, etc. from cellulose esters and ethers. BRIT. CELANESE, LTD., and (A) W. I. TAYLOR and R. P. ROBERTS, (B) H. DREYFUS, W. I. TAYLOR, and R. P. ROBERTS (B.P. 334,195 and Addn. B.P. 334,198, [A] 28.3.29, [B] 21.5.29).—Products of relatively low lustre are made by extruding solutions of cellulose esters or ethers in (A) a volatile solvent (acetone) or (B) a mixture of a volatile solvent and a diluent (water) into an evaporative medium which is at a higher temperature than the spinning solution, the latter being maintained at 10–20° below the minimum temperature required for the production of products of normal high lustre; the temperatures of both spinning solution and evaporative medium may be separately controlled by the methods of B.P. 320,632 (B., 1930, 53). F. R. ENNOS.

Treatment of materials containing cellulose esters. BRIT. CELANESE, LTD. (B.P. 316,521 and Addn. B.P. 318,468, [A] 29.7.29, [B] 2.9.29. U.S., [A] 28.7.28, [B] 1.9.28).—Fabrics, yarns, etc. of cellulose esters are superficially saponified by application of (A) a paste consisting of caustic alkali and a thickening agent (gum tragacanth, starch, etc.) or (B) a solution containing caustic alkali, after which they are immediately dried by heat and thoroughly steamed or washed, whereby the temperature at which they may be safely ironed or calendered is raised. F. R. ENNOS.

Compositions or materials comprising cellulose derivatives [of reduced inflammability]. BRIT. CELANESE, LTD. (B.P. 312,609, 27.5.29. U.S., 28.5.28).—Plastic or celluloid-like masses, moulding powders, films, lacquers, artificial fibres, etc. having a basis of cellulose esters or ethers are rendered less inflammable by incorporating therein, at any convenient stage, one or more aliphatic acyl derivatives of nuclear-chlorinated arylamines of the benzene series, *e.g.*, *o*-chloroacetanilide. F. R. ENNOS.

Treatment of cellulosic fibres with alkali. G. TAGLIANI, Assr. to CHEM. WORKS, FORMERLY SANDOX (U.S.P. 1,776,052, 16.9.30. Appl., 7.9.27. Ger., 29.10.26).—See B.P. 279,784; B., 1928, 228.

[Photographic] manufacture of wallpapers. J. MACCALLUM and M. O'HARA (B.P. 334,591, 7.6.29).

Copper from ammoniacal solutions (B.P. 313,045 and 310,425).—See VII. **Compound glass** (B.P. 316,955).—See VIII. **Saturant for fibrous bases** (B.P. 334,232).—See XIII. **Rubberised material** (B.P. 332,537).—See XIV. **Cellulose and pulp** (B.P. 332,935).—See XV. **Film-forming element** (U.S.P. 1,746,751). **Photographic paper and films** (B.P. 318,511 and U.S.P. 1,752,665).—See XXI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Fine structure of artificial silks in relation to dyeing and finishing processes. A. J. HALL (J. Soc. Dyers and Col., 1930, 46, 257–261).—The rigidity, creasability, lustre, and dyeing properties of viscose and cellulose acetate fibres are correlated with the

parallelism of their micelle chains and the effect on these of stretching. Methods of measuring these properties are described. Resistance to creasing decreased rapidly for cellulose acetate and less rapidly for viscose silk with increased stretching; a small amount of stretching is thus able seriously to reduce the resistance to creasing of artificial silk materials. The rigidity of artificial silk yarns increased with stretching. Curves show that cellulose acetate and viscose silk yarns gradually lose their power of recovering to their original length as the degree of stretching is increased; viscose yarn shows less power than cellulose acetate yarn to recover. It is considered that parallelism of the micelle chains in viscose fibres as produced by stretching at the time of spinning or during dyeing reduces the affinity of the fibres for direct dyes (cf. Hall, B., 1929, 430); parallelism also gives increased lustre. The retention of lustre by cellulose acetate silk when immersed in a stretched condition in boiling water is ascribed to the effect of the stretching in maintaining the cellulose acetate micelle chains parallel (cf. B.P. 277,089; B., 1929, 596). Greater parallelism of the micelle chains confers increased rigidity and creasability on both types of silks. A. J. HALL.

Diagnosis of colour faults in finished [wool] goods. F. L. GOODALL (J. Soc. Dyers and Col., 1930, 46, 263–267).—Residual alkali in scoured wool materials is frequently responsible for the appearance of various defects in subsequent dyeing processes. Wool containing alkali will, if dyed while still wet, dye a much fuller shade than wool free from alkali; the difference in shade is less marked if the wool is dried before dyeing. Alkaline moist wool is readily attacked by bacteria in such a way that it loses its affinity for acid and chrome colours and gains an increased affinity for basic dyes. Stained and off-shade faults may be produced during the steaming and particularly the drying of dyed wool containing residual alkali. The steaming of undyed wool containing alkali increases very considerably its affinity for dyes. Fading faults in dyed wool materials may be caused by the presence of alkali and sulphur dioxide. Thus bisulphite formed by interaction might combine with certain azo dyes to form azo sulphites strikingly different in colour from that of the parent dyes (cf. King, B., 1928, 639, 707; Goodall, B., 1928, 639). Such sulphur dioxide may be derived from the presence in the wool material of sulphur-dyed cotton yarn or wool yarn which has been previously stored, or from paper wrappers made from sulphite wood pulp, or may arise from oxidation of the natural sulphur content of wool itself. Again, under optimum conditions, sodium bisulphite may cause faults by complete reduction of the dye molecule, as in the presence of sodium hyposulphite; Erio Floxine 2GI is susceptible to such attack, and some vat dyes have been found to be affected. Azo sulphites are not so fast to light as are the parent dyes, so that uneven fading of dyed wool may result from the uneven distribution of sodium bisulphite within it. A. J. HALL.

Flash points of mixed solvents. SNELL.—See III. **Length changes of cotton hairs in solutions of caustic soda.** CALVERT.—See V.

PATENTS.

Manufacture of agents for wetting, cleansing, and emulsifying, and for protecting fibre. ORANIENBURGER CHEM. FABR. A.-G. (B.P. 310,941, 3.5.29, Ger., 3.5.28).—A neutral fat or a fatty acid is condensed with a hydrocarbon and an organic compound containing oxygen or sulphur, and sulphonated, in one or more stages. *E.g.*, a mixture of castor oil, xylene, and acetic anhydride is treated with chlorosulphonic acid at 27°.

C. HOLLINS.

Dyeing with the reserving of wool. Manufacture of reserving agents for wool. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 333,554 and 333,559, [A] 6.5.29, [B] 9.5.29).—(A) Wool in unions is reserved against direct cotton dyes by adding to the dye-bath an *N*-alkylated, -aralkylated, or -arylated amide of a sulphonic or carboxylic acid, *e.g.*, bis-(1:2-dichlorobenzene-4-sulphonyl)benzidine-2:2'-disulphonic acid. (B) Such amides are made by condensing 2 mols. of an aromatic sulphonyl or carboxylic halide with 1 mol. of an arylenediamine, the reactants being chosen so that the product contains one or more free sulphonic acid groups, but no nitro-, hydroxyl, or unsubstituted amino-group, and at least one of the reactants must contain a substituent *meta* to the reacting group. The bis-1:2-dichlorobenzene-sulphonyl derivatives of benzidine-2:2'-disulphonic acid and of 2:4-tolylenediamine-6-sulphonic acid are excluded from the claims. Examples are: bis-(2:4-dichlorobenzoyl)-*m*-phenylenediamine-4'-sulphonic acid, bis-(*o*-xylene-4-sulphonyl)-, bis-(2-chlorotoluene-4-sulphonyl)-, bis-(1:2:3-trichlorobenzene-4-sulphonyl)-, bis-[1-chloro-4-(1':2'-dichlorobenzene-4-sulphonamido)benzene-2-sulphonyl]-, bis-[3-(1':2'-dichlorobenzene-4-sulphonamido)benzene-3-sulphonyl]- and bis-(1:2-dichlorobenzene-4-sulphonamidobenzene-*m*-sulphonyl)-benzidine-2:2'-disulphonic acids, and bis-(*m*-sulphobenzoyl)-2:2'-dichlorobenzidine.

C. HOLLINS.

Dyeing etc. [with azo dyes containing a leucoanthraquinone or leuco-vat dye ester residue]. D. A. W. FAIRWEATHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 333,506, 8.2.29).—The dyes of B.P. 333,507 (B., 1930, 1019) are applied to the fibre and, if desired, developed thereon by oxidation and hydrolysis as in dyeing or printing with Soledon colours. The dyes may also be produced by coupling on the fibre, with or without subsequent development. Mordant metals may be applied before or after development. The leuco-ester may be the coupling component, *e.g.*, *p*-nitroaniline \rightarrow leuco-2-aminoanthraquinone sulphuric ester gives a red-purple changing to brown on development.

C. HOLLINS.

Dyeing of regenerated cellulose materials. IMPERIAL CHEM. INDUSTRIES, LTD., and (A) and (B) R. BRIGHTMAN, (C) R. BRIGHTMAN and W. L. B. WELLCOTT (B.P. 333,239, 333,243, and 333,260, [A] 7.2.29, [B] 8.2.29, [C] 5.3.29. Cf. B.P. 331,839; B., 1930, 983).—Viscose silk is dyed evenly by the application of disazo dyes of the types: (A) *p*-phenylenediamine \rightarrow 1 mol. of α -naphthol, α -naphtholsulphonic acid, or an *N*-substituted 2:8-aminonaphtholsulphonic acid, and 1 mol. of a 1:8- or 2:8-aminonaphtholsulphonic acid, the *p*-diamine being

applied as usual as a *p*-nitroaniline or a *p*-aminoacetanilide with reduction or hydrolysis after the first coupling; (B) a naphthylaminesulphonic acid, or an aminonaphtholsulphonic acid other than J-acid, \rightarrow middle component of the benzene series \rightarrow a 1:8-aminonaphtholsulphonic acid; (C) a 3:3'-diaminodiphenylcarbamide not carrying carboxylic or sulphonic groups \rightarrow 1 mol. of a carbonylated or sulphonated phenol or naphthol and 1 mol. of a sulphonated naphthylamide, naphthol, dihydroxynaphthalene, aminonaphthol, or β -substituted aminonaphthol, the carbamide dyes being also obtainable by phosgenation of suitable aminoazo compounds. Both (A) and (B) give blue or greenish-blue dyeings; (C) gives orange to brown and violet. Examples are: (A) 6-nitro-cresidine \rightarrow β -naphthol, reduced \rightarrow 2 S-acid; 5-nitro-*o*-anisidine \rightarrow N.W.-acid, reduced, \rightarrow γ -acid or S-acid; 4-chloro-5-nitro-*o*-anisidine \rightarrow benzoyl- γ -acid, reduced, 2 S-acid; (B) 2:8-aminonaphthol-3:6-disulphonic acid \rightarrow 2:5-dimethoxyaniline \rightarrow S-acid; γ -acid \rightarrow cresidine \rightarrow 2 S-acid; Brönner acid \rightarrow *m*-toluidine \rightarrow S-acid; Brönner acid \rightarrow *o*-anisidine \rightarrow H-acid; (C) 3:3'-diaminodiphenylcarbamide \rightarrow salicylic acid and γ -acid (yellowish-brown), or salicylic acid and M-acid (maroon), or *o*-cresotinic acid and benzoyl- γ -acid (pink); *m*-nitroaniline \rightarrow salicylic acid, reduced, and phosgenated with reduced *m*-nitroaniline \rightarrow H-acid (bluish-red); 4-nitro-*o*-anisidine \rightarrow Schäffer acid, reduced, and phosgenated (red-violet).

C. HOLLINS.

Colouring of [cellulose ester or ether] textile materials. BRIT. CELANESE, LTD., G. H. ELLIS, and W. B. MILLER (B.P. 332,624, 23.2.29).—The artificial silks, *e.g.*, cellulose acetate silk, are dyed with oxidation Aniline Black in the presence of considerably greater proportions of acid than those commonly used in the dyeing of cotton materials; the excess of acid may be in the form of an acid salt or mineral acid, but preferably as an organic acid. A satisfactory solution for dyeing acetate silk by the padding and ageing method is prepared by mixing a solution containing aniline hydrochloride 127 g., glacial acetic acid 25 g., water 248 g., alcohol 100 g., and 50% gum arabic solution 200 g. with a solution containing sodium chlorate 80 g., cupric chloride 3 g., and water 217 g., immediately before use.

A. J. HALL.

Production of differential [dyeing] effects on fabrics made of or containing organic esters of cellulose. LYONS PIECE DYE-WORKS (B.P. 310,844, 26.4.29. U.S., 1.5.28).—Fabric is saponified according to any desired pattern by application of an alkaline paste, by printing, brushing, or spraying methods, then drying, steaming, and washing; the saponified parts are then dyed with cotton dyes. The paste may contain the hydroxides or carbonates of sodium and potassium, together with a swelling agent such as pyridine, acetone, and alkali thiocyanates.

A. J. HALL.

Protection of wool, fur, feathers, hair etc. from attack by moth. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 333,584, 15.5.29).—Hydroxylated diphenyls, *e.g.*, 2:2'-dihydroxydiphenyl, its 3:3':5:5'-tetrabromo- or -tetrachloro-, dichloro-, dibromo-, and dichlorodibromo-derivatives, are used for mothproofing.

C. HOLLINS.

Production of white and coloured discharges [on ice colours from 2:3-hydroxynaphthoic arylamides]. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 332,986, 22.5.29).—Goods padded with a 2:3-hydroxynaphthoic arylamide are printed with a non-volatile acid and developed with a diazo compound which does not couple in acid medium. For coloured discharges an acid-coupling diazo compound or a soluble leuco-ester of a vat dye is used for development. Examples are: anilide, printed with aluminium sulphate, developed with diazotised 4-amino-4'-methoxydiphenylamine for white on blue; *p*-chloroanilide, printed with aluminium sulphate or lactic acid, developed with diazotised 4-amino-4'-methoxydiphenylamine for white on navy-blue; anilide, printed with diazotised 4-chloro-*o*-toluidine and aluminium sulphate, developed with diazotised 4-amino-4'-methoxydiphenylamine for red on blue; 5-chloro-*o*-toluidide, printed with diazotised *m*-chloroaniline and aluminium sulphate, developed with diazotised 6-benzamidocresidine for orange on violet; anilide, printed with leucotetrabromindigo hydrogen sulphate, ammonium oxalate and vanadate, and sodium chlorate, developed with diazotised 4-amino-4'-methoxydiphenylamine for light blue on dark blue. In each case the goods are passed through an acid bath after development. C. HOLLINS.

Protection of wool, fur, etc. from attack by moth and other textile pests. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 333,583, 15.5.29).—Organic fluorine compounds are applied to wool, fur, etc. as mothproofing agents. Examples are 4-fluoro-3-nitrobenzoic acid, fluoroacetic acid, 1:5-fluoronaphthalene-sulphonic acid, 4:4'-difluorodiphenyl, fluoro- ψ -cumene, fluoroform; the last-named is suitable for use as vapour in treating upholstered furniture. C. HOLLINS.

Improvement of textiles and the like by rendering them resistant to attack by textile pests [moths], bacteria, and mould. I. G. FARBENIND. A.-G. (B.P. 312,163, 14.5.29. Ger., 21.5.28).—The textile is treated with a quaternary phosphonium salt, and the phosphorus compound may, if desired, be insolubilised by incorporation of other salts on the fibre. Examples are: triphenylbenzylphosphonium chloride with or without after-treatment with potassium perchlorate; sulphonated chlorocresotic anilide as precipitant for triphenylbenzylphosphonium sulphate subsequently applied; benzyl-triethylphosphonium chloride; triphenyl-*p*-chlorobenzylphosphonium chloride and talcum. C. HOLLINS.

Treatment of textile filaments, yarns, threads, or the like. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 332,290, 11.1.29. Cf. B.P. 332,263; B., 1930, 944).—Yarns are treated in a continuous manner by means of suitable rotary, oscillatory, or reciprocatory devices at regular or irregular spaced intervals along their length (cf. B.P. 325,823; B., 1930, 553) with a mechanical reserve or a substance otherwise capable of modifying the dyeing properties of the yarn (*e.g.*, an alkali is used for saponifying cellulose acetate and ester yarns) and are then utilised in the manufacture of textile materials which are to be dyed subsequently; alternatively, the treated yarns are dyed before use in manu-

factured goods. Multicoloured goods are thus obtained. A. J. HALL.

Manufacture of vulcanised fibres and the like. I. G. FARBENIND. A.-G. (B.P. 312,998, 31.5.29. Ger., 4.6.28).—A roller or continuous process is employed in which the sulphuric acid ordinarily used is replaced by a mixture of sulphuric acid, an inorganic salt, and an organic substance which is soluble, but stable, in the acid; the destructive action of the parchmentsing acid is thus reduced. Artificial tanning agents (*e.g.*, phenol-formaldehyde condensation products) and ureas are suitable organic substances, and the amount of inorganic salt (*e.g.*, magnesium or ammonium sulphate) used may reach the saturation point. A. J. HALL.

Weighting of natural silk. R. CLAVEL (B.P. 332,778, 15.8.29. Ger., 16.3.29. Addn. to B.P. 266,640; B., 1927, 295. Cf. also B.P. 283,019; B., 1928, 260).—The modified process, in which an acid instead of an alkaline bath is used for fixing the silk treated with metallic salts, consists in treating the natural silk with stannic chloride solution, freeing it from excess of this solution by squeezing or centrifuging, and then treating it, without previous rinsing, in a fixing bath consisting of monosodium phosphate solution to which acids or acid salts and protective colloids may be added, squeezing, rinsing, and treating with a solution containing soap and sodium silicate. A. J. HALL.

Sizing of dyed yarns. BRIT. CELANESE, LTD. (B.P. 315,428, 13.7.29. U.S., 13.7.28).—Dyed yarns, especially of acetate silk, are sized in a substantially neutral (p_H 7–7.5) glue or gelatin bath at 60–80°, *e.g.*, a solution of gelatin (9 pts.), glycerin (2 pts.), castor oil (1.5 pts.), and sodium benzoate (0.015 pt.) in water (260 pts.) neutralised with sodium carbonate (1.33 pts.). Bleeding and change of shade on sizing are thus avoided. C. HOLLINS.

Treatment [to improve the hot-ironing properties] of [cellulose ester and ether] materials. BRIT. CELANESE, LTD. (B.P. 313,970, 20.6.29. U.S., 20.6.28).—The fibrous materials, particularly cellulose acetate silk, are dyed with logwood black (cf. B.P. 263,222; B., 1927, 186); the fusing point of the dyed material is thereby raised (220° to 240–260° in the case of cellulose acetate silk). A. J. HALL.

Machine for washing, dyeing, or like treatment. M. DASSONVILLE (B.P. 334,730, 26.9.29. Fr., 9.3.29).

Pirn tubes, cop tubes, cheese tubes, bobbins, and like supports for textile threads, yarns, and the like. BRIT. CELANESE, LTD., W. A. DICKIE, and F. C. HALE (B.P. 334,174, 21.5.29).

Mothproofing compounds (B.P. 333,561).—See III. **Colouring of varnishes** (B.P. 309,148).—See VI. **Coloured polyvinyl ester varnishes** (B.P. 334,145).—See XIII.

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

Determination of the proportion of sulphur dioxide converted into trioxide when gas from roasted pyrites is passed over catalysts. J. SANS

and A. EHLINGER (Bull. Soc. chim., 1930, [iv], 47, 759—760).—The gases from the catalyst chamber are dried in passing through phosphorus pentoxide, and then pass over potassium chloride, which absorbs the sulphur trioxide. The residual sulphur dioxide is determined.

C. W. GIBBY.

Extraction of potash from polyhalite. H. H. STORCH (Ind. Eng. Chem., 1930, 22, 934—941).—The highest concentration obtained at 25° by extracting equal parts of polyhalite and water was 5.07% K_2SO_4 after 504 hrs.' contact. This concentration was reached at 100° in 120 hrs., and whilst sufficiently rapid to indicate the value of the process as a means of making a direct fertiliser, the rate of extraction is too slow for its use as a leaching process for the production of potassium sulphate. When heated, vigorous evolution of steam occurs at 298° with the formation of an amorphous mass which sinters at 551°. The optimum temperature for later extraction with water appears to be 430—465°, and an 11.4% solution of potassium sulphate may be obtained. Countercurrent leaching is undesirable as the concentration of potassium sulphate results in the formation of $K_2SO_4 \cdot 5CaSO_4 \cdot H_2O$ which later re-forms polyhalite. On evaporation of calcined polyhalite extracts practically all the calcium sulphate is deposited as syngenite ($CaSO_4 \cdot K_2SO_4 \cdot H_2O$) and the system is essentially that of K_2SO_4 — $MgSO_4$ — H_2O . As mined, polyhalite may contain up to 10% NaCl, 90% of which may be separated by rapid washing with the loss of only about 4% K_2SO_4 .

