

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

JAN. 2 and 9, 1931.\*



### I.—GENERAL; PLANT; MACHINERY.

**Mechanical methods of dust collection.** M. A. LISSMAN (Chem. and Met. Eng., 1930, 37, 630—634).—The theory of cyclone dust separators is considered and mathematical expressions are deduced. D. K. MOORE.

**Friction of dry solids *in vacuo*.** P. E. SHAW and E. W. L. LEAVEY (Phil. Mag., 1930, [vii], 10, 809—822).—Measurements have been made *in vacuo*, by the tilt method, using every possible combination of the following materials: aluminium, silver, copper, nickel, iron, and soda-glass. After repeated heating and cooling in a vacuum, the friction rises to a value which is practically constant between 15° and 350°, except in two or three instances where there is a pronounced positive or negative temperature coefficient. The coefficient of friction,  $\mu$ , is much higher in a vacuum, especially for light loads, than when the materials are treated in the open air, and rises until complete annealing has taken place. The theory of the subject is discussed. M. S. BURR.

**Condensation of vapours.** C. C. MONRAD and W. L. BADGER (Ind. Eng. Chem., 1930, 22, 1103—1112).—The recent increase in liquor velocities in evaporators and condensers has rendered the study of heat transfer through the vapour film of greater importance than it was formerly. The theoretical work of Nusselt (Z. Ver. deut. Ing., 1916) is described. He obtained equations for the heat-transfer coefficient in the cases of (1) vapour condensing on a smooth plane surface making an angle with the horizontal; (2) vapour condensing on the outside of a horizontal tube; (3) as in the first case, but the vapour having appreciable velocity. He also investigated the effect of superheat and the presence of non-condensable gases with less completely satisfactory results. Experimental data obtained by various investigators are compared with Nusselt's equations, and the limitations of the latter are discussed. Whilst good agreement is usually obtained with horizontal tubes, discrepancies occur with vertical tubes owing to the effect of turbulence and the formation of drops; this will usually occur under industrial conditions. Nusselt's calculations on the effect of vapour velocity and superheat, again, are sound when turbulence is absent in the condensate film. C. IRWIN.

**Automatic gas-analysis apparatus dependent on the thermal conductivity of gases, and its industrial use.** P. JARRIER (Chaleur et Ind., 1930, 181; Fuel, 1930, 9, 458—462).—Gas-analysis apparatus based on the thermal conductivity of the gas, *e.g.*, the Siemens-Halske CO<sub>2</sub> recorder, offers certain facilities for the transmission of the readings to a distance or for

automatic regulation. These instruments are applicable to certain mixtures of two or more clean, dry gases, but are less generally applicable to the flue gases from industrial furnaces. The possible sources of error when they are so used, *e.g.*, contamination of the wires, the presence of water vapour or combustible gases, etc., are discussed. To obtain reliable readings constant attention to the filters and connexions is necessary, and the electric current and water current must be exactly regulated. A. B. MANNING.

**"Dry-ice."** KILLEFFER.—See VII. **Firing of boiler furnaces.** FREISE.—See VIII. **Measurement of liquid consistency.** FAWKES. **Photo-electric process control.** STYER and VEDDER; McMASTER.—See XI. **Universal thermostat.** FINK.—See XVIII. **Protection against toxics.** TURNER.—See XXIII.

### PATENTS.

**Heat-exchanging apparatus for fluids.** C. A. HUBERT (B.P. 337,236, 22.11.29).—The apparatus comprises a number of ribbed tubes, so stacked that the ribs come in exact vertical rows in order that cleaning jets may be effective when required.

B. M. VENABLES.  
**Tubular heat exchangers.** J. HOWDEN & Co., LTD., and J. H. HUME (B.P. 336,755, 21.9.29).—An exchanger comprising a number of tubes arranged in diagonal lines is provided with metallic filling between the tubes in one diagonal direction, so that the outer fluid flows in parallel sinuous paths and makes contact with a larger surface of metal. B. M. VENABLES.