C. A. KING.

Infusorial earth from Akhaltzikh and floridin from Kutais. A. MARKMAN and M. KOVALENKO (Masloboino Zhir. Delo, 1929, No. 8, 10—13).—Kutais (Caucasus) floridin, an oil clarifier, giving on analysis SiO_2 71.68, $(Fe_2O_3 + Al_2O_3)$ 7.15, Fe_2O_3 4.37, CaO 2.65, MgO 1.52, loss (at 105°) 11.05, (on ignition) 16.10%, is 1.5—2 times more active than infusorial earth as a decoloriser and absorbent for substances containing phosphorus. Optimal conditions were determined. The oil-absorption value of floridin is one half that of infusorial earth.

CHEMICAL ABSTRACTS.

Minimum voltage to reduce aluminium oxide. A. B. NEWMAN and G. G. BROWN (Ind. Eng. Chem., 1930, 22, 995—1000).—With the object of calculating the minimum electrical energy required, under ideal conditions, to electrolyse a saturated solution of aluminium oxide (in cryolite) at 900—1000°, the sp. heat of crystallised alumina has been determined from the diffusion of heat through a cylinder of the powdered material. The minimum voltage is computed to be 0.947 at 950°.

C. A. KING.

Technical manufacture of anhydrous aluminium chloride. C. WURSTER (Z. angew. Chem., 1930, 43, 877—880).—The various technical methods are reviewed historically, with especial regard to the development of a continuous process based on the interaction of carbon monoxide, chlorine, and alumina.

H. F. GILLBE.

Vanadium [pentoxide] catalyst. J. E. ADADUROV and G. K. BORESKOV (J. Appl. Chem., Russia, 1930, 3, 11—20).—A vanadium catalyst, immune against water, arsenious oxide, hydrochloric acid, and hydrofluoric acid, and giving 99% conversion of a gas containing

7% SO_2 and passing at a velocity of 100 c.c. per min. at 400°, the length of the contact layer being 20 cm., is prepared from a mixture of sodium or potassium silicate, vanadate, and stannate solutions. These are treated with small portions of sulphuric acid, heated at 50—70°, and the precipitate is collected, pressed, dried, and heated gradually to the contact temperature.

CHEMICAL ABSTRACTS.

Utilisation of gases obtained in distillation of phosphorus in furnaces of the type of blast furnaces. E. V. BRITZKE, N. E. PESTOV, and N. N. POSTNIKOV (Min. Suir. Tzvet. Met., 1929, 4, 375—387).—The gases, containing phosphorus and phosphine, are divided into two parts after removal of dust: with one, phosphoric acid is formed continuously by passing the gas with superheated steam and a little air through lime at 600—750°; the other is oxidised with air to phosphoric acid in a Cowper stove. The products are superphosphates containing 50% P_2O_5 and combustible gases at 500—600° with a calorific value not below that of blast-furnace gas.

CHEMICAL ABSTRACTS.

Separation of constituents of coal gas. THAU.—See II. **Recovery of lead from lead sulphate waste.** FINK and GREENSPAN.—See X. **Analysis of phosphoric acid.** ISHIBASHI.—See XVI.

PATENTS.

Treatment of gases [for sulphuric acid manufacture]. W. S. ALLEN, Assr. to GEN. CHEM. CO. (U.S.P. 1,743,700, 14.1.30. Appl., 17.11.25).—In the contact process for making sulphuric acid, the burner gases are freed from arsenic by passing them through granulated ilmenite, previously activated by heating with sulphuric acid.

W. J. WRIGHT.

Continuous production of hydrofluoric acid. G. L. SCOTT, Assr. to GEN. CHEM. CO. (U.S.P. 1,748,735, 25.2.30. Appl., 30.6.26).—Fluorspar and sulphuric acid are separately and continuously introduced into a cylindrical rotary furnace, in which they are subjected to thorough agitation to prevent caking, immediate contact with the heated surface of the furnace being prevented by allowing the materials to form a bed. Agitation is conveniently effected by means of iron rails of a length approximately equal to that of the cylinder, these rails tending to ride up the sides as the cylinder rotates, and then falling back with considerable force.

W. J. WRIGHT.

Ammonia-oxidising method. I. HECHENBLEIKNER and N. TITLESTAD, Assrs. to CHEM. CONSTRUCTION CO. (U.S.P. 1,748,646, 25.2.30. Appl., 18.5.27).—Difficulty in regulating the steam supply to stripping towers in which ammonia is recovered from ammonia liquor results in very rich gaseous mixtures for the oxidation process, with high combustion temperatures and consequent burning of the gauzes. External sources of heat may be entirely eliminated by utilising the heat of the exothermal reaction in the converter to separate the gaseous ammonia from the liquor, the process being made automatic by transferring the reaction heat to water, and using this to heat the stripping tower.

W. J. WRIGHT.

Method of concentrating caustic alkalis. R. M. WINTER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P.

332,250, 16.1.29).—The evaporating plant is constructed of, or lined with, copper-nickel alloy (20–50% Ni) if heated with steam, or of nickel-chromium alloy (20–50% Cr) if fired direct, the parts exposed to the alkali being resistant to corrosive action. The alloys should be substantially free from iron. [Stat. ref.]

H. ROYAL-DAWSON.

Production of saturated lime-water. NAAML. VENN. NECKAR WATERREINIGER MAATS., and J. A. HERINGA (B.P. 334,011, 5.7.29).—Water flows uninterruptedly at high velocity from below upwards through a quantity of lime sludge arranged upon a resistant layer of gravel spread over the whole cross-section of the saturator in such a manner that the sludge is wholly penetrated by the water.

H. ROYAL-DAWSON.

Extracting magnesia from dolomite. RHEINISCHE-WESTFÄLISCHE KALKWERKE (B.P. 319,690, 16.9.29. Ger., 26.9.28).—Half-burnt and ground dolomite is treated, before being wind-sifted, with sufficient water, and preferably also with carbon dioxide, to convert the magnesium oxide into the hydroxide or basic carbonate.

H. ROYAL-DAWSON.

Manufacture of sodium nitrate. G. H. GLEASON, Assr. to ANGLO-CHILEAN CONSOLIDATED NITRATE CORP. (B.P. 315,262, 14.6.29. U.S., 10.7.28).—To obtain a product suitable for agricultural purposes, in the form of spherical particles of 10–40-mesh and of 99% concentration, the commercial nitrate is heated at a temperature slightly above its m.p., *e.g.*, at 350°. At this temperature most of the impurities do not melt, and remain undissolved. After filtering, the fused material is conducted to a spray distributor, having nozzles of specified shape and dimensions, and projected into a cooling atmosphere, the spray particles being instantaneously chilled by suitable means, such as an air blast.

W. J. WRIGHT.

Production of potassium nitrate. P. HÖFER, and KALIFORSCHUNGS-ANSTALT GES.M.B.H. (B.P. 332,359, 13.6.29).—A hot solution of the nitrate of a readily hydrolysable metal, *e.g.*, aluminium or iron, is allowed to react with one of potassium chloride and then cooled to separate potassium nitrate. The mother-liquor is concentrated with the evolution of hydrogen chloride, either until nitrous fumes begin to escape, or until most of the chlorine has been expelled from the solution. Nitric acid, potassium chloride, and water are then added to restore the original concentration. The solution is cooled, potassium nitrate separated, and the operation repeated.

H. ROYAL-DAWSON.

Treatment of superphosphate. L. ADELANTADO (B.P. 333,518, 9.5.29).—Superphosphate is made neutral to methyl-orange by means of ammonia, and then lixiviated with water or water acidulated with sulphuric acid, the solution being evaporated to a suitable density for crystallisation. By treating the hot leach liquors with sulphuric acid, all the calcium is precipitated and a solution of orthophosphoric acid is obtained, which may be converted into monoalkali phosphates. Alternatively, these may be produced by treating the leach liquors with alkali sulphates instead of sulphuric acid.

W. J. WRIGHT.

Manufacture of sodium salicylate. M. E. PUTNAM, Assr. to DOW CHEM. CO. (U.S.P. 1,755,362, 22.4.30. Appl., 6.4.23).—Rhombic crystals of sodium salicylate hexahydrate are obtained by neutralising a hot solution of salicylic acid with sodium hydrogen carbonate to obtain a solution which does not deposit the anhydrous salt when cooled to 5–6°. After cooling to this temperature, the solution is seeded with hexahydrate crystals and set aside until 70% of its content of salicylate has crystallised. The mother-liquor is decanted and returned to the neutralising vat. A. R. POWELL.

Production of the cyanamides of the alkaline-earth metals and magnesium. N. CARO and A. R. FRANK, Asses. of STICKSTOFF-WERKE GES. M.B.H. (B.P. 332,468, 1.10.29. Ger., 1.10.28. Addn. to B.P. 279,812 and 281,611; B., 1929, 53, 206).—The reactions described in the prior patents are accelerated by the use as catalysts of silver, or copper, or salts of these metals, which, whilst they do not decompose the ammonia formed, act as catalysts for the water-gas reaction.

H. ROYAL-DAWSON.

Manufacture of sodium aluminate. J. V. N. DORR, G. M. DARBY, A. TERRY, JUN., and H. N. SPICER, Assrs. to DORR CO. (U.S.P. 1,747,759, 18.2.30. Appl., 11.2.25).—The proportions of bauxite to caustic soda should be regulated so that the molecular ratio of Na_2O to Al_2O_3 is about 2 : 1. Digestion is carried out in continuous agitators at atmospheric pressure and at a temperature above 87.7°, and during digestion the product is continuously diluted so as to maintain a sp. gr. of 1.20–1.32, the red mud being separated by sedimentation and decantation.

W. J. WRIGHT.

Manufacture of a base-exchange material. Base-exchange substance. Preparation of metallo-silicates. A. S. BEHRMAN, Assr. to PERMUTIT CO. (U.S.P. 1,756,623–5, 29.4.30. Appl., [A] 24.4.24, [B] 5.12.24, [C] 11.4.25).—(A) A solution of sodium silicate is treated with an excess of dilute sulphuric acid to obtain a colloidal solution of silica which, after cooling to 5°, is treated with a solution of sodium aluminate until the mixture reacts slightly alkaline. The stiff gel which rapidly forms is dried at 80° and wetted again, whereby it breaks up into vitreous granules having an exchange capacity of 250 grains of calcium carbonate per lb. (B) Bauxite, gibbsite, limonite, or other natural or artificial granular aluminium or iron hydroxide is screened through 20-mesh and the dust removed by sieving on 60-mesh. This product is then heated under pressure with a solution of sodium silicate to obtain a zeolite-like substance. (C) In carrying out the reactions described in (A), the two solutions are allowed to flow simultaneously into a small mixing vessel from pipes so proportioned as to give the correct mixture, and the mixture is allowed to fall continuously into a large receiving vessel through a long pipe which ensures thorough mixing in the receiver.

A. R. POWELL.

Improvement of glauconite [for water softening]. H. KRIEGSHEIM and W. VAUGHAN, Assrs. to PERMUTIT CO. (U.S.P. 1,757,373, 6.5.30. Appl., 4.5.25. Renewed 23.10.29).—The crude sand is heated for 30 min. at 50–60° with a 0.4–1% solution of sodium hydroxide to remove humus and disintegrate clayey material. The

sand is then stirred with a cold 1% solution of aluminium sulphate, drained, and agitated with a 2–3% solution of sodium silicate. The treatment removes all substances which impart colour or odour to water and at the same time hardens the sand. A. R. POWELL.

Production of calcined bauxite. F. C. FRANZ, Assr. to ALUMINUM CO. OF AMERICA (U.S.P. 1,756,425, 29.4.30. Appl., 19.6.25).—Bauxite is mixed with powdered coal and a binder, *e.g.*, iron ore or clay, and the mixture is burned on a grate by drawing air downwards through the mass by means of a suction fan. The product consists of porous sintered lumps suitable for the electrothermic production of pure alumina.

A. R. POWELL.

Recovery of copper [hydroxide] from dilute ammoniacal solutions. I. G. FARBERIND. A.-G. (B.P. 313,045, 18.5.29. Ger., 5.6.28).—The dilute cuprammonium solution is treated with sulphuric or hydrochloric acid in amount necessary to neutralise the ammonia contained in the complex compounds, the precipitated hydroxide being collected.

H. ROYAL-DAWSON.

Recovery of copper from spent copper lyes. I. G. FARBERIND. A.-G. (B.P. 310,425, 25.4.29. Ger., 25.4.28).—The lyes, *e.g.*, “precipitating water” obtained in the manufacture of cuprammonium silk, are treated with an acid-resistant adsorbent material such as silicic acid, active carbon, etc. The adsorbed metal is dissolved out by an acid (*e.g.*, sulphuric acid). The adsorbent may be used again after being washed free from excess acid.

H. ROYAL-DAWSON.

Production of iron carbonyl. VEREIN. STAHLWERKE A.-G. (B.P. 319,356, 9.9.29. Ger., 22.9.28).—Finely-divided copper is admixed with the iron to be acted on by carbon monoxide, to accelerate the reaction; similarly, copper oxide or similar compound may be added to iron oxide before reduction is effected.

H. ROYAL-DAWSON.

Production of aluminium sulphate. M. BUCHNER (B.P. 333,835, 20.11.29. Ger., 24.11.28).—Aluminium nitrate is heated with sulphuric acid under reduced pressure with admission of air or steam, the nitric acid being expelled at 300° and recovered. Apparatus of chromium-iron or chromium-iron-nickel alloy is used. To obtain aluminium nitrate free from iron, clay or other aluminiferous material is mixed with a deficiency of nitric acid and set aside for some days at a low temperature; it is then heated to 80–100° at 6–8 atm. and finally to 150–160°. The iron may also be completely removed by treating the aluminium nitrate solution with concentrated nitric acid or nitric acid vapour, whereby aluminium nitrate is precipitated, or by heating the solution to 130° to give it basicity, the basic aluminium nitrate remaining in solution at this temperature.

W. J. WRIGHT.

Manufacture of [finely-divided] bismuth hydroxide. I. G. FARBERIND. A.-G. (B.P. 332,504, 30.11.29. Ger., 3.12.28).—An aqueous solution of a bismuth salt is precipitated by means of an alkali such as ammonia, adherent water is removed from the precipitate by first washing with a solvent which is more volatile but soluble in water (methyl or ethyl alcohol, acetone),

then washing with a second volatile solvent (ether, ethyl chloride, chloroform), and finally evaporating the second solvent. The product is suitable for therapeutical purposes.

H. ROYAL-DAWSON.

Preparation of stannic oxide gels. J. J. ETRIDGE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 333,670, 5.6.29).—An aqueous solution of ammonia is added to an aqueous solution of a stannic salt, the conditions being so controlled that about 95% of the base is added before precipitation begins. The solution is then kept, and stannic hydroxide is precipitated in the form of a hydrogel, which is washed, collected on a filter, and slowly dried. If desired, other catalytic oxides may be combined with the gel by treating it with aqueous solutions of the corresponding salts. W. J. WRIGHT.

Zinc oxide manufacture. E. C. GASKILL, Assr. to ST. JOSEPH LEAD CO. (U.S.P. 1,743,964, 14.1.30. Appl., 17.11.26).—Zinc ore after roasting, sintering, and crushing is mixed with carbonaceous material and introduced into the preheater of a sealed electric-resistance furnace, whence it passes through a second preheater to the electrodes. The zinc vapour leaves the furnace through a lateral pipe leading into a vertical one, and at the point where the former enters the latter just sufficient air is admitted to oxidise the vapour, and this is ignited. The zinc oxide is drawn by means of a fan into collecting bags. An advantage of this process is that the zinc vapour leaving the furnace is free from sulphur oxides, since the absence of oxygen ensures the retention in the furnace of all the sulphur. The ignition of the vapour in a closed pipe instead of in the atmosphere gives a product of uniform average fineness between 0.4 and 0.25 μ .

W. J. WRIGHT.

Purification of zinc chloride. J. L. McCLELLAN, Assr. to NAT. VULCANIZED FIBRE CO. (U.S.P. 1,744,981, 28.1.30. Appl., 13.4.27).—The solution of zinc chloride, preferably of *d* 1.52, is treated at normal temperature with zinc nitrite in sufficient amount to react with all the ammonium chloride present, the mixture being agitated. When the reaction is complete, the temperature of the solution is raised to 82.2°, so as to decompose the ammonium nitrite, and then to the b.p. in order to precipitate iron salts as oxide, which is removed.

W. J. WRIGHT.

Manufacture of white zinc chloride. J. O. BETTERTON, Assr. to AMER. SMELTING & REFINING CO. (U.S.P. 1,747,751, 18.2.30. Appl., 1.9.27).—Small amounts of zinc are removed from lead bullion, after desilvering by the Parkes process, by circulating the molten bullion through a chamber containing chlorine, the temperature being maintained at about 340–400°. The zinc chloride formed is skimmed off, and freed from lead by melting it in presence of excess of zinc at about 370°, and, while fused, is treated with nitre to yield a white product.

W. J. WRIGHT.

Treatment of mixtures of oxy-salts of arsenic, antimony, and tin. M. F. PERKINS, C. W. HANSON, and B. M. O'HARRA, Assrs. to AMER. SMELTING & REFINING CO. (U.S.P. 1,756,007, 22.4.30. Appl., 20.1.27).—The alkali slag produced in the Harris process of lead refining is dissolved in warm water, the insoluble sodium antimonate collected and smelted with carbon

to produce antimony, the lead and copper are removed by agitation with tin, stannous salts, or sodium sulphide, and the purified liquor is electrolysed for the recovery of pure tin. The spent electrolyte is treated with milk of lime to remove arsenic as calcium arsenate, and the filtrate is evaporated for the recovery of sodium hydroxide and chloride for use again in lead refining.

A. R. POWELL.

[Electrolytic] manufacture of [per-]compounds containing oxygen. I. G. FARBERIND. A.-G. (B.P. 313,124; 23.5.29. Ger., 7.6.28. Addn. to B.P. 316,648; B., 1929, 814).—During electrolysis the electrolyte described in the prior patent is maintained saturated with a sparingly soluble mercury compound (such as a sulphate or phosphate).

H. ROYAL-DAWSON.

Cleansing compositions. HENKEL & Co. G.M.B.H. (B.P. 332,530, 22.3.29. Ger., 5.2.29).—The polish, suitable for aluminium utensils etc., consists of a mixture of trisodium or other phosphate and water-glass, with or without a further admixture of known cleansing agents. [Stat. ref.]

H. ROYAL-DAWSON.

Production of phosphates and hydrogen. BAYERISCHE STICKSTOFF-WERKE A.-G. (B.P. 308,684, 18.3.29. Ger., 26.3.28).—Phosphorus, or hydrogen phosphide, and water are caused to react under pressure at temperatures up to 600°, and in the presence of ammonia, alkalis, alkaline-earth metals, bases, or salts, to form phosphates and hydrogen. The solution of phosphates may be sufficiently concentrated that, on squirting from the reaction chamber, it forms a powdery product or it may be crystallised. Catalysts (metals, alloys, or various phosphorus compounds) may be used.

H. ROYAL-DAWSON.

Separation of oxy-salts from alkali mixtures containing same. H. HARRIS (U.S.P. 1,775,676, 16.9.30. Appl., 3.8.25. U.K., 8.8.24).—See B.P. 245,479; B., 1926, 236.

Production of soluble lead reagents. D. W. PARKES, Assr. to H. W. ROBINSON (Re-issue 17,804, 16.9.30, of U.S.P. 1,740,312, 17.12.29).—See B., 1930, 144.

Heat-insulating material (U.S.P. 1,757,470).—See I. **Products from coal** (B.P. 314,859). **Removing sulphur compounds from gases** (B.P. 305,026). **Ammonia from gas** (U.S.P. 1,747,616).—See II. **Welding agent** (B.P. 313,487).—See X. **Urea-calcium nitrate and fertilisers** (B.P. 332,945 and 332,948).—See XVI.