**Reduction of temperature [of air] by dehydration.** W. L. FLEISHER, ASST. to COOLING & AIR CONDITIONING CORP. (U.S.P. 1,749,763, 11.3.30. Appl., 8.8.28).—The apparatus comprises an inlet mixing chamber, a silica gel dehydrator, a cooling chamber, a spray chamber, an outlet mixing chamber, and a fan which delivers the air to the enclosure to be ventilated. The entering air is dehydrated to a very low dew point by the gel, and the latent heat of absorption is removed by coils cooled by an abundance of water, so that in the cooling chamber the air attains a moderately low dry-bulb and very low wet-bulb temperature. In the spray chamber a limited quantity of water is used over and over again, except for a little make-up, so that the temperature of the water and the actual or dry-bulb temperature of the air both become reduced to the wet-bulb temperature. In cases where it is desirable to recirculate air from the enclosure, part will be returned direct to the outlet mixer and the other part with some fresh air will enter the inlet mixer for treatment.

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



The proportion treated will be under the control of a dry-bulb thermostat placed in the return conduit; another thermostat with a wet bulb regulates the cooling water in the coil. B. M. VENABLES.

**Thermostats.** R. MACLAREN (B.P. 337,093, 10.8.29. Addn. to B.P. 307,135; B., 1929, 496).—A bimetallic thermostat as described in the prior patent is provided with a modified switch movement incorporating a magnetic blow-out. B. M. VENABLES.

**Drying of material.** L. N. LLOYD, and PHILLIPS ENG. CO., LTD. (B.P. 336,602, 9.5. and 3.8.29).—The material is spread in a thin layer upon the uppermost storey of a series of supports each comprising a number of tray-like devices adapted to discharge, at intervals, the whole of the load on one storey on to the next below. The trays are heated by internal or closely adjacent means, and the feed and discharge devices operate through air-locks so that drying may be effected in a vacuum. B. M. VENABLES.

**Rotary drying apparatus.** J. B. VERNAY (B.P. 336,874, 19.12.29. Fr., 21.12.28).—The material is passed in a zig-zag manner through a number of co-axial shells, the innermost of which is stationary and is provided with a heating jacket, whilst the others are rotated. A certain amount of grinding is effected during the drying, and air-locks are provided so that a vacuum may be applied. B. M. VENABLES.

**Spray dryer.** W. F. RICHARDS (B.P. 337,080, 2.8.29).—The spraying chamber is surrounded by a number of compartments containing bag filters; the inlet sprays of liquid are situated in the upper circumference, the inlet of drying gases is upwards from the bottom centre, and the passages to the filters are along the floor under the circumference of the inner chamber, which has a roof adjustable in height to vary the drying space. B. M. VENABLES.

**Crushing and grinding of solids.** M. P. APPLEBEY, F. POSTLETHWAITE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 336,730, 29.8.29).—Crushed material, *e.g.*, coal, is removed from a jaw crusher by means of upward currents of air or other gas as fast as it is ground. B. M. VENABLES.

**Reducing friable materials.** G. C. E. KEET (B.P. 336,687, 24.7.29).—The material is passed between rolls which are convex and concave or V-shaped, and the distance between their axes is continuously varied by means of a toggle mechanism. B. M. VENABLES.

**Disintegrating machines particularly applicable to rubber.** INTERNAT. PULVERISING & GRINDING MACHINES (PARENT) CORP., LTD., and P. LEGRAND (B.P. 336,828, 7.11.29).—Forms of disc grinders suitable for rubber are described, the discs of which are provided with rasp teeth. B. M. VENABLES.

**Pulverising mills.** BABCOCK & WILCOX, LTD. FROM FULLER LEHIGH CO. (B.P. 336,787, 14.10.29).—The machine is of the ball-mill type with hollow trunnions, the feed being through one trunnion and the discharge air-borne through the other trunnion; adjacent to the latter is a classifier for separation of oversize, which is returned to the mill through the same trunnion by means of a screw or other type of conveyor. B. M. VENABLES.