VIII.—GLASS; CERAMICS.

Physiological and chemical experiments with "U.-V." glasses and a method of determining their quality. H. VALENTIN (Pharm. Ztg., 1930, 75, 982—984, 995—998, 1005—1008).—A marked tanning of the skin and a slight improvement in strength were observed in children who had occupied for several months class-rooms having "Ultravit" glass windows. No increase in the hæmoglobin in the blood was noted. The germination period of seeds was considerably shortened under "Ultravit" glass, although the rapidity of growth of the plants was increased only by about

10%. Fungus cultures declined rapidly under "Ultravit" glass. Changes in chemical compounds are largely due to the action of the ultra-violet rays, but in some cases decomposition is caused by the visible rays. A simple method of testing "U.-V." glasses is described, which is based on the action of light in converting potassium nitrate solution into the nitrite. Of the glasses examined, the "Uviolglas" (Schott und Gen.) was the best and cheapest, whilst the glass used in the above biological tests, "Ultravit," was least transparent to ultra-violet rays.

F. SALT.

Determination of the degree of refractoriness of clays by their content of water of constitution.

N. P. CHIZHEVSKI (J. Russ. Met. Soc., 1926, 177—183).—Hygroscopic moisture, removed at 120°, had no effect on the refractory properties of Tomsk, Salair, and Borovitchy clays. The kaolin content (Sabell) and (approximately) the m.p. of clays are directly proportional to the percentage of water of constitution, the removal of which is nearly complete at 900°.

CHEMICAL ABSTRACTS.

Russian porcelain manufacture. I. F. PONOMAREV (Sep., 3 pp.).—Determinations of the limits of solubility at 1300° in fused felspars of the ingredients of the porcelain mass were made. The limit of saturation of the fused mass with kaolin was 14%, with quartz 70%, and with alumina 3.5%. With silica and alumina the limit of saturation occurred when the fused mass had the composition $K_2O, 1.15Al_2O_3, 12SiO_2$. With silica and kaolin no further saturation was observed.

CHEMICAL ABSTRACTS.

PATENTS.

[Roller-type] glass-annealing lehrs [for plate or sheet glass]. R. HADDAN. From SIMPLEX ENG. Co. (B.P. 331,927, 13.4.29).—A Lehr primarily intended to work in conjunction with glass-rolling apparatus is described. The claims cover the provision of a system of rollers for charging and conveying the sheets to and through the furnace, of doors for shutting off parts of the furnace when not required or for cleaning purposes, and of means for handling the annealed sheets at the cold end of the furnace, full details of which are given.

M. PARKIN.

Manufacture of compound glass. SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 316,955, 6.8.29. Fr., 3.8.28).—A sheet of plastic material, e.g., cellulose acetate or nitrocellulose, is superficially softened by immersion in a bath of substantially non-volatile plasticiser, e.g., benzyl alcohol, and while therein is placed between two glass sheets free from air bubbles and the composite sheet is united by gentle pressure. Vacuum may be used to assist removal of air bubbles. Alternatively, one or both sheets may be coated with a solution of the plastic material and the two united in the bath.

M. PARKIN.

Preventing or minimising the obscuration of windows and other transparent surfaces by moisture. H. E. GRIFFITHS (B.P. 332,966, 10.10.29).—A paste is claimed consisting of soap (preferably carbolie) mixed with glycerin, water, and a drying agent.

E. B. HUGHES.

Tunnel or channel dryer for earthenware and the like. H. HAAS (B.P. 314,520, 30.5.29; Ger., 30.6.28. Cf. B.P. 173,234; B., 1922, 969 A).—In a multi-chamber dryer the fan, which circulates the drying air, is situated between the drying chambers at one end and the humidifying or steaming chambers at the other, thus serving to raise the temperature of the ware before drying commences. The tunnel is closed at the inlet and opened only for the intermittent introduction of trucks of green ware.

M. PARKIN.

Manufacture of porous ceramic articles or products. R. LAHAUSOIS (B.P. 311,761, 22.4.29. Belg., 16.5.28).—Porous ceramic articles suitable for insulating purposes, abrasives, filters, etc. are prepared by intimately mixing dry ground porcelain mass with carbon grains of size suited to the degree of porosity required, moistening with water (or alkali, to form with the paste an enamel which coats the cells and promotes cohesion) sufficiently for "dry-moulding." The moulded shapes, set in airtight containers and packed round with a reducing agent (carbon), are then fired first at 1200—1300° in a reducing atmosphere, followed, after being removed and stacked so as to ensure good circulation of air, by a second firing under oxidising conditions at a red heat to burn out the carbon.

M. PARKIN.

Refractory composition and manufacture of articles therefrom. L. J. TROSTEL, Assr. to GEN. REFRACTORIES Co. (U.S.P. 1,752,867, 1.4.30. Appl., 30.3.27).—Either natural mullite or that produced from dumortierite or by calcining cyanite, andalusite, or sillimanite at 1400° forms 2—20% (preferably 10%) of a mixture with 60—70% of crude and 20—30% of calcined diaspore; such mixture, when formed into refractory articles, has a lower firing shrinkage than have mixtures usually employed.

M. PARKIN.

Manufacture of grinding wheels, hones, and the like. L. BRUNELLI, G. CARRA, and F. SOFIA (B.P. 332,524, 21.2.29).—Carbonaceous slag or waste from burnt coal is mixed with emery etc. and borax or powdered silica and baked at 800—1300°.

H. ROYAL-DAWSON.

Glass tank furnaces. HARTFORD-EMPIRE Co., Assees. of P. G. WILLETTS (B.P. 334,799, 27.11.29. U.S., 15.12.28).

Manufacture of laminated glass. L. J. KOLB, Assee. of A. G. WORRALL (B.P. 334,768, 25.10.29. U.S., 30.10.28).

Composition [of plaster and oil] for modelling, sculpture, pottery, etc. J. T. TUSSAUD (B.P. 334,000, 27.6.29).

IX.—BUILDING MATERIALS.

Calcium ferrites and iron cements. V. S. NAGAI and K. ASAOKA (J. Soc. Chem. Ind., Japan, 1930, 33, 312—315 B; cf. B., 1930, 907).—Iron cement contains a large amount of calcium ferrite, but sinters at 1450—1500°, i.e., rather above the corresponding temperature for Portland cement. This is due to the small amount of calcium aluminate and to the lack of eutectic formation.

C. W. GIBBY.

PATENTS.

Manufacture of cementitious material. H. BERRY (B.P. 332,925, 30.4.29).—In the production of cementitious material from a base material (e.g., aluminium silicates) and a binder comprising oxides or hydroxides of calcium or magnesium and sulphuric or sulphurous acid, or salts thereof, the original heating to calcine the raw material (of the binder) and/or a second heating is effected in an atmosphere containing sulphurous or sulphuric acid and, if desired, steam. Thus natural carbonates of calcium or magnesium may be calcined and then dipped into a solution (e.g., 10—15%) of aluminium sulphate acidulated with sulphuric acid, and the product heated at 700° (magnesium compound) or 800° (calcium compound) in an atmosphere of steam and acid vapour, cooled, and incorporated with a base material. Alternatively, blast-furnace slag containing silicates and lime may be heated in an atmosphere of steam and acid vapour at 700—900°.

S. K. TWEEDY.

Manufacture of waterproof concrete. R. CROSS, Assr. to SILICA PRODUCTS Co. (U.S.P. 1,744,869, 28.1.30. Appl., 20.4.25).—A bituminous substance, such as asphalt, is incorporated with a concrete mix of hydraulic cement, mineral aggregate, and a gelatinising clay, the mixture, after setting, being heated at a temperature sufficient to melt the asphalt.

W. J. WRIGHT.

Preparation for waterproofing cement used in buildings. A. STEWART (B.P. 334,053, 20.8.29).—A mixture of soft soap, ammonia, alum, washing soda, lard, boiled linseed oil, and water is boiled, cooled, and mixed with cement and sand.

L. A. COLES.

Hydraulic binding medium [impervious to sea- and hard water]. SOC. ANON. DES CHAUX ET CEMENTS DE LAFARGE ET DU TEIL (B.P. 317,783, 29.7.29. Fr., 22.8.28).—The product is prepared from 80—50 pts. of aluminous cement produced by fusion, clinkering, or fritting and 20—50 pts. of gypsum (raw or calcined at or above 200°) or of anhydrite; alternatively, a crushed mixture of calcium sulphate with the constituents of aluminous cement is calcined.

L. A. COLES.

Manufacture of porous [heat- and sound-]insulating bodies. H. BOHLANDER (B.P. 333,957, 28.5.29).—A mixture of two inorganic fibrous materials, one chosen for its insulating properties (e.g., slag-wool) and the other for its strength (e.g., asbestos), preferably with the fibres set parallel and with or without the addition of kieselguhr, magnesia, etc. and/or binding agents, is worked into a coherent, felt-like mass, e.g., by dipping in an aqueous solution of a binder or by spraying with steam or moist air.

L. A. COLES.

Preparation of asphalt emulsions. R. LICHTENSTERN (B.P. 332,591, 22.4.29).—From 1 to 3% of Trinidad or Bermudez asphalt or montan wax is mixed with bitumen, particularly petroleum asphalt, and a hot solution of caustic alkali is added. The emulsions become miscible with water in all proportions when the proportion of added asphalt or wax is increased up to 25—50%; the stability is further increased by the addition of resins and fatty acids etc.

H. ROYAL-DAWSON.

Manufacture of pavement materials. N. V. KONINKLIJKE STEARINE KAARSENFABR. GOUDA (B.P. 333,937, 23.5.29. Holl., 5.2.29).—Bituminous emulsions are mixed cold with stony material (e.g., basalt lava, blast-furnace slag, limestone) of porosity such that the emulsion adheres strongly to it; the stony material may, if desired, be pretreated with oils, bituminous materials, solvents, or mixtures of these. L. A. COLES.

[Rubber-bitumen] paving blocks. DUNLOP RUBBER CO., LTD., H. C. YOUNG, F. W. WARREN, and F. H. TOOP (B.P. 333,047, 29.7.29).—A paving block has a comparatively resilient rubber upper surface, produced as in B.P. 315,512 (B., 1929, 830); the lower portion of this surface contains a higher proportion of sulphur so as to form a vulcanite layer intermediate between the upper surface and the base. The base is composed of bitumen aggregate which is attached to the vulcanised rubber top by pressing, firmness of attachment being secured by means of undercut grooves and the application of a bond of bitumen or bitumen-rubber adhesive. D. F. TWISS.

Treatment of road-making materials. C. G. FOX (B.P. 332,897, 20.3.29).—The stone aggregate etc. is treated with solutions (e.g., 0.1–3%) containing pyridine or pyridine bases prior to impregnation with bitumen or tar emulsions. Alternatively, the bitumen or tar emulsions may be treated, preferably immediately before use, with solutions containing the bases, and then applied to the road-making material. S. K. TWEEDY.

Drying of lumber. J. P. WILSON, ASSR. to WILSON-OTWELL & CONE, INC. (U.S.P. 1,757,892, 6.5.30. Appl., 6.10.27).—Immediately after sawing, the lumber is immersed in hot water which is kept at the b.p. until the wood is heated uniformly throughout. The wood is then removed and dried artificially in the usual way. A. R. POWELL.

Treatment of wood and other fibrous material. G. E. S. SANNA (B.P. 311,227, 22.4.29. Nor., 7.5.29).—The material is coated or impregnated with a mixture of cyclic carbon compounds with other substances such as formaldehyde and acetone or alcohol, and is then heated at 120–170° and under 10–40 kg./cm.²

H. ROYAL-DAWSON.

Wood-impregnating product. Impregnation of wood. SOC. DE RECHERCHES ET DE PERFECTIONNEMENTS INDUSTRIELS (B.P. 310,804–5, 29.4.29. Fr., 30.4.28).—(A) The product comprises coal tar, creosote, or lignite oil, shale petroleum, etc., or a mixture thereof, having dissolved or combined therein, while hot, 1–5% of an antiseptic salt of a metalloid which improves the silting power of the base (e.g., a compound of antimony or arsenic, such as arsenic sulphide, halogen-containing compounds being excluded). Arsenic or its oxide, or antimony, or free sulphur may also be incorporated in addition. Arsenic sulphide may be produced *in situ* by interaction of sulphur and arsenious oxide. (B) The wood, if desired after preliminary heat treatment, is impregnated first with an antiseptic aqueous solution (A) having great penetrability which contains a solution of arsenic sulphide in ammonium hydrosulphide, and then with an antiseptic liquid the principal antiseptic constituent of which is insoluble in such aqueous solu-

tion. Solution A may also contain a soluble fatty acid salt (e.g., acetate, propionate), and/or an alkaline salt (alkali sulphide), and/or hydrophenolic compounds (wood tars), or a substance (ammonia, soap, or alkali resinate) capable of lowering its surface tension. The arsenic sulphide may be produced *in situ* by interaction of arsenic hydride and alkali sulphide.

S. K. TWEEDY.

Recovery of surplus preservatives in treatment of timber. R. N. RAWSON (U.S.P. 1,756,797, 29.4.30. Appl., 3.6.26).—The timber is immersed in the preservative, e.g., creosote, in a retort which is then evacuated and heated to above the b.p. of water at the internal pressure. The steam thus produced in the pores of the wood drives out the surplus preservative, which is removed from the retort prior to breaking the vacuum.

A. R. POWELL.

Plates, slabs, tiles, etc. for building and sound- or heat-insulating purposes. NAAML. VENN. DE NIEUWE ISOLEER MAATS. "DE NIM" (B.P. 314,354, 25.6.29. Holl., 25.6.28).

Sheets or slabs for building purposes. A. H. J. WRIGHT (B.P. 333,978, 6.6.29). L. A. COLES.

Road surface and its preparation. J. ROBINIUS (B.P. 334,597, 7.6.29).

Preparation of road-surfacing compositions. S. E. FINLEY (B.P. 334,701, 28.8.29).

Heat-insulating material (U.S.P. 1,757,470). **Wet-mixing apparatus** (U.S.P. 1,758,200).—See I.

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

Constitution of the Fe-C-Si system. A. KRÍŽ and F. POBOŘIL (Iron and Steel Inst., Sept., 1930. Advance copy. 20 pp.).—In the iron-carbon-silicon ternary system the eutectoid concentrations up to 6% Si, the maximum solubility curve of carbide in γ -solid solution, and the transformation points on heating have been determined. From these data the constitutional diagrams of the ternary iron-carbon-silicon alloys for 1, 2, 4, and 6% Si have been prepared.

C. J. SMITHELLS.

Mechanism of the dissolution of cementite in carbon steel and the influence of heterogeneity. E. WALLDOW (Iron and Steel Inst., Sept., 1930. Advance copy. 38 pp.).—The transformation of α - into γ -iron with simultaneous dissolution of Fe_3C is found to occur successively in different parts of a specimen during heating. It is suggested that this is due to uneven distribution of silicon which influences the transformation. Two new hardening structures are described, hypotroostite, which occurs in hypoeutectoid steels, and sorbotroostite, which occurs in eutectoid steels. C. J. SMITHELLS.

Physics of the hardening of steel. F. WEVER (Naturwiss., 1930, 18, 452–459).—The influence of the rate of cooling on the thermal transitions of carbon steels has been determined for velocities up to 10,000° per sec., and a space model showing the relationships between temperature, carbon concentration, and cooling velocity has been prepared, which, in conjunction with observations of the fine structure of the metal, supplies a simple

theory of the hardening process according to which martensite is a thermodynamically unstable phase. (H. F. GILLBE.)

Quantitative analysis of steels by spectrum analysis. F. TWYMAN and A. A. FITCH (Iron and Steel Inst., Sept., 1930. Advance copy. 12 pp.).—The method of wedge spectra, using the logarithmic sector, has been found satisfactory for the following determinations, all obtainable from one spectrogram: Si up to 0.8%, Cr up to 4%, Ni up to 5%, Cu up to 1%. The accuracy is 10% of the contained element in the case of silicon, and 5% in the other cases. One spectrogram can be prepared in $\frac{1}{2}$ hr., and measurement of each constituent takes 5 min. (C. J. SMITHELLS.)

Analysis of basic slags and representation of their composition in a triangular diagram. O. QUDRAT (Iron and Steel Inst., Sept., 1930. Advance copy. 9 pp.).—The usual methods for the determination of ferric oxide and of sulphide sulphur in basic slags are criticised and the following methods recommended. The total sulphur and the sulphate sulphur are determined gravimetrically; the difference indicates the content of sulphide sulphur, *a*. Then the sulphide sulphur, *b*, is determined iodometrically in a separate sample, and the bivalent iron contained in the solution of decomposed slag is determined oxidimetrically. The difference *a* — *b* indicates the sulphide sulphur, *c*, lost in the iodometric determination. This is equivalent to the tervalent iron which is reduced when the slag is decomposed. By subtracting this figure from that for the bivalent iron, the accurate figure for bivalent iron is obtained. For rapid iodometric determination of sulphide sulphur the tervalent iron is first reduced with zinc. (C. J. SMITHELLS.)

Reactions between iron sulphide, sulphur dioxide, and iron oxides in the metallurgy of copper. A. C. HALFERDAHL (Ind. Eng. Chem., 1930, 22, 956—963).—Copper calcines usually contain magnetite which is not reduced by ferrous sulphide below 1300°, the maximum temperature of smelting. In actual smelting practice it is proposed to maintain a lower-grade bath of matte in the hottest part of the furnace independently of the customary matte pool. The converter slag would be poured from a height in this hot part so that the slag would plunge into the matte bath and then flow to the other end of the furnace as in present practice. Siliceous flux would be charged to flux the reduced magnetite. In the smelting operation it is considered that the reduction of sulphur dioxide by carbon (coke) is not necessarily a desirable reaction, and the chief point is to maintain a hot focus at the hearth to accelerate reduction of magnetite by the low-grade matte. (C. A. KING.)

Relation between macro- and micro-structure in some non-ferrous alloys. M. L. V. GAYLER (Inst. Metals, Sept., 1930. Advance copy. 12 pp.).—Examination of the micro- and macro-structures of a 5% lead-tin alloy, a 7% copper-aluminium alloy, and an 11% silicon-aluminium alloy cast from various temperatures into different moulds shows that with increase of casting temperature the macrostructure becomes coarser and the microstructure finer. Variation in the ratio of the cross-section of an ingot to that of the mould affects both

structures, but if this ratio is kept constant and the alloy is cast just above the liquidus, casting into steel or graphite moulds has little effect on either structure; with higher casting temperatures the alloy cast in steel has a different macrostructure from, but similar microstructure to, the same alloy cast in graphite. At low casting temperatures the nature of the melting atmosphere does not affect the structure, but at higher temperatures hydrogen refines the macrostructure. Copper-aluminium alloys which have been freed from gas by the nitrogen process, and subsequently melted in a vacuum, still exhibit inverse segregation. The nature of the furnace atmosphere has little effect on the modification of silicon-aluminium alloys, and a normal alloy cannot be modified by the extremely rapid cooling produced by casting it into a heavy, water-cooled, copper mould. (A. R. POWELL.)

Influence of reheating on dilatation and hardness of tempered aluminium-silicon alloys. P. CHEVENARD and A. PORTEVIN (Compt. rend., 1930, 191, 252—254. Cf. Guillet and Ballay, B., 1930, 717).—The alloys, tempered at 525°, were reheated to various temperatures up to 400°, the heating being effected at the rate of 250° per hr., and then maintained at the desired temperature for 8 hrs. The diminution of dilatation at 20°, the increment in length, and the increase in Brinell hardness, for an alloy containing 0.94% Si are plotted against temperature of reheating. The first two curves have a maximum at 275°, and show that between 200° and 350° a reheating for 8 hrs. suffices to precipitate the whole of the silicon, but that above that temperature re-dissolution occurs. The hardness curve shows a maximum at the lower temperature of 175°, due to the fact that though hardness is increased by precipitation of the silicon, it is decreased by the diminished fineness of structure consequent on reheating. It is concluded that silicon as an independent constituent does not explain the contraction resulting from reheating aluminium-copper alloys (cf. B., 1928, 301).

(C. A. SILBERRAD.)

Unusual corrosion of aluminium by alkali. O. W. STOREY (Trans. Amer. Electrochem. Soc., 1930, 58, 43—48).—A peculiar case of pitting of aluminium electric-oven walls was found to be caused by sodium hydroxide formed from the sodium silicate adhesive used in sticking together the corrugated sheets of asbestos which form the insulation. Asbestos millboard coated with sodium silicate so as to make it adhere directly to aluminium does not cause appreciable corrosion of the metal, but when the sodium silicate is on the other side of the millboard and excessive moisture is present the sodium hydroxide formed by hydrolysis diffuses through the asbestos layer and attacks the aluminium. Methods of avoiding this type of corrosion are indicated. (H. J. T. ELLINGHAM.)

Effect of addition of salts and bases on corrosion by hypochlorite. G. N. QUAM (Food Ind., 1930, 2, 121—122).—The corrosive action of hypochlorite solutions on metals is reduced by the addition of sodium phosphate (0.1—0.5%), sodium hydroxide and carbonate (0.5%), or calcium hydroxide (0.1%).

CHEMICAL ABSTRACTS.

Action of boiling barium chloride solutions on metals. V. ZEMLYANITZUIN and P. DOBROVOLSKI (J. Chem. Ind., Russia, 1929, 6, 1047).—The solubility of copper and of lead increases with the duration of immersion and the concentration of the solution. The solubility of iron or aluminium is maximal after 8–16 hrs., a protective coating of hydroxide being formed.

CHEMICAL ABSTRACTS.

Electrolytic recovery of lead from lead sulphate waste. C. G. FINK and L. GREENSPAN (Trans. Amer. Electrochem. Soc., 1930, 58, 69–74).—Electrolysis of a solution containing (per litre) 50 g. of lead sulphate, 200 g. of sodium hydroxide, and a small amount of glue, at 55° with a cathodic current density of 7 amp./ft.² in a diaphragm cell, yielded crystalline but strongly adherent deposits of lead at current efficiencies over 90%. Red lead is simultaneously formed at the anode, but the best yield of this substance was obtained by electrolysis of a solution containing (per litre) 55 g. of lead sulphate and 240 g. of sodium hydroxide at 65–70° with a current density of 400–500 amp./ft.² at a lead-silver alloy anode. The anolyte was moderately stirred and the current efficiency of red lead production reached 23%. Since sodium sulphate can be crystallised from the spent electrolyte and freed from lead compounds by washing with sodium hydroxide solution, it is possible to recover three separate products from this single electrolytic process.

H. J. T. ELLINGHAM.

Town's gas for heat-treatment furnaces. WALTER. —See II. **Determination of Curie points.** REGNER. —See XI.

PATENTS.

Metallurgical furnace. R. D. PIKE (U.S.P. 1,746,904, 11.2.30. Appl., 3.9.27).—High reaction temperatures with the use of oxygen or oxygenated air are attained by inducing combustion downwardly in the central lower part of the furnace within an inverted conical cavity formed by the charge, the walls being protected by feeding additional material as the charge is melted away. The base of the cone is formed by a water-cooled bell and the mixture of fuel and oxygen is injected through a pipe into the bottom of the hollow cone. Damage to the refractory lining is thus minimised.

C. A. KING.

Cupola furnace. T. P. ANTHONY (U.S.P. 1,747,208, 18.2.30. Appl., 27.3.29).—A section of the wall of a cupola is made into an air-regenerative system by allowing some of the furnace gases to pass through the hollow section and around air ducts leading to the tuyères.

C. A. KING.

Iron smelting. R. FRANCHOT, Assr. to FERRO CHEMICALS, INC. (U.S.P. 1,756,349, 29.4.30. Appl., 15.3.28).—About 20–25% of the blast gases is removed from just above the hot zone of the iron blast furnace and utilised for preheating the blast air to 370–480°. The ore charge is so regulated that the ratio $\text{CaO} + \text{MgO} : \text{SiO}_2 + \text{Al}_2\text{O}_3$ does not exceed 8 : 10. A. R. POWELL.

Case-hardening of ferrous articles. P. A. E. ARMSTRONG (U.S.P. 1,748,378, 25.2.30. Appl., 6.10.28. Renewed 31.10.29).—The articles are heated at below 750° in a current of nitrogenous gas produced by the

decomposition of ammonia on the surface of hot carbon activated with a nickel catalyst. A. R. POWELL.

Case-hardening [steel] by nitrogenisation. ELECTRO METALLURGICAL CO., Asses. of A. B. KINZEL (B.P. 314,423, 27.2.29. U.S., 27.6.28).—The articles are heated at below 580° in a molten bath containing a high proportion of alkali cyanide, e.g., in a eutectic mixture of sodium and potassium cyanides. A. R. POWELL.

Nitridation of steel articles. R. SERGESON, Assr. to CENTRAL ALLOY STEEL CORP. (U.S.P. 1,748,623, 25.2.30. Appl., 28.3.29).—A nitride case-hardened layer of relatively large thickness can be obtained by heating alloy steels in ammonia, provided that after working the metal into shape it is annealed at 900°, quenched, and tempered at 650° before nitriding. A. R. POWELL.

[Removal of nitrogen from] steel. M. A. GROSSMANN and D. WILLIAMS, Assrs. to CENTRAL ALLOY STEEL CORP. (U.S.P. 1,748,217, 25.2.30. Appl., 28.7.27).—The steel is treated in the ladle with a small quantity of ferrozirconium containing silicon, calcium fluoride being used as a flux. The nitrogen is converted into zirconium nitride, part of which is removed in the slag.

A. R. POWELL.

Iron-carbon alloy. A. SAUVEUR, Assr. to AMER. ROLLING MILL CO. (U.S.P. 1,745,645, 4.2.30. Appl., 4.6.25).—A steel capable of being quenched or case-hardened contains more than 0.05% C and not more than 0.035% Mn, 0.035% Si, 0.01% P, and 0.03% S.

C. A. KING.

Manufacture of low-carbon ferro-alloys [non-rusting iron]. F. M. BECKET, Assr. to ELECTRO METALLURGICAL CO. (U.S.P. 1,748,750, 25.2.30. Appl., 25.2.27).—An iron-chromium alloy with a relatively high carbon content is blown with pure oxygen until the carbon is reduced to 0.2% and the metal becomes very hot; hydrogen is then blown through the bath until the carbon is reduced to 0.1%.

A. R. POWELL.

Silicothermic metallurgy. [Manufacture of ferro-chromium.] W. C. READ, Assr. to ELECTRO METALLURGICAL CO. (U.S.P. 1,758,465, 13.5.30. Appl., 21.11.28).—A mixture of finely-divided chromite, ferrochrome-silicon, sodium nitrate, and lime is ignited from below, e.g., by charging it on to a bath of molten iron or slag or by means of a primer consisting of a mixture of 4 pts. of dry sodium dichromate and 1 pt. of silicon. The resulting product contains more than 60% Cr, less than 3% Si, less than 0.1% C, and about 0.2% N.

A. R. POWELL.

High-silicon and high-manganese steel. W. C. HAMILTON, Assr. to AMER. STEEL FOUNDRIES (U.S.P. 1,746,586, 11.2.30. Appl., 21.4.27).—A pearlitic manganese steel containing 0.2–0.6% C, 1–2.25% Mn, and 0.5–1.5% Si is claimed.

A. R. POWELL.

[Iron-nickel] magnetic material. G. W. ELMEN, Assr. to BELL TELEPHONE LABS., INC. (U.S.P. 1,757,178, 6.5.30. Appl., 2.4.29).—A magnetic material having a constant permeability above 500 consists of an alloy of iron with 30–70% Ni and up to 12% Cr, Mo, and/or W.

A. R. POWELL.

Non-magnetic material. G. W. ELMEN, Assr. to BELL TELEPHONE LABS., INC. (U.S.P. 1,745,612, 4.2.30.

Appl., 24.11.28).—An iron alloy applicable to the armouring of submarine signalling cable contains 20–30% Ni, 3–4% Mo, and 0.15% Mn. C. A. KING.

[Nickel-iron-silver] alloy. M. E. BARKER, Assr. to H. H. SEMMES (U.S.P. 1,757,508, 6.5.30. Appl., 5.9.28).—An alloy of 37–47% Ni, 45–55% Fe, and 3–13% Ag, preferably 50% Fe, 42% Ni, and 8% Ag, is claimed. A. R. POWELL.

Deoxidation of copper. C. S. SMITH, Assr. to C. R. HAYWARD (U.S.P. 1,755,309, 22.4.30. Appl., 8.7.26).—Molten copper is deoxidised by poling in the usual way until the metal contains 0.12–0.15% O and is then cast into rods or bars. The latter are heated at 750–850° in an atmosphere of hydrogen until the desired reduction in the oxygen content is obtained. The metal is then severely worked by rolling or forging in a non-oxidising atmosphere at 900–950°. A. R. POWELL.

Aluminium alloy. H. C. HALL and T. F. BRADBURY (B.P. 334,430, 24.10.29).—An alloy of aluminium with 0.1–4% Mg, 0.5–2.5% Ni, 0.3–1.5% Fe, 0.5–5% Si, 0–0.5% Ti, 0–0.2% Mn, 0.01–0.2% Cr, and 0–0.5% Cu, to which is added 0.1% Na just before casting, is claimed. A. R. POWELL.

Aluminium-base alloys. ALUMINIUM, LTD., Assees. of R. S. ARCHER and L. W. KEMPF (B.P. 334,656, 5.7.29. U.S., 22.12.28).—Aluminium alloys suitable for the manufacture of pistons for internal-combustion engines contain 7–15% Si, 0.2–3% Mg, 0.5–7% Ni, and 0.3–7% Cu. The preferred composition comprises aluminium alloyed with 14% Si, 2% Ni, 1% Mg, and 0.75% Cu; the alloy is aged at 125–150° for 25 hrs. after quenching from 530°. A. R. POWELL.

Production of tarnish-resisting silver and silver plate. W. S. MURRAY, Assr. to ONEIDA COMMUNITY, LTD. (U.S.P. 1,758,293, 13.5.30. Appl., 19.9.24. Renewed 3.10.29).—Silver or silver plate is immersed in a cold solution containing 20 g. of iodine and 40 g. of potassium iodide per litre or heated at 175° in an atmosphere containing iodine vapour, whereby it becomes coated with a tarnish-resistant film of iodide which may be burnished and polished. A. R. POWELL.

Welding agent for magnesium alloys. I. G. FARBERIND, A.-G. (B.P. 313,487, 25.4.29. Ger., 12.6.28).—The flux consists of a mixture of halides, the cations of which are lithium and potassium, and the anions either chlorine and fluorine or bromine and fluorine. The mixture may be employed in the dry state, as a paste, or, preferably, as a saturated solution. H. ROYAL-DAWSON.

Production of tinplate. RASSELSTEINER EISENWERKS-GES. A.-G., and H. FRÄMBS (B.P. 310,061, 20.4.29. Ger., 20.4.28).—The annealing treatment of the plates consists in heating them either singly or in small packages to about 900°. H. ROYAL-DAWSON.

Manufacture of bearing metals. R. J. SHOE-MAKER, Assr. to S. & T. METAL Co. (U.S.P. 1,745,720–1, 4.2.30. Appl., [A] 29.4.27, [B] 16.9.27. Renewed, [B] 8.11.29).—A bearing-metal alloy comprises lead with (A) up to 1% Na, 0.1–0.25% Ca, and 0.02–0.1% Al; with or without 1–5% Sn; or (B) 0.01–0.05% Li,

0.2–0.8% Na, 0.1–0.7% Ca, 1–2% Sn, and 0.02–0.1% Al. A. R. POWELL.

[Inhibitor for use in] pickling of metals. BARRETT Co., Assees. of P. J. COLE and C. BANTA (B.P. 334,418, 11.10.29. U.S., 31.10.28).—About 0.02% of a naphtho-nitrile or other aromatic nitrile is added to a sulphuric acid pickling bath. A. R. POWELL.

Chemical reagents. [Inhibitors for iron-pickling baths.] F. H. RHODES (U.S.P. 1,746,676–1,746,680, 11.2.30. Appl., 18.7.29).—The inhibitor consists of (A) a monoalkyl-substituted quinoline; (B) a monosubstituted acridine derivative; (C) an amino-derivative of acridine, e.g., 3:6-dimethyl-2:7-diamino-acridine; (D) dinaphthacridine or a similarly constituted derivative of acridine; or (E) diquinolyl or a derivative thereof. A. R. POWELL.

[Inhibitor for baths used in the] pickling and cleaning of metals. P. I. MURRILL, Assr. to R. T. VANDERBILT Co. (U.S.P. 1,748,494, 25.2.30. Appl., 2.5.29).—The inhibitor comprises a compound having the general formula $C_nH_{2n}(NRX)_2$, where X is an acid radical and RN is an organic base, e.g., an alkylene-dipyridonium salt. A. R. POWELL.

Degreasing of metal articles. A. K. CROAD, From UDYLITE GES.M.B.H. (B.P. 308,363, 22.3.29).—Turkey-red oils or alkyl-substituted aromatic sulphonic acids or their salts are added as wetting agents to the usual aqueous solutions of alkaline substances employed for cleaning articles to be electroplated. H. ROYAL-DAWSON.

Production of a phosphate coating on metal. M. GREEN and V. M. DARSEY, Assrs. to PARKER RUST-PROOF Co. (U.S.P. 1,755,391, 22.4.30. Appl., 29.4.29).—The articles are dipped for a short time in a freshly prepared bath before being transferred to the main parkerising bath. A. R. POWELL.

Manufacture of chromium-coated wire. H. K. RICHARDSON, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,745,912, 4.2.30. Appl., 3.5.23. Renewed 8.9.26).—Nickel-steel wire is cleaned by making it first the cathode in a bath of alkali hydroxide or carbonate, and then, after washing, the anode in a dilute sulphuric acid bath until it becomes passive. The wire is then chromium-plated by passing it continuously through the usual chromic acid bath. A. R. POWELL.

Separation of comminuted matter (U.S.P. 1,758,422).—See I. **Calcined bauxite** (U.S.P. 1,756,425). **Copper from spent lyes** (B.P. 310,425). **White zinc chloride** (U.S.P. 1,747,751). **Treatment of mixed oxy-salts** (U.S.P. 1,756,007).—See VII. **Sheets for building purposes** (B.P. 333,978).—See IX. **Nickel anode** (U.S.P. 1,757,714). **Arc-welding and cutting** (U.S.P. 1,746,081 *et seq.*).—See XI. **Film-forming element** (U.S.P. 1,746,751). **Reproducing designs in metal** (B.P. 331,856).—See XXI.

XI.—ELECTROTECHNICS.

Materials for dry cells. I, II. S. MAKINO (J. Soc. Chem. Ind., Japan, 1930, 33, 326).—I. Neither the *E.M.F.* nor the capacity of a dry cell is influenced by the carbon content of the particular kind of carbon used.

They are but little influenced by the difference in resistance between natural and artificial graphite.

II. The capacity of a cell is definitely related to the grain size of the manganese dioxide and graphite, and to the rate of discharge. The electrode distance must be changed according to the rate of discharge.

C. W. GIBBY.

Hydrolysis in standard cells. G. A. HULETT (Trans. Amer. Electrochem. Soc., 1930, 58, 13—18).—The *E.M.F.* of two standard cells, made up in 1904, decreased by over 100 microvolts during the first 2 years, but subsequent changes have not exceeded 60 microvolts, and the values in 1928 were not more than 20 microvolts from those in 1906. A cell made up in 1906 with mercurous sulphate which had been washed with "equilibrium water" (water which has been shaken with mercurous sulphate and mercury at 25°), and with equilibrium water saturated with cadmium sulphate crystals, and the cell filled with this latter solution, has kept the most constant *E.M.F.* of any, the change in 23 years being only 5 microvolts. Other recent work in the author's laboratory (A., 1929, 1147, 1392) is discussed. The marked changes in *E.M.F.* of cells made up with mercurous sulphate in relatively large crystals is ascribed to the formation of a crust of basic sulphate on the crystals when the soluble products of hydrolysis diffuse away. The upper layers of the mercurous sulphate will be affected first and the *E.M.F.* will not alter until this change has penetrated to the layer in contact with the mercury. The behaviour of cells with mercurous sulphate layers of different thicknesses is in harmony with this explanation.

H. J. T. ELLINGHAM.

Magnetometric determination of the Curie points. A. REGNER (Iron and Steel Inst., Sept., 1930. Advance copy. 22 pp.).—Three simple devices for the determination of the Curie points are described. With a low-frequency magnetometer, a resistance furnace fed by alternating current is used for heating, as well as for generating an alternating magnetic field. The *E.M.F.* induced in a secondary winding, of which the specimen forms the core, is applied to the grid of a thermionic valve, and changes in the permeability of the specimen are observed. A high-frequency magnetometer, based on the resonance principle, has the furnace coil and specimen as part of a receiving condenser circuit. Another method employs the change in amplitude of the anode current of a valve-generator according to the conditions in the oscillating circuit, of which the furnace and specimen form a part. With these devices A2 temperatures are shown by sharply-reversible changes, the existence of two cementite changes at 60° and 210° is confirmed, and steels containing 0.4—0.9% C are shown to lose their magnetism between A1 and A3.

C. J. SMITHELLS.

Smoke prevention. GIBBS. **Dew point of flue gases.** JOHNSTONE.—See I. **Reduction of alumina.** NEWMAN and BROWN.—See VII. **Corrosion of aluminium by alkali.** STOREY. **Electrolytic recovery of lead from lead sulphate waste.** FINK and GREENSPAN.—See X. **Purification of water.** BARTOW and JEBENS.—See XXIII.

PATENTS.

Electrochemical electric current rectifiers. H. ANDRÉ (B.P. 334,093, 4.10.29. Fr., 5.10.28).—An anode of porous silver sulphide impregnated with regenerating material, *e.g.*, sulphuric acid, and a cathode of oxidisable metal are arranged in contact within an evacuated, hermetically-sealed vessel. A method of manufacturing the anode is claimed. J. S. G. THOMAS.

Nickel anode. G. B. HOGABOOM, Assr. to HANSON-VAN WINKLE-MUNNING Co. (U.S.P. 1,757,714, 6.5.30. Appl., 28.2.29).—The anode contains more than 98% Ni, more than 0.1% Si, and 0.1% Mn, and is substantially free from carbon. A. R. POWELL.

Electric arc-welding and cutting. (A) H. M. HOBART, (B) P. K. DEVERS, (C) I. LANGMUIR and P. P. ALEXANDER, (D, F, G) E. THOMSON, (E) E. THOMSON and P. P. ALEXANDER, and (H—K) P. P. ALEXANDER, Assrs. to GENERAL ELECTRIC Co. (U.S.P. 1,746,081, 1,746,191, 1,746,196, 1,746,202—5, 1,746,207—9, and 1,746,210, 4.2.30. Appl., [A] 29.6.26, [B] 25.6.26, [C] 5.9.25, [D, F] 26.8.25, [E] 6.10.25, [F] 18.7.29, [G] 3.9.29, [H] 26.12.24, [J] 7.10.25, [K] 10.1.27. Renewed [I] 8.10.27, [K] 6.1.30).—The arc produced between the welding rod and the work is surrounded by a non-oxidising atmosphere comprising (A) helium; (B) argon, diluted or not with hydrogen or nitrogen; (C) nitrogen containing about 6% H₂ to prevent formation of nitrides; (D) a mixture of carbon monoxide and hydrogen, *e.g.*, water-gas; (E) the vapour of ethyl or methyl alcohol, which dissociates into a mixture of carbon monoxide and hydrogen; (G) a mixture of carbon dioxide and propane or other gaseous hydrocarbon to form water-gas; (H) hydrogen; (I) coal gas or natural gas (this is particularly adapted for blowing away the molten metal during cutting operations with the arc); (J) ammonia or a volatile organic amine which dissociates into nitrogen and hydrogen in the arc; or (K) the vapour of a hydrocarbon when a carburised weld is required. (F) The gas is admitted into the arc through a valve automatically regulated by the voltage of the arc. A. R. POWELL.

Indirectly-heated cathodes for vacuum tubes. ARCTURUS RADIO TUBE Co., Assees. of S. RUBEN (B.P. 306,832, 25.2.29. U.S., 25.2.28).—A metal filament, *e.g.*, of tantalum, coated with oxide produced by heating, is coated with material of high dielectric strength, *e.g.*, silica. J. S. G. THOMAS.

[Wood-pulp filling for] primary and secondary electric batteries. M. HOLBROOK (B.P. 314,010, 27.2.30).