**Grinding, crushing, and mixing mills.** J. WASS (B.P. 336,212, 6.7.29).—An edge-runner mill, preferably with a conical bottom and correspondingly shaped roller, is provided with means by which the roller can be lifted out of action so that mixing can continue by means of stirring blades alone. B. M. VENABLES.

**Centrifugal machine for clarifying and purifying liquids.** RAMESOHL & SCHMIDT A.-G. (B.P. 336,865, 10.12.29. Ger., 10.12.28).—A machine suitable for separating two liquids (also solids), when the heavier liquid is desired of maximum purity, is described. There are three concentric separating zones, and the heavier liquid finally leaves inwards through a filter. Application to cleaners' solvent liquid is indicated. B. M. VENABLES.

**Centrifugal machine.** C. and F. GREAVES (B.P. 336,743, 12.9.29).—A method of feeding the material to, and supporting the inclined axes of, the planetary baskets of a centrifugal machine is described. B. M. VENABLES.

**Sterilisation of liquids by heating.** N. J. NIELSEN (B.P. 337,027, 27.6.29).—The liquid to be treated, *e.g.*, milk, is heated while under pressure by heat exchange with another liquid, *e.g.*, water, which is also under a pressure sufficient to prevent the formation of steam or vapour. The water flows in a continuous circuit: from a reheater to an exchanger, where it flows countercurrent to the arriving milk, then through another exchanger countercurrent to the departing milk (which is thereby cooled), back to the reheater. B. M. VENABLES.

**Apparatus for the hydrogenation of liquids, solids in suspension, or gases.** TECHNICAL RES. WORKS, LTD., and E. J. LUSH (B.P. 336,569, 11.7.29).—Forms of apparatus are described in which the catalyst, consisting of nickel wire or turnings activated by anodic oxidation, is supported in masses not materially exceeding 6 in. thick. The cages and container may be formed of copper or a copper-nickel alloy. B. M. VENABLES.

**Dust filters.** W. M. ODDIE (B.P. 337,304, 30.1.30).—Filter bags, supported at the top by individual chains, have inlets at the bottom in the form of inverted truncated cones within which are heavy correspondingly shaped plugs supported by a continuation of the chains. When a bag is in the operative stretched position the plug valve will be open, but when the suspending chain is slackened the inlet will be closed and the bag simultaneously given a jolt. On restretching the bag the dislodged dust will slide out through the inlet cone. B. M. VENABLES.

**Deposition of dust or the like from an atmosphere in which it is suspended.** WOODALL-DUCKHAM (1920), LTD., and A. MCD. DUCKHAM (B.P. 336,201, 6.6.29).—Air containing dust, *e.g.*, coal from a pneumatic separator, is repeatedly circulated round a depositing vessel, and sprays of steam or other suitable liquid, gas, or vapour are admitted countercurrent to the air so that the density of the solid particles is changed relatively to the air, both by coagulation and by admission of a gas (steam) of lower density than the air. B. M. VENABLES.

**Separation of dust from air or gases.** G. RAW (B.P. 336,221, 4.7.29).—Dust, *e.g.*, coal dust from a



pneumatic separator, is coagulated by admission of a mist of liquid to the air while in a substantially stagnant state; the mist should be too fine to settle except in combination with the dust, and the quantity of liquid should be so small that the moisture is practically imperceptible. B. M. VENABLES.

**Extraction of dust and grit from air, flue gas, or other medium.** PNEUMATIC CONVEYANCE & EXTRACTION (1929), LTD., and W. A. SMITH (B.P. 336,620, 17.6.29. Addn. to B.P. 256,063).—The air to be cleaned is passed through an involute passage, entering at the point of largest radius, which has a V-shaped outer wall and a slit along the apex of the V for the outlet of dust concentrated in a small quantity of air to a surrounding casing acting as a settling chamber. A pair of dust extractors may be attached to the twin inlets of an extraction fan having by-passes to transmit the reduction of pressure to the settling chambers also. B. M. VENABLES.

**Liquefaction of air and other gases.** M. HAZARD-FLAMAND (B.P. 336,798, 17.10.29. Fr., 18.10.28).—The air after compression to 200 atm. is passed through one side, say the inner, of three heat exchangers in series, and a part of it also through a fourth, after which the same portion is expanded through a constriction without doing work, and thus is partially liquefied. The unliquefied gas is passed backwards through the outer sides of the fourth and second exchangers, together with the other portion of gas that was tapped off between the third and fourth exchangers, which portion has been expanded while doing external work. The first exchanger is cooled by water, and the third by the vaporisation of liquefied ammonia. A purifier may be placed between the first and second exchangers. B. M. VENABLES.

**Gas-liquefying plant.** A.-G. BROWN, BOVERI & Co. (B.P. 337,325, 22.2.30. Ger., 18.3.29).—A centrifugal compressor has its bearings lubricated by the liquefied gas. B. M. VENABLES.

**High-vacuum vapour pumps.** W. GAEDE (B.P. 337,214, 11.11.29. Ger., 13.11.28).—A pump of the diffusion type is described. The diffusion passage is cooled externally by a jacket and internally by a coil, and any mercury condensed on the latter is prevented from dropping into the hot mercury jets by a hood. B. M. VENABLES.

**Manufacture of vessels and other structures that are proof against chemical and atmospheric influences.** P. PICK (B.P. 316,134, 4.7.29. Ger., 23.7.28).—Constructional elements are formed from perforated metallic plates covered with one or more layers of plastic material which is unvulcanised (but capable of being afterwards vulcanised), the sandwich produced being hydraulically pressed while subjected to a vacuum. The metal plate may have stiffening ribs pressed out of the plate, but they should not extend to the edges, and the plastic material may be in layers of different hardness indissolubly welded and having the air removed by the pressure and vacuum. B. M. VENABLES.

**Apparatus for carrying out chemical reactions.** BRIT. CELANESE, LTD. (B.P. 310,415, 15.4.29. U.S., 25.4.28).—A mixing vessel with fixed and moving vertical blades is described. B. M. VENABLES.

**Photometers.** HOLOPHANE, LTD., and H. S. ALLPRESS (B.P. 336,897, 20.1.30).—The instrument is suitable for expressing the illumination inside a room as a proportion of that of the sky. B. M. VENABLES.

**Measuring the temperatures of incandescent bodies.** KAISER-WILHELM INST. F. EISENFORSCHUNG (B.P. 336,296, 12.7.29. Ger., 31.10.28).—An optical pyrometer is provided with a parallel-faced filter which cuts off all light except red and green (or another pair of selected colours), and with a wedge-filter which permits one of those colours, e.g., red, to pass through freely, but cuts off the other, green, in proportion to the thickness of the wedge. To measure the temperature, the wedge is adjusted so that a whitish mixed colour is transmitted; then should the observed body become hotter the light will become green, or if cooler red. This method is stated to be less affected by the nature of the emitting surface than are those which depend on the total radiation. B. M. VENABLES.

**Measurement of humidity.** CAMBRIDGE INSTRUMENT Co., LTD., J. L. ORCHARD, and H. T. GLOVER (B.P. 336,855, 2.12.29).—A pair of equal coils of wire forming resistance thermometers are placed close together in a gas stream, one being kept dry and wholly exposed to the gas, the other being wholly covered with fabric which is kept wet. They form two arms of a Wheatstone bridge, and electrical devices are combined with the other two arms of the bridge to compensate for variations from standard of the dry-bulb temperature, so that the galvanometer may be calibrated to read directly in percentage of relative humidity. B. M. VENABLES.

**Apparatus for heat treatment of liquids.** W. WINSHIP (B.P. 337,762, 3.5.29).—See U.S.P. 1,712,372; B., 1929, 579.

**Testing [the physical properties of] materials.** R. ESNAULT-PELTERIE (U.S.P. 1,781,002, 11.11.30. Appl., 4.4.27. U.K., 4.2.27).—See B.P. 289,960; B., 1928, 553.