Thermoelectronic rectifier. S. LOEWE (B.P. 308,311, 21.3.29. Ger., 21.3.28).

Thermo-electric batteries. J. PETRÍK (B.P. 313,602, 30.5.29. Czechoslov., 15.6.28).

Electric-discharge tubes. S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 334,519, 3.5.29).

Cathode-ray tubes. ASSOCIATED TELEPHONE & TELEGRAPH Co. (B.P. 313,882, 11.6.29. U.S., 18.6.28).

Röntgen tubes. F. ROTHER (B.P. 334,149, 24.12.29).

Röntgen-ray apparatus. SIEMENS-REINIGER-VEIFA GES. F. MEDEZIN. TECH.M.B.H. (B.P. 334,818, 2.1.30. Ger., 23.2.29).

Acetylene (B.P. 332,917).—See II. Photochemical polymerisation (U.S.P. 1,746,168).—See III. Zinc oxide (U.S.P. 1,743,964). Treatment of mixed oxy-salts (U.S.P. 1,756,007). Per-compounds (B.P. 313,124).—See VII. Magnetic material (U.S.P. 1,757,178). Non-magnetic material (U.S.P. 1,745,612). Degreasing metal articles (B.P. 308,363). Chromium-coated wire (U.S.P. 1,745,912).—See X. Articles from thermoplastic materials (B.P. 333,879).—See XIV. Surgical pads (B.P. 333,980).—See XX. X-Ray screens (B.P. 318,152). Reproducing designs in metal (B.P. 331,856).—See XXI.

XII.—FATS; OILS; WAXES.

Examination of soaps containing salicylic acid for free salicylic acid. E. SCHLENKER (Chem. Umschau, 1930, 37, 262–263).—A salve prepared from potash soap, vaseline oil, and salicylic acid was examined by a modified extraction method, and the total absence of free salicylic acid was confirmed, double decomposition to sodium salicylate and free fatty acid having occurred. A special extraction procedure was necessary, as by the usual methods unsaponifiable matter and potassium salicylate were carried over into the clear ethereal extract in the presence of soap and vaseline; with soda soap troublesome emulsions were formed from which extracts free from sodium salicylate were unobtainable.

E. LEWKOWITSCH.

Lipeometer fat test for determining fat and oils in fat-bearing materials, press cake, or refuse. R. SCHWARZ (Oil & Fat Ind., 1930, 7, 335–336, 347).—The variation of sp. gr. of solutions of fats in *o*-dichlorobenzene with the amount of fat present is practically linear, hence the fat content of seeds, press cakes, etc. may be determined rapidly as follows. The material (100 g., ground to 60–80-mesh) is stirred with 600 g. of the solvent (in an aluminium beaker) at ordinary temperatures, and after 5 min. the solution is filtered under suction and its sp. gr. measured with a hydrometer ("lipeometer"), the scale of which is graduated to read directly in percentages of oil; suitable temperature correction is made from a previously-prepared scale. The results by this method agreed well with those from the standard A.O.C.S. determination (mean error 0.09%).

E. LEWKOWITSCH.

[Separation and determination of] solid fatty acids. W. F. BAUGHMAN and G. S. JAMIESON (Oil & Fat Ind., 1930, 7, 331–332).—A method is described by which the lead salt-alcohol separation may be applied to the oil sample without preliminary isolation of the total fatty acids as required by the Twitchell process (B., 1921, 817 A). Not more than 6 g. of oil (containing 1–1.5 g. of solid acids) are saponified with 40 c.c. of alcoholic potash (40 g. per litre of 95% alcohol), the excess alkali is neutralised with glacial acetic acid (one drop in excess being added), and the solution is made up to 150 c.c. with 95% alcohol, then heated to boiling, and hot lead acetate solution (5 g. per 50 c.c. of alcohol; the excess of lead salt is required in the presence of potassium soaps and potassium acetate) is run in. The mixture is allowed to cool slowly and is kept overnight at 15°. The precipitated lead soaps

are collected, washed with cold 95% alcohol until the washings remain clear on dilution with water, and redissolved by boiling with about 100 c.c. of alcohol and 0.5 c.c. of glacial acetic acid. The lead salts obtained by cooling, filtering, and washing as before are heated with hydrochloric acid to recover the solid fatty acids, which are dried at 110° (in a stream of carbon dioxide if isooleic acid is present). Results in good agreement with those from the Twitchell method are obtained, isooleic acid being separated quantitatively in both cases.

E. LEWKOWITSCH.

Determination of unsaturation of fats and fatty acids. III. Wijs iodine value. J. VAN LOON (Chem. Umschau, 1930, 37, 257–262; cf. B., 1930, 724).—Contrary to the experiences with fresh oil, a linseed oil that had been stored some time showed a Wijs iodine value that gradually increased with the time of reaction (cf. Schmidt-Nielsen and Owe, "Bestimmung der Jodzahl," Oslo, 1923); this was shown to be due to gradual depolymerisation of the old oil and not to substitution of halogen by the reagent, which with fresh oils, fatty acids, and esters (in the absence of conjugated acids) gives constant and accurate figures, even with great excess of reagent and prolonged reaction time. For the most accurate discriminatory purposes it is best to perform the Wijs test on the total fatty acids previously freed from unsaponifiable matter, as the latter (e.g., of parsley-seed oil) may contain appreciable amounts of highly unsaturated substances in which substitution may also occur.

E. LEWKOWITSCH.

Determination of iodine values. S. YUSHKEVICH (Masloboino Zhir. Delo, 1930, No. 2, 9–16).—A comparative study. For rapidity and cheapness Kaufmann's method is preferred.

CHEMICAL ABSTRACTS.

Composition of the saturated fatty acids of Japanese sardine oil. H. IKUTA and S. UENO (J. Soc. Chem. Ind., Japan, 1930, 33, 245–246 B).—The solid fatty acids (24–26%; m.p. 52–52.5°, iodine value 24.5, neutralisation value 207.3) of a sardine oil (iodine value 179.1, thiocyanogen value 90.2, saponif. value 193.8, n_D^{20} 1.4801, m.p. of mixed fatty acids 35–35.6°) were examined by fractional distillation of the methyl esters. Palmitic acid (the principal constituent), myristic and stearic acids were isolated, and the presence of butyric (?), arachidic, and behenic acids, and of an acid, $C_{24}H_{48}O_2$, was recognised.

E. LEWKOWITSCH.

New hydrocarbons produced during hydrogenation of fish oils. S. UENO (J. Soc. Chem. Ind., Japan, 1930, 33, 264–266 B).—Volatile products obtained during the hydrogenation of fish oils had saponif. value 131.0, iodine value 25, acid value 95.0. Examination of the unsaponifiable fraction of these products showed it to consist mainly of hydrocarbons (probably isoparaffins). Fractional distillation of the unsaponifiable portion insoluble in methyl alcohol gave mixtures of hydrocarbons, acetyl value 0, yielding no ether-insoluble polybromides. Hydrocarbons with 1–2 double linkings are also present. The hydrocarbons $C_{13}H_{28}$, $C_{16}H_{34}$, $C_{17}H_{36}$, $C_{18}H_{38}$, $C_{19}H_{40}$, $C_{20}H_{42}$ were isolated, and $C_{10}H_{22}$ and $C_{11}H_{24}$ may have been present (cf. occurrence of pristane in shark-liver oil). The greater part of the hydrocarbons in the products examined

appear to have been produced during the hydrogenation.

E. LEWKOWITSCH.

Semi-drying oils and their oxidation in presence of different catalysts. V. CHELINCHEN (Masloboino Zhir. Delo, 1929, No. 5, 49–52).—The semi-drying oils contain little or no linolenic acid or its isomerides, 10–20% of saturated acid, and 73–85% of linoleic and oleic acids (1.5–2.2:1). The drying oils contain 23–41% of linoleic acid, or, if only 14% is present, a high percentage (62–74%) of linolenic acid is present; the percentage of oleic acid varies widely, and that of the saturated acids varies from 4.5 to 9%. Semi-drying oils could be made to dry by partial oxidation in presence of catalysts (lead oxide, cobalt oxide, manganese dioxide, but not other metallic oxides, sulphur monochloride, dimethylaniline, or charcoal).

CHEMICAL ABSTRACTS.

Application of the thiocyanogen value. IV. **Analysis of oils containing linolenic acid.** I. **Composition of chrysalis oil.** W. KIMURA (J. Soc. Chem. Ind., Japan, 1930, 33, 262–264B).—Kaufmann's work (cf. B., 1929, 401), showing that linolenic acid absorbs thiocyanogen at two double linkings only, is confirmed. A chrysalis oil, having iodine value 141.8 (Wijs), thiocyanogen value 93.0, total fatty acids 93.1%, solid fatty acids 23.89%, unsaponifiable matter 0.99%, has been analysed by the thiocyanometric method, which is considered less susceptible to experimental error than the bromination method. The liquid fatty acids are determined as oleic acid 29.2, linoleic acid 35.9, and linolenic acid 34.9% (cf. 29.8%, 48.9%, 21.3%, respectively, determined by bromination; B., 1929, 607).

E. LEWKOWITSCH.

Colorimetric determination of minute quantities of dissolved phosphorus in oils. H. WAEL (Dansk Tidsskr. Farm., 1930, 4, 197–208).—Stich's method (B., 1927, 851) is examined critically; it is necessary to control the concentration of phosphorus, oil, and reagents so that the silver phosphides produced do not coagulate, and the amount of silver nitrate solution added to the standard and unknown samples must be the same. In the modified test proposed, accurate results are obtained when the phosphorus content of the oil does not exceed 1 in 10,000; if more is present (determined by an approximate preliminary test) the sample should be suitably diluted with almond oil before the final determination, for which standard solutions are prepared containing 0.1, 0.09, 0.081, 0.073, 0.066, 0.059, and 0.053 mg. of phosphorus in 1 g. of oil made up to 10 c.c. with a mixture of ether, alcohol, and acetone (40:20:5 by vol.); then 1 g. of the (diluted) sample is diluted with this mixed solvent to 10 c.c. and 0.2 c.c. of a solution of silver nitrate in acetone (0.25 g./100 c.c.) is added to each solution, the colour developed being compared after 15 min.

E. LEWKOWITSCH.

Extinction of ethylene dichloride flames. JONES and KENNEDY.—See II. **Use of soya-bean oil in paints.** BELYAEV.—See XIII. **Fat content of Gruyère cheese.** SAJOUS.—See XIX. **Castor oil soaps in spirituous preparations.** MEYER.—See XX.

PATENTS.

Manufacture of soaps containing fat solvents. S. ZIMMERMANN (B.P. 334,177, 25.2.29. Austr., 2.10.28).—A small quantity of *n*-propyl or isopropyl alcohol is added, before, during, or after saponification, in order to facilitate the incorporation of large amounts of fat solvents in acid soaps.

E. LEWKOWITSCH.

Manufacture of powdered soap. A. WELTER (B.P. 332,590 and 332,599, 22.4.29).—(A) Electrolytes (*e.g.*, salt) are added to the liquid soap in order to cause salting out during the subsequent heating with open compressed steam or direct fire *etc.* to above 100° (4–10 atm.); the heated, grained-out soap is settled for a short time and atomised. (B) If compressed air (*e.g.*, at 10 atm.) is pumped into the pressure vessel containing the liquid soap, before or during the first stage of heating the soap and prior to atomisation, the use of hot air to dry the powder produced is rendered unnecessary.

E. LEWKOWITSCH.

Extraction of fat or other matters from animal carcasses, slaughterhouse refuse, *etc.* A. SOMMERMEYER (B.P. 310,542, 26.4.29).—The mixture of liquid and solid products from a continuous digester is separated continuously by a mesh screening device while being subjected to pressure in a digester. Hydrolysis of the glue-yielding constituents is thus greatly reduced.

E. B. HUGHES.

Decolorising fatty substances with adsorbents. P. W. PRUTZMAN (U.S.P. 1,745,952, 4.2.30. Appl., 20.4.27).—The oils *etc.* are decolorised by heating, out of contact with air, with a powdered magnesium silicate such as that described in U.S.P. 1,598,254 (B., 1926, 937), at temperatures rising from 105° to above 123°; the oil is cooled to the lower temperature before filtering.

E. LEWKOWITSCH.

Treatment of linseed oil. B. H. THURMAN, Assr. to GOLD DUST CORP. (U.S.P. 1,745,877, 4.2.30. Appl., 29.7.26).—Drying and semi-drying oils are polymerised by heating to 260–315° under a pressure of 2 in. Hg, or less, while a stream of non-oxidising gas or steam is passed through the mass; traces of metallic catalysts, *e.g.*, 0.01–0.1% of lead, may be used to accelerate the process, which yields, within 6 hrs., highly bodied pale oils of low acid value.

E. LEWKOWITSCH.

Production of edible oils from crude cottonseed oil. W. KELLEY, Assr. to FILTROL CO. OF CALIFORNIA (U.S.P. 1,747,675, 18.2.30. Appl., 19.7.26).—The crude oil is agitated with 5–10% of water at about 47°, in order to coagulate colloidal matter which is removed with the water; the oil is then stirred at 100–105° with an acid-activated absorbent clay ("Filtrol") and, after filtering, is heated to about 233° at a pressure below 60 mm., while a current of superheated steam is passed through it to assist the removal of free fatty acids.

E. LEWKOWITSCH.

Manufacture of linoleum cement and linoleum. ARMSTRONG CORK CO., Assees. of E. CLAXTON (B.P. 305,656, 22.1.29. U.S., 9.2.28).—An anticoagulant, *e.g.*, rosin, resin acid or ester (1 pt., which furnishes the resinous constituent of the cement), is added to the drying oil (3 pts.) in order to delay coagulation when the mixture

is heated and oxidised at 77—195° in the usual apparatus until the product contains not more than 7% of matter extractable by light petroleum (*i.e.*, unoxidised oil), in addition to the anticoagulant. The oil may then be thickened to the desired consistency by continued heat treatment, with or without continued oxidation treatment. [Stat. ref.] E. LEWKOWITSCH.

Disintegration of cellular matter (U.S.P. 1,746,731).—See I. **Lubricants** (U.S.P. 1,758,446 and 1,758,598).—See II. **Wetting agents etc.** (B.P. 310,941). **Colouring of oils, fats, waxes** (B.P. 309,148).—See XIII. **Esters of *Hydnocarpus* fatty acids** (B.P. 311,236).—See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Special primers for house paints. F. C. SCHMUTZ, F. C. PALMER, and W. W. KITTELBERGER (Ind. Eng. Chem., 1930, 22, 855—860).—An accelerated test cabinet, representing wet exterior wall conditions, was devised for testing the relative durability of various paint systems. A series of trials is detailed and illustrated. The incorporation of varnish, *e.g.*, spar varnish, or synthetic resin varnish, into priming coats gives improved adhesion to wood, regardless of the type of pigment used. S. S. WOOLF.

Effect of priming-coat reduction and special primers on paint service on different woods. F. L. BROWNE (Ind. Eng. Chem., 1930, 22, 847—854).—The durability of paint systems on various soft woods is influenced considerably by the different adhesion of the priming coat to the dense summer wood and the lighter spring wood, respectively. A series of exposure tests of standard paints on woods that had given evidence of poor adhesion for paint in an earlier series of exposures is detailed. The function of the primer was studied by variation in the proportion of linseed oil and turpentine used in the priming coat, use of thinners other than turpentine, *e.g.*, benzene, reputed to afford better penetration of soft resinous wood, and the use of special primers containing red lead, zinc dust, aluminium powder, etc. The results of these exposures (lasting over 4 years) indicate that the flaking of paint from summer wood cannot be prevented by any of the means adopted in this series of trials, and the problem of adhesion is considered to be a fundamental one remaining unsolved. S. S. WOOLF.

Use of soya-bean oil in paints. N. BELYAEV (Masloboino Zhir. Delo, 1929, No. 6, 15—16).—The oil cannot substitute linseed or hempseed oil.

CHEMICAL ABSTRACTS.

Adhesion-tension cell in paint investigations. E. L. McMILLEN (Ind. Eng. Chem., 1930, 22, 890—893).—The accuracy of "wettability" measurements with the Bartell-Osterhof cell (B., 1928, 1) is improved by using more accurate surface tension values, constant temperature, and more densely packed pigment cake (by means of smaller pigment additions and higher pressures, *viz.*, 535 atm.). Results obtained with lithopone and various liquids, under these improved conditions, are of the same order as those previously obtained without the refinement of method (*cf.* B., 1930, 110). Confirmation was thus obtained of the fact that, contrary to accepted

views, the best flow characteristics are obtained with liquids of low wettability. The theory of wetting and of the adhesion-tension cell is discussed with reference to paint problems. It cannot yet be stated whether plasticity observed in mixtures of pigments with good wetting liquids is due to adsorbed liquid layers or to flocculation. S. S. WOOLF.

Wetting of pigments and other powders. W. D. HARKINS and R. DAHLSTROM (Ind. Eng. Chem., 1930, 22, 897—902).—The total energy of immersion of powdered crystalline titanite, stannic, and zinc oxides in various liquids was determined in a calorimeter, details of which are given. The values are higher for polar than for non-polar liquids. The data indicate that nearly all the energy is liberated in the adsorption of a unimolecular film from the liquid on the surface of the powder; thus very small quantities of impurities in the liquids often have a great effect on the wetting of powders. The orientation of molecules and the general energy exchanges at solid-liquid interfaces are also discussed. S. S. WOOLF.

Secondary esters and their use in lacquers. J. G. PARK and M. B. HOPKINS (Ind. Eng. Chem., 1930, 22, 826—830).—The properties of *sec.*-butyl-, amyl-, and -hexyl acetates, *e.g.*, solvent power (dilution ratio etc.), water-solubility, evaporation rate, are tabulated. From observation of stability and resistance to blushing of lacquers containing them etc., these products are considered satisfactory for use in base lacquers or thinners. S. S. WOOLF.

Properties of shellac films. I. Resistance of shellac films from various varnishes to action of water and chemicals. M. VENUGOPALAN and M. RANGASWAMI (Ind. Eng. Chem., 1930, 22, 911—913).—The resistance to water, brine, acid, alkalis, alcohol, sulphur dioxide, and ammonia of shellac films deposited from solutions in methyl, ethyl, and propyl alcohols and from optimum mixtures containing these (*cf.* B., 1930, 110) was observed. The films from varnishes containing mixed solvents were, in general, less affected than those from single-solvent varnishes. Mixtures of methyl or ethyl alcohol with acetone are best adapted for shellac varnish making. S. S. WOOLF.

Resin formation in benzoles. HOFFERT and CLAXTON.—See II. **Viscosity etc. of acetylcellulose jelly.** ARAKI and KUSAGAWA.—See V.

PATENTS.

Manufacture of aqueous coating compositions. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 333,570, 13.3.29).—Water is emulsified with at least twice its quantity of an anhydrous liquid cellulose lacquer which contains only constituents practically insoluble in water and in which chiefly solvents boiling below 150° are used. Cheapness, relative freedom from objectionable odours, and non-inflammability are claimed. S. S. WOOLF.

Dyeing or colouring of varnishes, waxes, fats, oils, and higher fatty acids. I. G. FARBENIND. A.-G. (B.P. 309,148, 5.4.29. Ger., 5.4.28).—These materials are coloured by dissolving in the liquid or molten substance a non-sulphonated, non-carboxylated mordant

dye and treating the solution (or mixture during dissolution), in absence of water, with a heavy-metal salt of an organic acid, preferably a resin acid, higher fatty acid, or naphthenic acid. *E.g.*, a solution of 4-chloro-*o*-aminophenol \rightarrow 2:3-hydroxynaphthoic anilide in warm cellulose laurate varnish is treated with cobalt naphthenate (red-violet); 4-nitro-*o*-aminophenol \rightarrow phenylmethylpyrazolone in warm linseed oil with cobalt stearate (yellow-brown); 3:5-dichloro-*o*-aminophenol \rightarrow β -naphthol in molten carnauba wax with copper naphthenate (blue-red); 3:5-dichloro-*o*-aminophenol \rightarrow 2:3-hydroxynaphthoic anilide and triethanolamine in warm benzylcellulose varnish with cobalt naphthenate (violet). C. HOLLINS.