**Conservation of heat in a steam power plant.** STEAM HEAT CONSERVATION Co., LTD., H. P. H. ANDERSON, and J. McCULLUM (B.P. 336,599, 13.4.29).

**Absorption refrigerating apparatus.** G. BEAUMONT (B.P. 337,912, 30.9.29).

**Refrigerating apparatus of the absorption type.** A. SILBERNAGEL (B.P. 337,964, 1.11.29. Switz., 12.11.28).

**Absorption machines for refrigerating or heating.** SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 317,052, 6.8.29. Ger., 9.8.28).

**Safety devices for hydro-extractors.** D. K. and A. F. TULLIS (B.P. 337,670, 20.12.29).

**Apparatus for automatically discharging the centrifuge drum of a continuously operating high-speed centrifuge.** K. Egg (B.P. 337,970, 5.11.29).

**[Manufacture of segmental] grinding wheels.** CARBORUNDUM Co., LTD. From CARBORUNDUM Co. (B.P. 336,631, 17.7.29).



**Appliances for viewing fluorescent and phosphorescent substances [for advertising purposes].**

L. A. LEVY (B.P. 336,317, 19.7. and 4.12.29).

**Manufacture of sheets, films, etc.** (B.P. 315,840 and 318,250).—See V. **Tunnel kilns** (B.P. 336,171).—See VIII. **Zinc alloys for bearings** (B.P. 336,881).—See X.

**II.—FUEL; GAS; TAR; MINERAL OILS.**

**Transformation of fatty acids during geological periods.** IV. G. STADNIKOV and Z. VOSSCHINSKAJA (Brennstoff-Chem., 1930, 11, 414—416. Cf. Stadnikov and Weizmann, B., 1929, 1002).—"Balkashite," a sapropelite from Lake Balkash (cf. Zelinski, B., 1926, 226), has been formed by the oxidation, polymerisation, and dehydration of the fats and fatty acids of a green alga, *Botryococcus Braunii*, K., which grows in enormous quantities in the lake. Chemical and microscopical examination shows it to be closely related to the Siberian and Moscow boghead coals, and to resemble the saponifiable fraction of coorongite. A. B. MANNING.

**Significance of spores in the correlation of coal seams.** I. **The Parkgate seam—South Yorkshire area.** L. SLATER, M. M. EVANS, and G. E. EDDY (Dept. Sci. Ind. Res., Fuel Res., Phys. and Chem. Survey of Nat. Coal Resources No. 17, 1930, 28 pp.).—The object of the investigation was to determine whether a single spore type, by its presence in any part of a seam, or by its concentration in certain parts, or the distribution of the spores as a whole, is characteristic and would therefore serve as a basis for the correct identification and correlation of different coal seams. The major portion of the Parkgate seam is composed of three bands, viz., "tops," "middle coal" or "hards," and "bottoms"; the tops and bottoms are both bright in appearance, consisting mainly of clarain, whilst the middle coal consists essentially of durain. Three pillar samples (i.e., seam sections) of Parkgate coal were taken from different districts, and a series of thin sections, cut at right angles to the bedding plans, were prepared; the whole area of each thin section was examined carefully and the spore exines were classified. It was found that (a) spore remains were more numerous in the hards than in the tops or bottoms; (b) in certain thin sections, principally in the hards, they were more numerous than in others; (c) most of the megaspore types recorded were present in all three pillar samples; (d) only certain types of megaspores showed a similarity of distribution in each pillar sample; (e) certain of the remaining types were concentrated in a similar manner in two of the pillar samples, but in the third showed considerable variation either in a part or throughout the whole of the sample. It is suggested that despite such irregularity of distribution, the presence of these spores may be used for correlation purposes.

C. B. MARSON.

**Some aspects of the problem of cleaning Witbank coal.** I. J. BLOM and T. J. W. JORDEN (Fuel, 1930, 9, 464—476).—Five samples of coal from the Witbank deposits were crushed and screened, and each of the sized fractions (0.4 mm., 0.4—1.0 mm., 1—2 mm., 2—3 mm., 3 mm.) was submitted to a float-and-sink

analysis. No sharp bends were observed in the washability curves, which were examined from the viewpoint of dividing the coals into three fractions: (1) coking coal of normal ash content, e.g., 7%; (2) steam coal of high ash content, e.g., 13—14%; and (3) waste. The ash content of the raw coals varied from 9.7 to 11.8%. An examination of the float-and-sink data by Bird's method (2nd Internat. Conf. on Bit. Coal, 1928, 2, 82) indicates that the separation at 7% ash content would be very difficult in an ordinary washing plant, but could probably be effected by means of the Lessing Rational Coal-Cleaning Plant (*ibid.*, 1928, 2), after removal of the dust which would be added to the steam coal. The relation between the ash content and the sp. gr. of the fractions of each sample of coal is roughly linear. Caution is necessary, however, in applying Lategan's method of determining the ash content (B., 1927, 833) to coal-cleaning operations; it may become accurate enough for practical purposes if a "proportionality curve" is determined for each separate colliery and is used only between the limits within which it has been shown to be valid. The variation in the sulphur and nitrogen contents of one of the coals (T. & D.B.) has been studied. The sulphur content at first falls rapidly with decreasing sp. gr. of the separated fractions up to a point corresponding with 9.5% of ash, due probably to the removal of pyrites, and thereafter remains more or less constant at 0.7—0.8%. The nitrogen content increases in steps as the ash content falls, corresponding probably with the successive removal of the fusain and durain as the washing proceeds. An examination of the ash composition curves shows a change in the nature of the inorganic constituents with increasing ash content of the floated material at the point corresponding with 8% of ash for Oogies coal and 9—10% for Douglas coal. It is concluded that these values represent the "inherent ash" of the coals (cf. Mott and Wheeler, B., 1927, 802). A. B. MANNING.

**Oxidation of mineral coals in an atmosphere of oxygen.** Y. O. GABINSKI (Sci. Mag. Met. Cath. Dnepropetrovsk, 1929, 1, 199—207).—Curves showing the loss in weight of coals as a function of the time of exposure to oxygen are characteristic for the particular coals. The reduction of cokability due to treatment with oxygen is a qualitative measure of the stability of the coal. CHEMICAL ABSTRACTS.

**Determination of cokability of U.S.S.R. coals by Meurice's method.** Y. O. GABINSKI (Sci. Mag. Met. Cath. Dnepropetrovsk, 1929, 1, 177—198).—Concordant results were obtained with Russian coals. CHEMICAL ABSTRACTS.

**Coking of Kuznetz coals (Kemerovo, Siberia).** P. G. RUBIN and Y. O. GABINSKI (Sci. Mag. Met. Cath. Dnepropetrovsk, 1929, 1, 33—79).—Unlike Donetz and most European coals, Kuznetz coals exhibit a cokability which increases with an increase in the amount of volatile matter. CHEMICAL ABSTRACTS.

**Cokability and stability of Donetz coal.** P. G. RUBIN and Y. O. GABINSKI (Sci. Mag. Met. Cath. Dnepropetrovsk, 1929, 1, 81—124).—Good coking coals gave a Meurice value of at least 120, medium 70—100, and poor less than 70. The stability ratios (of Meurice



value of original coal to that after exposure to oxygen for 6–7 days) were, respectively, 100, 80–90, and 50–60%.

CHEMICAL ABSTRACTS.

**Dehydrating action of coal ash.** M. TANAKA (Rep. Lab. S. Manchuria Rly., 1929, 36–37).—The composition of the ash of coal and oil shale has been found to resemble that of acid clay. When applied to the synthesis of anthraquinone derivatives the dehydrating powers of Fushun coal ash and Japanese acid earth were very similar, being fairly active at 260°, although slightly inferior to that of anhydrous aluminium chloride.