Manufacture of coloured polyvinyl ester varnishes. I. G. FARBENIND. A.-G. (B.P. 334,145, 3.12.29. Ger., 3.12.28).—Polyvinyl ester varnishes are coloured by means of water-insoluble amino- or polyamino-anthraquinone dyes, *e.g.*, 1:4-diamino-2:3-diphenoxy-anthraquinone (red), 1:4-di-*p*-toluidinoanthraquinone (green), 1-*p*-toluidino-4-methylaminoanthraquinone (greenish-blue), 1:4-di-*p*-toluidino-8-hydroxyanthraquinone (olive-green); the second and fourth of these are soluble in the varnish and give transparent films. C. HOLLINS.

Manufacture of synthetic gummy or resinous materials. CANADIAN ELECTRO PRODUCTS CO., LTD., Assees. of (A) F. W. SKIRROW, (B) H. W. MATHESON and F. W. SKIRROW (U.S.P. 1,746,615 and 1,746,665, 11.2.30. Appl., [A] 15.8.27, [B] 28.2.27).—Resins useful as substitutes for chicle are obtained by exposing a mixture of a vinyl ester (acetate) and an aldehyde (acetaldehyde) to (A) actinic rays, *e.g.*, from a mercury-arc lamp, preferably at 30–40°, or (B) to heat-treatment at 100°, with or without a catalyst (hydrogen chloride, hydrogen peroxide). Contact with iron or copper should in each case be avoided. C. HOLLINS.

Manufacture of [plastic] artificial masses. CON-SORT. F. ELEKTROCHEM. IND. G.M.B.H., and O. DÖRR, Assees. of DÖRR & HOFMAN (B.P. 313,578, 14.6.29. Ger., 14.6.28. Cf. B.P. 271,090; B., 1928, 793).—Filling materials, *e.g.*, ground slate, cork, or wood meal, are incorporated in a solution of an alcohol-soluble polymerised vinyl ester, in such proportions that the product can be sprayed. E. LEWKOWITSCH.

Manufacture of condensation products from a urea and formaldehyde. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 332,634, 19.4.29).—The dispersions or solutions obtained by condensing urea or its derivatives with formaldehyde in organic solvents (cf. B.P. 319,251; B., 1929, 1047) are submitted to steam-distillation (if desired, with the application of a vacuum and/or moderately superheated steam) in order to remove the volatile constituents and solvents. The residual watery gel is allowed to settle until the bulk of the water has separated spontaneously, and is then rolled, kneaded, and dried at 50–60°, to yield a clear glassy product which may be subjected to fractional extraction with organic solvents. E. LEWKOWITSCH.

Resinous substances and compositions containing them. BRIT. CELANESE, LTD. (B.P. 311,657,

13.5.29. U.S., 12.5.28).—Lactic acid, preferably of 90% concentration, is heated under reflux for several hours, and the volatile matter is then distilled off. The light-coloured resinous product (m.p. 30–35°) may be used in conjunction with cellulose acetate, resins, plasticisers, solvents, colouring materials, etc. S. S. WOOLF.

[Resinous] saturant for fibrous bases and its preparation. I. J. NOVAK (B.P. 334,232, 27.5.29).—Cresylic acid is condensed with formaldehyde by heating with 0.25–0.75 mol. of ammonia per mol. of cresylic acid, and the product is heated with tung oil until the solution is clear at normal temperature; it is afterwards cooled to retard further reaction. F. R. ENNOS.

Process and apparatus for making white lead. E. C. WALKER (B.P. 334,751, 11.10.29).—See U.S.P. 1,732,490; B., 1930, 156.

Manufacture of phenolic resins. N. STRAFFORD and E. E. WALKER, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,776,202–3, 16.9.30. Appl., 30.6.28. U.K., 7.7.27).—See B.P. 296,514; B., 1928, 826.

Manufacture of abrasive article [flexible sand-paper]. R. P. CARLTON, Assr. to MINNESOTA MINING & MANUF. CO. (U.S.P. 1,775,631, 16.9.30. Appl., 13.3.26).—See B.P. 267,516; B., 1928, 826.

Manufacture of moulded articles [from wood chips or sawdust and synthetic resin]. BOIS BAKÉLISÉ and LA BAKELITE (B.P. 334,804, 6.12.29. Fr., 7.12.28).

Polymerisation process (U.S.P. 1,746,168). **Polymerised vinyl esters** (B.P. 319,682). **Polymerised styrene** (B.P. 307,936). **Anti-agers** (B.P. 333,941).—See III. **Azo pigments** (B.P. 332,932 and 332,940).—See IV. **Shaped fibrous materials** (B.P. 312,178). **Cellulose compositions of low inflammability** (B.P. 312,609).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Extraction of gutta-percha leaf. A. W. K. DE JONG (Rec. trav. chim., 1930, 49, 827–840).—Extraction of finely-powdered gutta-percha leaf with benzene at 25° and addition of 85–95% alcohol to the extract precipitates a gutta-percha which, after melting in water, pressing to a thin film, and then fusing at 100° for 1 hr., contains a large proportion of second-grade (immature) material. It is shown that second-grade gutta-percha is the pure hydrocarbon modified by melting. Shavings of gutta-percha melted in water at 62° can be drawn out to give threads which shrink to the original size in warm water. Gutta-percha appears to exist in three forms with differing solubilities in benzene and light petroleum; the difference is attributed to the size of the particles. Extraction of the leaf with light petroleum (b.p. 80–100°) at 35° and cooling the extract to 17° gives a pure gutta-percha only slightly soluble in benzene at 16°. This method of extraction is recommended.

H. BURTON.

Conditions affecting the vulcanisation of rubber. V. **Effect of accelerators on the heat of vulcanisation.** Y. TOYABE (J. Soc. Chem. Ind., Japan, 1930, 33, 275–276 B; cf. B., 1930, 520).—The heat of vulcanisation of rubber compounds with different amounts of

sulphur and various organic accelerators was followed by means of a differential thermocouple. The profiles of the heating curves were similar to those obtained with unaccelerated compounds, except that the temperature maxima (corresponding to disaggregation maxima of the rubber hydrocarbons) appeared earlier in the former case. The degree of sulphur combination is much the same in both cases. The experiments showed that the accelerators favour the combination with sulphur by promoting the disaggregation of the micellar structure of the rubber.

E. LEWKOWITSCH.

PATENTS.

Dispersed rubber isomeride. H. L. TRUMBULL, Assr. to B. F. GOODRICH Co. (U.S.P. 1,744,844, 28.1.30. Appl., 2.6.27).—Rubber isomerides, *e.g.*, of the thermoplastic type obtainable by heating with isomerising agents, are subjected to mechanical working on a warm mill, while a hydrophilic colloid, *e.g.*, casein, glue, or colloidal clay, is incorporated, and water is then added gradually. A dispersion is thus finally obtained with the colloid paste as continuous phase surrounding the rubber isomeride in such condition that dilution and concentration are possible. If the rubber isomeride is brittle an alternative procedure is to reduce it to powder and subject a mixture with water and a protective colloid to the action of a colloid mill.

D. F. TWISS.

Rubber latex cement and its preparation. B. DEWEY and E. C. CROCKER (U.S.P. 1,745,084, 28.1.30. Appl., 20.11.24).—Finely-divided sulphur is dispersed into rubber latex together with a smaller proportion of bentonite; the presence of the latter effectively maintains the sulphur in suspension. A permanent suspension of this type, capable, when dried, of vulcanisation to a hard condition, can be produced by adding a mixture of flowers of sulphur (90 pts.), bentonite (15 pts.), saponin (3 pts.), and water (590 pts.) to latex (462 pts.) containing about 38% of rubber.

D. F. TWISS.

Product and process for rubber compounding. A. B. COWDERY, Assr. to BARRETT Co. (U.S.P. 1,743,433, 14.1.30. Appl., 22.11.24).—The volatile constituents are distilled from coal tar in such a way as to give a residue containing a high proportion (60%) of uncombined carbon or carbonaceous matter insoluble in benzene. The residue, having *d* 1.30–1.35 and m.p. above 176°, can be incorporated in rubber up to the extent of about 15%, and does not produce excessive discoloration of brown mixtures.

D. F. TWISS.

Vulcanisation of rubber. W. SCOTT, Assr. to RUBBER SERVICE LABS. Co. (U.S.P. 1,743,243, 14.1.30. Appl., 28.9.27).—An accelerator of vulcanisation is formed by heating an organic derivative of ammonia, *e.g.*, piperidine or diphenylguanidine, with a cyanogen halide reaction product of a mercaptan, *e.g.*, with the product from cyanogen chloride and sodium mercaptobenzthiazole.

D. F. TWISS.

Manufacture of elastic products. I. G. FARBENIND. A.-G. (B.P. 318,967, 3.9.29. Ger., 13.9.28).—Highly elastic products are made from esters of polymeric carbohydrates containing residues of saturated or unsaturated cyclic-substituted fatty acids in addition to one or more residues of simple saturated or unsaturated fatty acids, *e.g.*, cellulose naphthenate oleate or starch naphthenate oleate stearate. These can be

worked by any process suitable for working rubber, and can be calendered and vulcanised. They may be used alone or mixed with colouring matters, natural or synthetic rubber, other polymeric carbohydrate compounds, etc.

D. F. TWISS.

Manufacture of goods of rubber or similar material. DUNLOP RUBBER Co., LTD., and (A) W. H. CHAPMAN, D. W. POUNDER, and E. A. MURPHY, (B) E. A. MURPHY and E. W. B. OWEN (B.P. 332,525–6, 21.3.29).—(A) Mixtures of compounded aqueous dispersions of rubber or similar material to which lather-forming substances, *e.g.*, ammonium oleate, have been added together with substances having delayed coagulating action, *e.g.*, sodium silicofluoride or ammonium persulphate, are converted into a frothy condition by stirring with mesh-work paddles and/or by injecting air or other means. The frothy mass is poured into open moulds and allowed to coagulate, with or without the aid of heat; it is then vulcanised and dried. Alternatively, the coagulating agent may be added to the froth subsequent to its formation, the agitation being continued for a short time longer to ensure uniform distribution. The product is a material of sponge-like or cellular structure. (B) Frothed mixtures of compounded aqueous dispersions of rubber etc. produced as above are formed into shape by spreading, pouring into shallow trays, or by dipping processes etc., and are then “set” by heating in a gentle current of air or by other means. Articles of sponge rubber, *e.g.*, sheet for mats, insulating material, etc., also vulcanite sponge sheet, are thus obtained.

D. F. TWISS.

Rubberised material. P. H. W. CLOUD (B.P. 332,537, 19.4.29).—A rubber felt-like material, which may be moulded or patterned, is produced by mixing rubber with animal fibres and fillers, with or without a vulcanisation accelerator and sulphur, and heating to a temperature below that capable of deleteriously affecting the animal fibres. [Stat. ref.]

D. F. TWISS.

Direct production of filaments and threads [of rubber etc.]. DUNLOP RUBBER Co., LTD., E. A. MURPHY, and D. F. TWISS (B.P. 333,005, 13.6.29. Addn. to B.P. 311,844; B., 1929, 612).—The partly set filaments are passed between even-speed rollers which may be rubber-coated and/or embossed. Tape-like filaments are thus produced.

D. F. TWISS.

Manufacture of articles [*e.g.*, cables covered with gutta-percha] from thermoplastic materials. W. S. SMITH, H. J. GARNETT, and J. N. DEAN (B.P. 333,879, 16.5.29).

Polymerisation process (U.S.P. 1,746,168). **Polymerised diolefines** (B.P. 333,894 and 333,872). **2-Chlorobenzthiazoles** (B.P. 310,815). **Anti-agers** (B.P. 333,941).—See III. **Paving blocks** (B.P. 333,047).—See IX.

XV.—LEATHER; GLUE.

Sorrels: new tanning plants. **Tannin content of species of *Rumex*.** P. SORS (Collegium, 1930, 324–329).—Analyses of the roots of various species of Hungarian sorrels yielded the following results for tans and soluble non-tans (%) on a moisture-free basis: *Rumex maritimus* traces; *R. paluster* (*limosus*) 6.33, 4.59; *R. conglomeratus* 6.33–10.77, 8.87–19.97;

R. sanguineus 12·56, 13·56; *R. odontocarpus* (*Senophyllus biformis*) 3·16, 5·23; *R. hydrolapathum* 11·50—21·32, 7·64—18·98; *R. crispus* 3·62—6·24, 6·63—21·55; *R. patientia* 16·55—21·4, 11·37—29·30; *R. acetosella* traces; *R. acetosa* 2·78—22·66, 7·20—37·40. Some qualitative reactions of these plants are given. The plants are costly to gather, and the tans: non-tans ratio is poor, but can be improved by enrichment or by sulphiting. The liquors are very liable to fermentation owing to high content of non-tans. The high content of dyestuff of some varieties, e.g., *R. patientia*, unfits them for tanning purposes, since they render the leather harsh and brittle. Two of the varieties richest in tannin, *R. acetosa* and *patientia*, are utilised largely for other purposes and the roots form a valuable by-product.

D. WOODROFFE.

Differentiation of vegetable tannins by means of antipyrin-hydrochloric acid and the quartz lamp.

W. APPELIUS and L. KRIGUELOUKIS (Collegium, 1930, 330—333).—25 C.c. of an infusion of a tanning material or solution of an extract containing about 10% of tannin are boiled under reflux in a flask fitted with a separating funnel, 20 c.c. of a 1% solution of antipyrin and 30 c.c. of a 10% hydrochloric acid solution are added, and boiling is continued for 20 min. The product is filtered through a hardened filter and left in contact with wool strips for 15 min.; these are then washed in cold water, drained, touched with 10% caustic soda, and while still damp examined by ultra-violet light. Characteristic colour reactions and fluorescences are obtained before and after treatment with dilute sodium hydroxide.

D. WOODROFFE.

Problem of tanning. P. D. ZACHARIAS (Rev. gén. Colloid., 1930, 8, 260—264).—A résumé of the author's publications during the last 30 years, showing that they contain a generalised theory of the process of tanning based on an analogy with dyeing. E. S. HEDGES.

Evaluation of spent tan liquors by interferometry.

F. ENGLISH (Collegium, 1930, 310—323).—It is shown that the interference produced by 1 g. of tannin or other substances in tan liquors is independent of the concentration of the tan liquors. It is proposed, therefore, to use the interferometer to evaluate spent tan liquors, for which purpose it is shown that the equivalent (interference) and percentage content of the tannin irreversibly absorbed by hide powder are important. These figures are obtained by either shake or filter-bell methods of analysis followed by a washing of the tanned hide powder. Both methods yield approximately the same figures for the irreversibly adsorbed tannin. Particulars are given for the evaluation of the interference equivalents for the tannin, adsorbed non-tans, and unadsorbed non-tars from the figures obtained on the analytical tannin solutions, but these can only be applied to spent liquors from pure materials. The factors vary with the composition of the spent liquors.

D. WOODROFFE.

PATENTS.

Production of tannin extracts and cellulose or pulp. FORESTAL LAND, TIMBER, & RAILWAYS CO., LTD., and R. O. PHILLIPS (B.P. 332,935, 25.1.29).—Suitable tannin-containing woods or barks, e.g., quebracho, chestnut, wattle bark, are extracted with a solution of a mixture of sodium sulphite (3 pts.) and sodium bicar-

bonate (1 pt.), using about 50 lb. of mixed chemicals per ton of wood. The resulting tan liquor is led off and the residual cellulose or pulp treated as usual.

D. WOODROFFE.

Moth-protection of fur etc. (B.P. 333,583—4).—See VI.

XVI.—AGRICULTURE.

Soil acidity and soil adsorption. O. LEMMERMANN and L. FRESenius (Verhandl. II Komm. Internat. Bodenkundl. Ges., 1929, A, 36—43; Bied., Zentr., 1930, 59, 385—386).—For the preparation of soil suspensions for p_H determinations a soil:water ratio of 1:1 or 1:0·5 is recommended for sands and loams. For moorland soils a wider ratio (up to 1:25) may be used. With fresh moist samples contact for 15—20 min. is necessary; a shorter period suffices for dried soils. With the quinhydrone electrode equilibrium of potential is not instantaneous, but is accelerated by the use of considerable excess of quinhydrone. Observations of alkaline soils must be made quickly. For the determination of the buffer capacity of soils a 1:1 soil:water ratio is to be used. Daikuhara's method for lime requirement gives uncertain values for practical use. The merits of the "hydrolytic acidity" method and of titration curves for determining lime requirements are discussed. In obtaining soil-titration curves the samples of soil (10 g.) are allowed to react with acid or alkali in 100 c.c. of fluid for 3 days before p_H values are determined.

A. G. POLLARD.

Effect of artificial acidification on soil and on plant growth.

E. F. VON OLDERSHAUSEN (J. Landw., 1930, 78, 241—287).—Soils treated with varying proportions of hydrochloric and sulphuric acids are examined by means of pot cultures and by analysis. The development of acidity in soils is closely related to the removal of bases. Acidity, as a result of the removal of electrolytes from the soil, causes a peptisation of the particles and, as a consequence, an increase in the total surface area of the soil and a decreased permeability to water. Reduced plant growth in acid soils is caused not only by actual acidity, but by alterations in the physical properties of soils and a lack of exchangeable nutrient bases. Lining counteracts the removal of bases and improves physical conditions, but normal plant growth cannot be attained without the simultaneous use of fertilisers.

A. G. POLLARD.

Influence of adsorbed ions on soil reaction.

B. AARNIO (Bull. Agrogeol. Inst. Finland, 1927, No. 22, 13 pp.).—When very dilute acid is added to soil the reaction becomes slightly more basic than in aqueous solution, owing to liberation of adsorbed hydroxyl ions and exchange with anions of the acid. Hence ionic exchange appears to occur among anions.

CHEMICAL ABSTRACTS.

Comparative rate of percolation of water in different soils. G. J. BOUYOUCOS (J. Amer. Soc. Agron., 1930, 22, 438—445).—The method of measurement consists in allowing a soil to slake in excess of water into its ultimate natural structure, stirring gently, and then applying suction and measuring the rate of flow of the water.

CHEMICAL ABSTRACTS.

Carbon dioxide production in soils. T. OEHLER (Z. Pflanz. Düng., 1930, 9B, 415—421).—Artificial

aeration of soil in pot cultures increased crop yields and also the production of carbon dioxide by soil organisms.

A. G. POLLARD.

Potash content of Palatinate soils and their appropriate fertiliser treatment. M. KLING and O. ENGELS (Z. Pflanz. Düng., 1930, 9B, 409—415).—Results of the examination of soils by the seedling method are discussed and recommendations given for suitable potash fertilisation of a number of crops.

A. G. POLLARD.

Utilisation of increasing dressings of potash by different varieties of barley. H. LIESEGANG (Z. Pflanz. Düng., 1930, 9B, 397—409).—The increase in the grain : straw ratio, the weight per 1000 grains, and the proportion of potash in the grain, following potash fertilisation, is compared for five varieties of barley.

A. G. POLLARD.

Action and utilisation of various water-insoluble phosphates in different soils, especially regarding their influence on the phosphate content of plants. M. CLEMM (J. Landw., 1930, 78, 289—308).—Comparison is made of the efficiencies of Rhenania phosphate and basic slag. In Neubauer tests the ammonium citrate-soluble phosphate of Rhenania phosphate was more effective on clay soils than the citric acid-soluble phosphate of slag. Both fertilisers were highly efficient on light sandy soils of weakly acid reaction. In loams of considerable alkalinity seedlings did not take up phosphate from either fertiliser, and in some cases there was an apparent transition of phosphorus from plant to soil. In pot cultures Rhenania phosphate (citrate-soluble) was superior to basic slag (citric acid-soluble). In all cases the increased phosphate assimilation by barley, following fertilisation, was definitely marked by the increased phosphate content of the grain. Changes in the phosphate content of the straw were less definite and were absent where alkaline soils rich in phosphate were used. Ammonium citrate-soluble phosphate affects more particularly the phosphate content of the grain, whereas citric-soluble phosphates chiefly influence that of the straw.

A. G. POLLARD.

Liming as a factor in the mobilisation of phosphoric acid in podsols. D. ASKINASI and S. JARUSSOV (Trans. Inst. Fertilisers, 1928, 57, 5; Bied. Zentr., 1930, 59, 393).—Field and pot-culture observations show that the liming of podsols accelerates the decomposition and mineralisation of humus. The latter process is associated with an increased proportion of nitrogen and phosphate in the soil solution and a corresponding increase in the crop yield and in its nitrogen and phosphate contents.