H. J. DOWDEN.

**Determination of fusain.** F. HEATHCOAT (Fuel, 1930, 9, 452–458).—Fusain is determined by a method depending on its inertness towards oxidising agents. The dry and powdered coal sample is refluxed with Schultze's solution (3 g. of potassium chlorate in 100 c.c. of 2*N*–4*N*-nitric acid), filtered through a fritted glass crucible, washed, transferred to a beaker, boiled for 1 hr. with *N*/6-sodium hydroxide, filtered, washed, dried, and weighed. The ulmin material and the organised plant remains are completely oxidised and removed by this treatment. The result is low owing to the partial conversion of the fusain into alkali-soluble products, but from an examination of a series of coal dusts and slurries it appears that, in general, the errors arising from the oxidation of fusain are not serious. The method possesses decided advantages over that based on the microscopical examination of the particles. The examination of the sieved fractions of a hand-picked "fusain" shows that the estimated fusain content is higher in the finer sizes than in the coarser.

A. B. MANNING.

**Determination of total acid in peat.** E. SAUER and H. J. STEINMETZ (Kolloid-Z., 1930, 53, 218–224).—An excess of calcium bicarbonate solution is added to the peat and the excess is titrated after reaction is complete. The method gives more reliable results than are obtained by adding calcium carbonate and determining the liberated carbon dioxide. The accuracy of the method increases with the degree of dispersion of the peat, and the material is best dried and ground in a porcelain ball-mill. The presence of neutral salts does not affect the results. Several kinds of peat examined showed a remarkable agreement in total acidity.

E. S. HEDGES.

**Comparison of recent methods of determining the agglutinating power of bituminous coals.** G. AGDE and A. WINTER (Brennstoff-Chem., 1930, 11, 394–396).—The agglutinating values of mixtures of a caking coal and anthracite have been determined by the following methods: Meurice-Kattwinkel (B., 1926, 207, 905), Meurice-Gabinsky (Mitt. Lehrst. Eisenhütten- u. Brennstoffkunde Dnepropetrovsk, 1929, 1, 177), and Marshall-Bird (cf. Johnson and Yancey, B., 1930, 1052). Each method gave results for the different mixtures which were proportional to those of the other methods. This proportionality failed, however, when coals of different type were compared. The methods of Kattwinkel and of Gabinsky may fail with some coals, due to segregation of the sand during the heating. The Marshall-Bird method is the most reliable, giving less

variation from the mean value with repeated determinations.

A. B. MANNING.

**Plastic range of coking coals.** W. DAVIDSON (Fuel, 1930, 9, 489–492).—The plastic ranges of 12 coking coals have been determined by a modification of Foxwell's method (B., 1924, 4 *seq.*). Whilst the beginning of the plastic range did not vary with the rate of heating (from 1–5°/min.), the extent of the range increased with increased rate of heating. The volatile matter evolved before the beginning of the plastic range and that evolved during the plastic range were determined separately. There is a distinct relationship between the nature of the coke obtained and the percentage of volatile matter evolved subsequent to the plastic range, and it is considered probable that if the volatile matter retained by a coal at the end of the plastic range could be diminished, *e.g.*, by adding a material of low volatile matter content, or a coal having a higher temperature and lower volatile content at the end of the plastic range, a coke having less fissures would be obtained. This supposition was confirmed by blending a Scottish coal with finely-ground high-temperature coke, whereby a coke of increased size and resistance to shatter was obtained.

A. B. MANNING.

**Production of strong coherent coke by the low-temperature carbonisation of brown coal mixed with tar.** C. STAEMMLER (Brennstoff-Chem., 1930, 11, 413–414).—Mixtures of brown coal with a brown-coal generator tar in the ratio 1:1 were carbonised in a small aluminium retort, but failed to give a coherent coke. The character of the coke was greatly improved by preoxidising the tar with air, preferably in the presence of a catalyst, *e.g.*, manganese or lead dioxide, but even under these conditions the process was uneconomical.