A. G. POLLARD.

Quantitative analysis of phosphoric acid. VI. Determination of phosphoric acid in a phosphate fertiliser. M. ISHIBASHI (Mem. Coll. Sci. Kyoto, 1930, 13A, 291—301; cf. A., 1929, 529, 783).—The author's improved volumetric phosphomolybdate method is especially suitable for the determination of small quantities of phosphoric acid. For the extraction of free phosphoric acid acetone is more satisfactory than either alcohol or ether.

C. W. GIBBY.

Effect of soil type and fertiliser on the nitrate content of the expressed sap and the total nitrogen content of the tissue of the small grains. R. L.

COOK (J. Amer. Soc. Agron., 1930, 22, 393—407).—Nitrogenous fertilisers increased, whilst other fertilisers diminished, the nitrate content of the sap, which varied directly with the tissue-nitrogen. Spring grains possessed greater ability to accumulate sap-nitrate than did winter grains. The tissue-nitrogen diminished steadily during the growing season.

CHEMICAL ABSTRACTS.

Effect of sodium nitrate on the composition of the expressed sap of the small grains. M. M. MCCOOL and M. D. WELDON (J. Amer. Soc. Agron., 1930, 22, 434—437).—The phosphorus content of the sap of barley, oats, rye, and wheat increased with the age of the crop, whilst the potassium content tended to decrease. Application of sodium nitrate diminished the sap-calcium, and large applications diminished the sap-potassium also. The sap-calcium at first diminished with increasing age of the crop, increasing near maturity. The magnesium content fluctuated less than that of other elements on application of sodium nitrate.

CHEMICAL ABSTRACTS.

Artificial manures in forest management. KUHNERT (Z. Pflanz. Düng., 1930, 9B, 385—392).—Results of fertiliser trials with several species of trees show that nitrogenous fertilisers, used in appropriate complete fertiliser combinations, increase both the height and diameter of the trees.

A. G. POLLARD.

Preparation of stable arsenical insecticidal suspensions slightly soluble in water. A. N. KRESTOVNIKOV and G. F. LYUTRINGSCHAUZEN (Min. Suir. Tzvet. Met., 1929, 4, 431—437).—Suspensions of the compounds $3\text{Cu}(\text{AsO}_2)_2$, $\text{Cu}(\text{OAc})_2$, and $\text{Cu}(\text{AsO}_2)_2$ are prepared by passing through a colloid mill 1—5 g. of the suspension in 1 litre of water and 1% of starch or 10% of kaolin.

CHEMICAL ABSTRACTS.

Control of certain fruit diseases with "flotation sulphurs." M. A. SMITH (Phytopath., 1930, 20, 535—553).—Trials with flotation sulphur (from gas-purifying process) on a number of fungus diseases of fruit are recorded. Particle size of flotation sulphur varied from 1 μ to 5 μ in diam. as against 6—200 μ for ground sulphur and 20—130 μ for sulphur flowers. When washed free from sodium thiosulphate and thiocyanate, flotation sulphur produced no foliage injury when applied in higher concentrations than is usual in practice. Very satisfactory results with apple scab, brown rot, and cherry-leaf spot are recorded, and when combined with oil emulsion flotation sulphur satisfactorily controlled peach leaf-curl.

A. G. POLLARD.

Fruit-fly control. L. B. RIPLEY and G. A. HEPBURN (Farming in S. Africa, 1929, 4, 345—346, 357).—The strongest attractant is fermenting wheat pollard. With pure compounds as attractants, an optimal concentration exists; a 0.1% solution of linalyl acetate gives promising results. The strongest repellent is peppermint oil.

CHEMICAL ABSTRACTS.

Soil as source of infection of honey. LOCHHEAD and FARRELL.—See XIX.

PATENTS.

Manufacture of (A) a compound of urea and calcium nitrate, (B) solid urea or fertilisers containing urea. J. Y. JOHNSON. From I. G. FARBERNIND. A.-G. (B.P. 332,945 and 332,948, 29.4.29).—(A) Calcium

nitrate (1 mol. to 2–6 mols. of urea) is added to urea solutions; the compound is more stable than urea alone. (B) To avoid the increased temperatures necessary to remove the last 2–3% of water from molten urea, about 10% of diammonium hydrogen phosphate, phosphoric acid, formamide, or other substance which lowers the m.p., is added to the urea solution; the solution is then evaporated down to 0–2% of water, the pressure being adjusted to maintain the b.p. above the mixed m.p. The melt is then granulated by spraying or running into mineral oil etc. C. HOLLINS.

Sodium nitrate (B.P. 315,262). **Superphosphate** (B.P. 333,518).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Polyfructoses and their determination. I. **Determination of lævulosin for the detection of artificial invert sugar in honey.** C. I. KRUISHEER (Rec. trav. chim., 1930, 49, 841–849).—The presence of artificial invert sugar in honey is shown by the determination of the amount of lævulosin (Wohl, A., 1890, 1085) present. This is formed when a concentrated solution of lævulose is heated with a small amount of acid. The method described is as follows. Honey (10 g.) is diluted with water (75 c.c.), boiled, cooled, and the solution fermented during 48 hrs. with yeast (5 g., + 10 c.c. of sterile water). Portions of the resulting solution, after neutralisation and removal of alcohol by evaporation, are inverted by heating with hydrochloric acid for 10 min. and 3 hrs., respectively, and their lævulose contents determined by the hypiodite method. The difference (if any) in the values indicates the lævulosin content. Lævulosin, unlike other lævulose derivatives, is incompletely hydrolysed during the short treatment. H. BURTON.

Physical chemistry of starch and of bread-baking. I. Alteration of X-ray spectrum of starch in the baking and the ageing ["staling"] of bread. II. The modification of starch with V-spectrum (gelatinisation spectrum) is the equilibrium form at a high, and that with B-spectrum (retrogradation spectrum) at a low, temperature. III. First and second stages of gelatinisation. J. R. KATZ (Z. physical. Chem., 1930, 150, 37–59, 60–66, 67–80).—I. [With L. M. RIENTSMA, T. B. VAN ITALLIE, and (Frl.) L. HEYNA.] X-Ray analysis of wheat or rye starch, of dough, and of bread in various conditions gives the following results: (a) The original flour, whether of wheat or rye, gives a characteristic interference pattern (A-spectrum) which is maintained unaltered in the "risen" dough; (b) in the crumb of freshly-baked bread the starch gives a different spectrum (gelatinisation or V-spectrum); (c) in the crumb of stale bread a third spectrum (retrogradation or B-spectrum) is obtained, closely resembling that of the original flour, but not identical with it; (d) freshly-baked crust, in so far as it is formed by rapid dehydration of the original dough, shows practically only the A-spectrum. It is inferred that the baking operation causes a profound change in the starch, probably of a chemical nature, provided sufficient water is present, as in the crumb. Staleness is the result of a reversion of the gelatinised starch to a substance very similar to the original.

Staleness is not dependent on loss of water, and does not ensue, at least for weeks, if the baked bread has been dried with 96% alcohol.

II. [With L. M. RIENTSMA.] A true physico-chemical equilibrium subsists between the modifications of starch (called, respectively, β_B - and α -starch) which give rise to the B- and V-spectra. The former is stable below, and the latter above, 60°. The results of X-ray analysis are in agreement with those of earlier experiments on the swelling capacity of untreated, of gelatinised, and of retrograded starch.

III. [With L. M. RIENTSMA and T. B. VAN ITALLIE.] The gelatinisation of starch takes place in two distinct stages. The first (corresponding with the condition in newly-baked bread) is completed when starch is heated with a limited quantity (50–100%) of water at 100°, or with excess of water at 62.5°; the second requires the heating of starch with excess of water at 90–100°. Although the product of the second stage (starch paste) shows a higher swelling capacity and a higher proportion of soluble amyloses, it gives, after dehydration with alcohol, the X-ray spectrum characteristic of α -starch. It also undergoes retrogradation to β_B -starch when kept at the ordinary temperature, although more slowly than does the product of the first stage. The results are discussed in relation to the microscopical appearance of gelatinised starch and the chemical reactions of starch paste. F. L. USHER.

Fermentation of honey. LOCHHEAD and FARRELL.—See XIX.

PATENTS.

Synthetic gummy materials (U.S.P. 1,746,615 and 1,746,665).—See XIII. **Methyl α -hydroxyethyl ketone and diacetyl** (B.P. 315,264).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Preservation of sweet white wines. L. MOREAU and E. VINET (Ann. Falsif., 1930, 23, 408–412).—Wines cannot with certainty be kept free from further fermentation by increasing up to the toxic limit the waste products of the life of the yeast. This was, however, accomplished by successive heatings to a temperature above the thermal death point of the yeast and subsequent re-infection after each heating. It is necessary to treat these wines with sulphurous acid to prevent recurrence of fermentation entirely, and, owing to combination, large doses of sulphurous acid are required to produce the requisite concentration of the free acid. B. W. TOWN.

Determination of ferrous iron in white wines. P. MALVEZIN (Ann. Falsif., 1930, 23, 412–414).—The wine is treated with potassium ferrieyanide and the tint compared with that given with a standard solution of ferrous iron. B. W. TOWN.

Fermentation products from cornstalks. C. S. BORUFF [with A. M. BUSWELL] (Ind. Eng. Chem., 1930, 22, 931–933; cf. B., 1930, 117).—The anaerobic fermentation of cornstalks in the presence of overflow liquor from a sewage disposal plant, which served as an inoculum and as a suitable source of nitrogen for the bacteria, was studied by determining the composition of the residue and the proportions of the products (carbon dioxide, hydrogen, methane, and volatile acids)

formed. It was shown that the gases and acids formed represent 118% of the organic matter decomposed, which is higher than the theoretical yield from either cellulose or pentosans, indicating a partial decomposition of the lignin.

F. R. ENNOS.

Fermentation of honey. LOCHHEAD and FARRELL.—See XIX.

PATENTS.

Preparation of acetylmethylcarbinol [methyl α -hydroxyethyl ketone] and diacetyl [by fermentation]. T. H. VERHAVE, SEN. (B.P. 315,264, 25.6.29. Holl., 10.7.28).—A yield of 35% of the carbinol is obtained by intensively aerating a fermenting mash containing a carbohydrate (beet molasses, maize flour), a source of nitrogen, a phosphate, a carbonate (chalk), and a bacterial culture normally producing $\beta\gamma$ -butylene glycol (*Aerobacter aerogenes*, *Clostridium polymyxa*, etc.). The aeration (e.g., 10–25 cub. m. per hr. per hectolitre) may be commenced shortly after fermentation has become evident, and may be progressively increased.

C. HOLLINS.

Active substances from hypophysis glands (B.P. 333,155).—See XX.

XIX.—FOODS.

Oxidation-reduction in milk. Methylene-blue reduction test. H. R. THORNTON and E. G. HASTINGS (J. Dairy Sci., 1930, 13, 221–245).—The chief variations in the results of the test, which depends on the removal of dissolved oxygen by bacteria followed by the reduction of the dye by the milk constituents, are due to the different rates of uptake of oxygen by different bacterial species, and the removal of bacteria in the cream. Shaking decreases the time of reduction.

CHEMICAL ABSTRACTS.

Variations in the density of the whey with the ageing of milk. F. BERNARDINI and E. A. GAUTHIER (Annali Chim. Appl., 1930, 20, 236–238).—When milk is kept either at 3° or at the ordinary temperature, the percentage of fat remains unchanged after a month. At 3° the density of the serum remains virtually constant for 5 days, falls somewhat during the succeeding 5 days, and then keeps constant again; the total fall amounts to about 0.001. At the ordinary temperature the loss in density is more rapid and more considerable, owing to attack of the proteins and sugars, not by lactic organisms, but by moulds.

T. H. POPE.

Milk of the silver fox. O. LAXA Ann. Falsif., 1930, 23, 404).—A complete analysis is given.

B. W. TOWN.

Processed cheese. H. L. TEMPLETON and H. H. SOMMER (J. Dairy Sci., 1930, 13, 203–220).—Sodium citrate gave a firmer body to cheese than did sodium phosphate. The tin foil is darkened by over 2% of phosphate. Cheese body depends largely on the water content, but varies also with the p_H ; an apparatus for determining cheese body is described. The water-soluble nitrogen in cheese is increased by processing.

CHEMICAL ABSTRACTS.

Fat content of Gruyère cheese. P. SAJOUS (Ann. Falsif., 1930, 23, 396–403).—It is suggested that the manufacture of Gruyère cheese should be standardised and classified as (a) "tout-gras" or "extra

gras" (45% of fat); (b) "gras" (40%); and (c) a quality containing below 40% of fat, calculated on dry material. The fat content of a cheese is roughly fourteen times that of the milk from which it is prepared.

B. W. TOWN.

Effect of processing ice-cream mixtures at different pressures when the milk solids-not-fat content is varied. W. H. E. REED and E. R. GARRISON (Missouri Agric. Exp. Sta. Res. Bull., 1929, No. 128, 41 pp.).—The temperature to which an ice-cream mixture can be frozen is determined by the composition and the pressure; the temperature is lowered by an increase in the non-fatty solids. Mixtures with a high content of non-fatty solids require a lower processing pressure than those of lower content.

CHEMICAL ABSTRACTS.

Vitamin-G [vitamin- B_2] in certain meats and meat by-products. R. HOAGLAND and G. G. SNIDER (J. Agric. Res., 1930, 41, 205–220).—Feeding experiments with young albino rats have been carried out to determine the relative amounts of vitamin- B_2 in certain meats, compared on the air-dry, fat-free basis. From 15 to 25% of the ration, dried beef, pork, or lamb, furnished sufficient vitamin- B_2 for excellent growth. Beef spleen contained about the same amount as beef itself, whilst beef and pork liver and beef kidney contained 5–8 times as much vitamin as did beef, pork, or lamb; 3% of the ration of beef liver, 3.05% of pork liver, and 2.77% of beef kidney furnished an ample supply of the vitamin.

E. HOLMES.

Soil as a source of infection of honey by sugar-tolerant yeasts. A. G. LOCHHEAD and L. FARRELL (Canad. J. Res., 1930, 3, 51–64).—Sugar-tolerant yeasts, capable of fermenting honey, are regularly found only in apiary soils; they are not members of the normal field-soil microflora. These yeasts, which remain viable during the winter in frozen soil, reduce to seven types, five being classified as *Zygosaccharomyces* and the others as species of *Torula* and *Mycotorula*, respectively.

T. H. MORTON.

Effect of preservatives on fermentation by sugar-tolerant yeasts from honey. A. G. LOCHHEAD and L. FARRELL (Canad. J. Res., 1930, 3, 95–103).—With the object of finding a suitable preservative for honey, the rates of fermentation have been measured of an 80% honey solution inoculated with a mixed culture of yeasts in presence of various antiseptics. Sodium hypochlorite and chloramine-T did not prevent fermentation in a concentration of 100 pts. of available chlorine per million. Fermentation was inhibited by the following substances in the concentrations indicated: hydrogen peroxide 0.5%, hexylresorcinol 0.01%, sodium salicylate 0.06%, sodium borate 0.1%, sodium sulphite 0.01%, sodium hydrogen sulphite 0.025%. Considerations such as taste and legal limits indicate that the last three substances are suitable preservatives.

R. K. CALLOW.

Does sugar inversion affect pectin jelly formation? G. M. COLE, R. E. COX, and G. H. JOSEPH (Food Ind., 1930, 2, 219–221).—Invert sugar and dextrose give satisfactory jellies with the correct amounts of pectin and acid. Sucrose in jellies continues to invert during storage and does not affect the quality of the jelly. Jellies can be made when glycerol, ethyl alcohol,

or glacial acetic acid are used instead of sucrose. Pre-mature gelation is discussed. CHEMICAL ABSTRACTS.

Detection of artificial invert sugar in honey. KRUISHEER. **Starch and bread-baking.** KATZ.—See XVII.

PATENTS.

Treatment of wheat germ for use as an edible food substance. K. P. WANKLYN and F. STACEY (B.P. 332,847, 31.10.29).—Wheat germ will keep indefinitely if dried at 57° under reduced pressure for 8 hrs. and milled. E. B. HUGHES.

Food product. E. B. BROWN, C. N. FREY, and H. M. HARKINS, Assrs. to STANDARD BRANDS, INC. (U.S.P. 1,755,864, 22.4.30. Appl., 2.9.27).—A bread improver is prepared by heating under pressure a mixture of starch and yeast. After conversion of the starch into sugar is complete the product is neutralised and then reacidified with lactic acid. E. B. HUGHES.

Treatment of milk. H. E. MORTLAND, Assr. to PEVELY DAIRY Co. (U.S.P. 1,757,646, 6.5.30. Appl., 6.2.28).—To skimmed milk (1000 pts.) at 62.8° is added gradually a cold mixture of cane sugar (35 pts.), lime (7 pts.), and water (50 pts.), and the product after being condensed in the vacuum pan is suitable for use as a filler in ice-cream etc. E. B. HUGHES.

Meat-curing method. R. E. JORDAN, Assr. to ALLBRIGHT-NELL Co. (U.S.P. 1,755,992, 22.4.30. Appl., 13.5.27).—The meats or meat products are circulated on a conveyor in a vertical smoke-house. The method is claimed to avoid the formation of an impervious crust of coagulated albumins, accelerate penetration of curing gases, and minimise shrinkage. E. B. HUGHES.

Sterilisation of food. O. H. HANSEN, Assr. to HANSEN CANNING MACHINERY CORP. (U.S.P. 1,756,549 and 1,756,550, 29.4.30. Appl., 15.6.29).—(A) The material, e.g., peas, is sterilised by heating in a relatively dry condition, partly cooled, and after addition of cool sterilised brine the product is canned. (B) Soft fruits, requiring surface sterilisation only, before being canned are immersed in a relatively small quantity of the liquid at a suitable temperature. E. B. HUGHES.

Manufacture of [concentrated] vegetable foods. E. H. MILES and G. REILLY (U.S.P. 1,775,966—7, 16.9.30. Appl., 8.7.26. U.K., 15.7.25).—See B.P. 256,765 and 274,051; B., 1928, 425.

Disintegration of cellular matter (U.S.P. 1,746,731).—See I. **Fat from carcasses etc.** (B.P. 310,542). **Edible cottonseed oil** (U.S.P. 1,747,675).—See XII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Castor oil soaps in spirituous [medicinal] preparations. W. MEYER (Arch. Pharm., 1930, 268, 358—371).—The author has examined the possibility of detecting adulteration of pharmaceutical preparations, especially those containing olive and linseed oil soaps, by the substitution for pure alcohol of a product to which castor oil (1%) and sodium or potassium hydroxide (0.4%) had been added. Indications of the presence of ricinoleic acid in the mixtures of fatty acids isolated from such products are afforded by (a) determination of constants, especially the acetyl value, (b) incomplete

miscibility with petroleum fractions (15%), (c) formation of sec.-octyl alcohol when the sodium salts are fused with potassium hydroxide (5%), and (d) appearance of a bluish-green colour when the mixture is treated with antimony trichloride and then with chloral hydrate (8%), the numbers given in parenthesis being the smallest percentages of ricinoleic acid which can be identified. The reactions enabled adulterations to be detected in eleven official preparations, but failed with three others. A comprehensive scheme for the detection of denaturants in spirituous products is given.

H. E. F. NOTTON.

Liquor plumbi subacetatis fortis: its preparation and assay. P. A. BERRY (Chem. Eng. Min. Rev., 1930, 22, 421—423).—The use of insufficient litharge in the preparation of this liquor results in a solution low in basicity, whilst an excess, although increasing the basicity, results in the crystallisation of a basic acetate, $\text{Pb}(\text{OAc})_2 \cdot 2\text{PbO} \cdot 4\text{H}_2\text{O}$, with an attendant reduced lead and acetate content. The white deposit normally formed when making this solution is composed of this basic acetate together with basic lead carbonate. The following method for the preparation is suggested: 320 g. of litharge are triturated with 100 c.c. of water and the paste is transferred to a bottle with a further 650 c.c. of water. After shaking, 240 g. of acetic acid are added and the mixture is shaken for 30 min. and then occasionally for 30 min. After setting aside for 24 hrs. the mixture is filtered and the filtrate made up to 1 litre with water. In a proposed method of assay, the lead is determined by the addition of a known excess of standard oxalic acid solution followed by its determination in the filtrate with 0.1N-potassium permanganate, and the acetic acid is calculated from the result of a determination of the total acidity of the filtrate, the oxalic acid content being known from the first titration. This method is equally suited for lead acetate.

E. H. SHARPLES.

Detection of arsenic in medicinal substances and comparison of the sensitiveness of Bettendorff's and Bougault's reagents. A. GRIPPA (Annali Chim. Appl., 1930, 20, 249—257).—Examination of a large number of substances shows that, whereas Bougault's reagent (20 g. of sodium hypophosphite, 40 c.c. of water, and 180 c.c. of concentrated hydrochloric acid) is capable of detecting 0.01 mg. of arsenic per c.c. or g. of material, Bettendorff's reagent is unable to detect less than 0.02 mg. of arsenic. Contrary to statements made, the addition of potassium iodide to Bougault's reagent does not increase the sensitivity and is useful only with ferric compounds to reduce these to the ferrous state.

T. H. POPE.

Assay of ipomea. L. E. WARREN (J. Assoc. Off. Agric. Chem., 1930, 13, 377—383).—Comparison is made of the United States and French official methods with that of Jenkins (B., 1914, 885) for the analysis of ipomea (the dried root of *Ipomea orizabensis*, Ledenois). The French method gives lower values than the others. The modified procedure recommended is as follows: 10 g. of the sample are boiled under reflux for 30 min. with 50 c.c. of alcohol and the mixture is then percolated with warm alcohol to obtain about 95 c.c. of percolate (or more if the last runnings give more than a faint cloudiness when poured into cold water). The volume of

the percolate is adjusted to 100 c.c. Of this 25 c.c. are evaporated on a water-bath to remove alcohol, the residue being washed with water (with kneading) and finally dissolved in alcohol together with portions filtered from the wash-water. The solution is evaporated, dried at 100°, and weighed. A. G. POLLARD.

Bio-assay of capsicum, U.S.P. X. J. C. MUNCH (J. Assoc. Off. Agric. Chem., 1930, 13, 383—385).—Comparison is made of the response of the individual to piperine, with the sensation of pungency in the throat produced by swallowing standard proportions of extract. A. G. POLLARD.

Influence of the pulping of fresh gentian root on its content of glucides. C. BÉGUIN (J. Pharm. Chim., 1930, 12, [viii], 213—219).—During the pulping of fresh gentian root, hydrolysis of the osides is accelerated by the soluble ferments: 21.3% of the holosides hydrolysable by invertin, 19.7% of those hydrolysable by emulsin, and 48.2% of the gentiopicroside are hydrolysed. On keeping the pulp, hydrolysis proceeds regularly, except that due to the emulsin, which seems to be most rapid during the first hour. In 2 hrs. all the gentiopicroside is destroyed, no reaction for gentiogenol is obtained, and there remains only a small amount of product hydrolysable by emulsin. E. H. SHARPLES.

Preservation of gentian root by alcohol vapour. C. BÉGUIN (J. Pharm. Chim., 1930, 12, [viii], 49—63).—Roots sterilised by means of alcohol vapour give on subsequent treatment tinctures similar to, but somewhat weaker than, those obtained directly from fresh roots. The vapour treatment extracts some proportion of the active principles, 6—8% of the content when the roots are whole, and 20—30% when they are cut. S. I. LEVY.

Peppermint oil of the Saratov district. O. V. SOBOLEVSKAYA (Trans. Sci. Chem.-Pharm. Inst., Moscow, 1928, No. 19, 186—194).—The oil of plants screened or unscreened from sunlight contained 47.7—53.05, 51.38—55.38% of menthol and 11.76, 2.96—5.06% of menthone, respectively. In a wet climate the yield of oil is higher, but the quality inferior.

CHEMICAL ABSTRACTS.

Rumanian peppermint oil and the position of the peppermint oil industry in Rumania. E. KOPP (Pharm. Zentr., 1930, 71, 577—582).—The yearly production and the quality of the oils from peppermint plants imported from England and Italy and grown in Rumania since 1926 and the economics of the industry are described. The commercial oils produced at present, in a yield of 30 kg. per hectare, have: yields from green plant 0.20—0.24%, d_{20}^{20} 0.9042—0.9113, α_D $-21^\circ 10'$ to $-27^\circ 9'$, n_D^{20} 1.4602—1.4618, solubility in 70% alcohol 1 in 2.7—3.0 pts., acid value nil, ester value 25.2—44.8, acetyl value 179.2—200.7, ester menthol 3.3—12.5%, free menthol 46.7—50.3%.

E. H. SHARPLES.

Soaps containing salicylic acid. SCHLENKER.—See XII.

PATENTS.

Manufacture of therapeutic agents [against carcinoma, spirochaetes, and trypanosomes]. I. G. FARBERIND. A.-G., Asses. of C. F. BOEHRINGER & SÖHNE, G.M.B.H. (B.P. 318,556, 5.9.29. Ger., 5.9.28).—

The calcium or other alkaline-earth salts of 3- ω -sulphomethylamino-3'-amino- and 3:3'-di- ω -sulphomethylamino-4:4'-dihydroxyarsenobenzenes are suitable agents. Other known salts [cf. B.P. 3615 of 1912 (B., 1912, 844) and B.P. 214,237 (B., 1925, 691)] are ineffective against carcinoma. C. HOLLINS.

Manufacture of aralkyl esters of the *Hydnocarpus* fatty acids. I. G. FARBERIND. A.-G. (B.P. 311,236, 6.5.29. Ger., 7.5.28).—The fatty acids from varieties of *Hydnocarpus*, obtained by saponification, are converted by usual methods into aralkyl esters, which are stable, odourless, and pleasant-tasting oils valuable against leprosy etc. The benzyl esters, b.p. 180—230°/1 mm., of the acids of chaulmoogra oil, and the phenylethyl esters, b.p. 190—240°/1 mm., of the acids of *H. wightiana*, are described. C. HOLLINS.

Rendering irradiated ergosterol stable. A. HOME-MORTON. From F. HOFFMANN-LA ROCHE & Co. A.-G. (B.P. 334,002, 1.7.29).—Deterioration of irradiated ergosterol, as measured by the decrease in optical activity, is prevented by the addition of small amounts of phenols to the ergosterol or ergosterol solution before or after irradiation. E. H. SHARPLES.

Extracts of animal or vegetable organs or parts. SOC. CHEM. IND. in BASLE, W. MERKI, and P. SCHEIDEGGER (B.P. 333,159, 2.4.29. Addn. to B.P. 285,856; B., 1929, 150).—Animal organs generally (other than sex organs) or vegetable parts are cooled in solid carbon dioxide to -70° and pulverised before extraction with solvents. C. HOLLINS.

Preparation of active substances from hypophysis glands. I. G. FARBERIND. A.-G. (B.P. 305,475 and Addn. B.P. 333,155, [A] 4.2.29, Ger., 4.2.28, [B] 5.2.29).—(A) The whole gland, without separation of posterior and anterior lobes, is treated below 70° with acid (hydrochloric acid at 15° for 30 min.) or alkali (0.5N-sodium hydroxide at $35-40^\circ$ for 20 min.), the duration of treatment being varied according to the concentration of the reagent and the temperature; the hydrolytic agent is then neutralised, albuminates and globulinates are removed by centrifuging, and a solution of active substances is obtained which may be further purified by precipitation of ballast substances with acetone, the latter being then removed in a vacuum. The albuminates and globulinates yield a further quantity of active substances on repeating the hydrolytic treatment. (B) The whole gland is hydrolysed by fermentation, e.g., with papain or by autolysis, at a determined p_H , which is adjusted, if necessary, to precipitate ballast substances during or after hydrolysis. C. HOLLINS.

Surgical pads or the like. JOHNSON & JOHNSON (GREAT BRITAIN), LTD. From JOHNSON & JOHNSON (B.P. 333,980, 7.6.29).—Substances are incorporated with the pad, which, on exposure to radiant energy, cast a distinctive shadow or image on a radiograph or screen. E. H. SHARPLES.

Manufacture of inactive menthol. K. SCHÖLLKOPF and A. SERINI, Assrs. to RHEINISCHE KAMPFER FABR. GES.M.B.H. (U.S.P. 1,776,087—8, 16.9.30. Appl., [A] 29.6.27, [B] 30.6.27. Ger., [A] 15.12.26, [B] 15.2.27).—See B.P. 285,403 and 285,394; B., 1928, 873, 691.

2-Chlorobenzthiazoles (B.P. 310,815). Hydroxy-

1' : 8'-naphthoynlenenaphthiminazoles and derivatives (B.P. 316,143).—See III. Bismuth hydroxide (B.P. 332,504).—See VII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Amount of metallic silver in unexposed photographic emulsions, and formation of nuclei. H. ARENS and J. EGGERT (Z. wiss. Phot., 1930, 28, 178—190).—Repetition of Weigert and Lühr's experiments (B., 1930, 303) on the determination of the amount of metallic silver in an unexposed emulsion has yielded results suggesting that the method of analysis used is incapable of effecting a complete separation of primary silver from silver thiosulphate complex formed in development, the values obtained for the amount of the former thus being too high. The same conclusion is reached from a consideration of the amount of photo-silver obtained by extrapolating the mass of the latent image, and of the amounts of such substances as thiosinamine and methylene-blue required to affect the sensitivity. Finally, comparison of the rate of growth of the nuclei in the physical development of an ordinary photographic emulsion with that of an emulsion of colloidal silver of approximately the same degree of dispersity indicates that the amount of primary silver is much less than Weigert and Lühr's value.

R. CUTHILL.

Gases produced by the decomposition of nitro-cellulose and cellulose acetate photographic films. J. C. OLSEN, A. S. BRUNJES, and V. J. SABETTA (Ind. Eng. Chem., 1930, 22, 860—863).—The nature and quantity of gases evolved in the decomposition by heat of cellulose films in atmospheres of nitrogen and oxygen were studied. Details of analytical methods and results are given. Three highly toxic gases (carbon monoxide, nitrous fumes, and hydrocyanic acid) may be present. The origin and variation in amount of these with experimental conditions is discussed. The safety storage conditions for these materials are indicated. S. S. WOOLF.

Effect of temperature on the photographic plate. N. BARABASCHEV and B. SEMEJKIN (Z. wiss. Phot., 1930, 28, 221—228).—Density measurements on plates exposed at temperatures between 2° and —21° have shown that as the temperature of exposure falls the gradation of the plate becomes less.

R. CUTHILL.

PATENTS.

Film-forming element. H. E. VAN DERHOFF, Assr. to EASTMAN KODAK CO. (U.S.P. 1,746,751, 11.2.30. Appl., 21.8.25).—For the manufacture of films from dopes containing derivatives of cellulose a cylinder comprising a supporting metallic backing covered with a polished layer of nickel and an outer adherent layer of chromium is used. The chromium is polished to the degree required for the film surface. C. A. KING.

Matting a cellulose ester film for photographic purposes. E. LOMBERG (B.P. 308,358, 21.3.29. Ger., 22.3.28).—The rear side of the film is treated with cellulose ester solution containing starch as the matting agent.

J. W. GLASSETT.

Treatment of cellulose and like films with gas. CELLOPHANE SOC. ANON. (B.P. 332,631, 28.3.29. Fr., 21.2.29).—A band of gas-permeable paper or fabric

is wound up with the film in contact with the surface, thereby permitting treatment of the film surface with gas to be carried out in the rolled state, as, for example, in the development of diazotypes with ammonia gas.

J. W. GLASSETT.

Manufacture of photographic paper and films. FILM OZAPHANE (B.P. 318,511, 6.8.29. Fr., 4.9.28).—A cellulose film or a suitable support coated with a gelatin layer, which may be rendered matt by the addition of kaolin, is treated with a highly volatile solution of a light-sensitive diazo compound. The rapid volatilisation of the solvent prevents the penetration of the sensitive compound, which is thereby concentrated at the surface. A formula for a suitable sensitising solution is given.

J. W. GLASSETT.

Manufacture of sensitised velvet paper for photographic uses. D. FRIEDLÄNDER, Assr. to A. MEURSING and E. J. GRATAMA (U.S.P. 1,752,665, 1.4.30. Appl., 7.7.27. Ger., 7.7.26).—A well-sized paper support is covered with an adhesive medium not readily soluble in water and is then sprinkled with a fine fibrous dust, e.g., white wool powder. After drying, the excess dust is removed with a soft brush and the paper coated with the sensitive emulsion.

J. W. GLASSETT.

Transfer picture and its manufacture. E. HENTSCHEL (B.P. 331,761, 16.9.29. Ger., 18.10.28).—The support is coated with a thin layer of wax upon which the pictures are printed with a pigmented solution of a cellulose derivative, or, alternatively, the pictures are stamped out from a finished film of a cellulose ester and attached to the wax surface. A coating of an adhesive resin (dammar or mastic) is then applied.

J. W. GLASSETT.

Preparation of photographic diazotypes. F. VAN DER GRINTEN (B.P. 332,552, 20.4.29. Holl., 22.2.29).—The surface of the diazotype is sized during development by the incorporation of sizing agents such as sodium stearate, water-soluble casein, cows' milk, egg albumin, flour, etc. in the solution of the coupling agent. A further sizing may be given after development by treatment with a plain aqueous solution of these agents. Suitable working formulae for the process are given.

J. W. GLASSETT.

X-Ray screens. SCHERING-KAHLBAUM A.-G. (B.P. 318,152, 19.8.29. Ger., 28.8.28).—The fluorescent layer is coated directly upon a lead-glass plate.

J. W. GLASSETT.

Manufacture of [light]-sensitised element, and production of photographic images thereon. E. E. E. LEHMANN (U.S.P. 1,753,708, 8.4.30. Appl., 26.4.27. Ger., 27.4.26).—Diazotised *p*-aminodiphenylamine or a derivative is deposited upon a suitable carrier, exposed, and developed by coupling with β -naphthol solution to produce a negative, or, by treatment with an oxidising solution (dilute, acidified potassium dichromate solution) to give a positive.

J. W. GLASSETT.

Colour photography. W. V. D. KELLEY (U.S.P. 1,753,379, 8.4.30. Appl., 9.11.25).—One side of a doubly-coated film is exposed behind a red-selection negative, developed in acid or neutral diaminophenol, and then iron-toned to give a blue image. After washing, clearing, and drying, the same side is exposed under the green-selection negative and the reverse side under

the blue-selection negative. These are developed as before, bleached in a copper sulphate differential-hardening bath, fixed, and then dyed with magenta and yellow dyes, respectively, which are absorbed in the hardened parts. The yellow image may be obtained alternatively by an ammonium metavanadate bath.

J. W. GLASSETT.

Means for reproducing designs in metal by aid of photography. F. W. MILLER (B.P. 331,856, 9.4.29).—A glass plate coated with a mixture of dextrin and glucose or similar substances sensitised with ammonium dichromate is exposed behind a photographic positive of the design and then dusted with a metallic powder or other conducting medium which adheres to the unexposed parts of the surface only. After applying a coating of collodion and allowing it to set, the combined metallic coating and collodion film is floated off the glass plate and transferred, collodion film underneath, to a gelatin-coated support. Leads are attached to the metal picture, the whole is immersed in a plating vat, and a convenient thickness of a suitable metal is deposited on the original metal picture. The fine metal network is then stripped and pressed into a suitable support.

J. W. GLASSETT.

Preparation of light-sensitive layers. G. KÖGEL (U.S.P. 1,776,155, 16.9.30. Appl., 4.12.28. Ger., 13.12.27).—See B.P. 302,282; B., 1930, 303.

Production of [composite] photographic prints. SOC. TECH. D'OPTIQUE ET DE PHOTOGRAPHIE (S.T.O.P.) (B.P. 334,708, 3.9.29. Fr., 6.10.28).

Cellulose compositions of low inflammability (B.P. 312,609).—See V.

XXII.—EXPLOSIVES; MATCHES.

PATENTS.

Nitration of wood pulp and the like. V. PLANCHON (B.P. 333,817, 31.10.29. Fr., 18.1.29).—Sheets of wood pulp etc. are immersed a small distance apart in a nitrating bath kept at about 15° for the first 8–10 hrs. and then at 30° for a further 2 hrs.; collodion of low viscosity is produced.

H. ROYAL-DAWSON.

[Non-corrosive] priming mixture. J. D. McNUTT, ASST. to WINCHESTER REPEATING ARMS CO. (U.S.P. 1,755,330, 22.4.30. Appl., 23.3.27).—A mixture of mercury fulminate, barium nitrate, lead thiocyanate, ground glass, and a basic organic compound, such as diphenylamine, is claimed.

W. J. WRIGHT.

Explosive composition. F. OLSEN (U.S.P. 1,758,169, 13.5.30. Appl., 16.10.25).—A bursting charge for projectiles, which melts below 100°, consists of trinitrotoluene mixed with guanidine nitrate or picrate. The mixture detonates readily, but is sufficiently insensitive to shock to permit of its use in armour-piercing shells.

W. J. WRIGHT.

Combustible [compositions for tracer projectiles]. S. ST. P. MEEK (U.S.P. 1,756,255, 29.4.30. Appl., 8.9.25).—The use of mercury compounds, either as oxygen carriers, *e.g.*, mercurous nitrate and mercuric oxide, or as fuel, to decrease the rate of burning and increase the brilliance, *e.g.*, mercuric thiocyanate, or as inert substances, to increase brilliance, *e.g.*, mercurous chloride, is claimed.

W. J. WRIGHT.

XXIII.—SANITATION; WATER PURIFICATION.

Purification of water by electro-osmosis. E. BARTOW and R. H. JEBENS (Ind. Eng. Chem., 1930, 22, 1020–1022).—The purification of water by the removal of salts from a central section of a cell through diaphragms to anode and cathode cells has been developed in Germany. An apparatus for this purpose consists of a battery of 10 cells. The water in course of purification passes through the central compartment of each cell in turn, and the anode and cathode waters are removed by wash-water. The average voltage across the first four cells may be 27.5, increasing towards the end of the battery to 110 volts as the purification proceeds. Treatment of zeolite-softened water and regular tap water produced water of the same quality. Electrolytes were reduced from about 700 to 2.3 pts. per million, and the ash (chiefly silica) to 9 p.p.m. The cathode water contains hydroxide and is essentially lime water, and the anode water could be used as a bleaching agent. Water purified in this manner may substitute distilled water for most purposes, as the small residual content is chemically inert. The cost of production is said to be much lower than that of distilled water.

C. A. KING.

Removal of phenolic tastes in water supplies. R. D. SCOTT (Ohio Conf. Water Purif., IX Rep., 1929, 59–60).—The taste of chlorophenol was removed by addition of 3–5.7 or 1.8–3.3 pts. in 10⁶ of chlorine for 0.1 or 0.01 pt. of phenol in 10⁶, respectively. Taste probably due to vegetation etc. was not removed by 10 pts. of chlorine in 10⁶. CHEMICAL ABSTRACTS.

Control of phenol tastes [in water] by means of increased lime treatment. J. S. SCOTT (Ohio Conf. Water Purif., IX Rep., 1929, 61–63).—Excess of lime removes phenolic tastes from chlorinated water.

CHEMICAL ABSTRACTS.

Ammonia-chlorine process [for water] as a means for taste prevention and effective sterilisation. M. M. BRAIDCH (Ohio Conf. Water Purif., IX Rep., 1929, 67–83).—Pre-ammoniation prevents rather than destroys phenolic tastes and odours, chloroamine being produced.

CHEMICAL ABSTRACTS.

Maintaining chemical balance to resist corrosion, and its application to a recent development in water softening with lime. C. P. HOOVER (Ohio Conf. Water Purif., IX Rep., 1929, 96–99).—An indicator test, and a process whereby the carbonate hardness is reduced by means of lime approximately to the solubility limit of the calcium carbonate and magnesium hydroxide, are described.

CHEMICAL ABSTRACTS.

Fermentation of cornstalks. BORUFF and BUSWELL.—See XVII.

PATENTS.

Water softener. S. S. SADTLER (U.S.P. 1,761,411, 3.6.30. Appl., 10.1.27).—A small amount of an indicator, *e.g.*, phenolphthalein or a chrome-blue, is added to the softening reagent, *e.g.*, sodium bicarbonate, to indicate when the desired degree of alkalinity is reached for domestic or laundry purposes.

C. JEPSON.

De-aeration and evaporation of liquid (U.S.P. 1,758,566).—See I. **2-Chlorobenzthiazoles** (B.P. 310,815).—See III. **Base-exchange material** (U.S.P. 1,756,623–5). **Glauconite for water softening** (U.S.P. 1,757,373).—See VII.