A. B. MANNING.

**Possibilities of the utilisation of coke fines and its value in Upper Silesia.** P. DAMM and F. WESE-MANN (Stahl u. Eisen, 1930, 50, 1495–1500).—Under the economic conditions in Upper Silesia, the value of coke fines for use in gas producers or as an addition to coking coals is shown to be small, but the fines are of considerable value for firing boilers, for use as a reducing agent in zinc smelters, and, above all, for briquetting. The economics of the use of coke dust for these purposes is discussed in considerable detail.

A. R. POWELL.

**Utilisation of coke breeze by the Trefois producer.** A. L. HOLTON and H. C. APPLEBEE (Gas J., 1930, 192, 151–158).—The producer is fitted with rotary grate and water jackets, and has a daily capacity of 10–12 tons of fuel, making about 1½ million cub. ft. of gas at 115–130 B.Th.U./cub. ft. In its original form clinker troubles were severe, and necessitated allowance for a lower repose angle of the ashes and better arrangements for breaking clinker. All types of coke breeze, except that entirely passing a ½-in. screen, have been used; no difficulties were experienced with fuels having high moisture contents, but dust and tar deposits were sometimes found. Full details are given for a number of tests, in England and abroad, showing a gas-making efficiency as high as 88.5%. Control of the machine is easy and its flexibility considerable. The application of the producer to the heating of horizontal retorts has been



achieved, and the advantages arising from its use are discussed.

R. H. GRIFFITH.

**Friability of mid-temperature cokes.** S. W. PARR and D. R. MITCHELL (Ind. Eng. Chem., 1930, 22, 1134—1135).—One-ton charges of Illinois, Iowa, and West Virginia coals were prepared in an experimental plant, using a standard 12-in. oven operated according to the Parr process, the special characteristics being quick carbonisation and use of the mid-temperature range; all the coals were preheated at about 300° and then charged hot into the coke oven, the coking temperatures ranging from 750° to 900°. Shatter tests on the resulting cokes showed that the shatter index (2 in.) varied from 57 to 79, that shortening of the preheating period slightly increased the strength of the coke, and that an increase in coking time effected an improvement in the quality of the coke. It is concluded that mid-temperature cokes are somewhat tougher than cokes made by high-temperature processes; cokes made at 750–900° contain 4–5% of volatile matter and are very desirable as domestic fuel, being smokeless, of low ignition temperature, and of high physical strength.

C. B. MARSON.

**Reactivity of metallurgical coke.** M. DUFRANE (Rev. Mét., 1930, 27, 509–511).—Reactivity of fuel is defined by the extent to which the reaction  $C + CO_2 = 2CO$  proceeds in the forward (endothermic) direction. High reactivity of blast-furnace coke is a cause of irregularities in working the furnace. Samples of coke from three sources have been studied at 800°, 900°, 1000°, and 1100°. The reactivity increases rapidly with rise in temperature. Treatment of a very reactive coke in a slurry of lime or cement is found to reduce the reactivity at all temperatures to a value below that of the other (untreated) materials; the compressive strength is raised in consequence.

E. H. BUCKNALL.

**Gas-, coke-, and by-product-making properties of American coals and their determination.** A. C. FIELDNER, J. D. DAVIS, and D. A. REYNOLDS (Ind. Eng. Chem., 1930, 22, 1113–1123).—An experimental carbonising apparatus, taking a charge of 75–100 lb. of coal, is described and the results obtained with two gas coals which had previously been tested under full-scale conditions are discussed. Tests were carried out at temperatures between 500° and 1100° and the yields and quality of coke, gas, tar, light oil, and liquor determined. Close agreement between the experimental and full-scale methods was obtained in the case of gas, coke, and tar yields, but the experimental method gave lower ammonium sulphate yields, which is attributed to the catalytic effect of the iron retort in decomposing ammonia. The experimental coke had approximately the same volatile matter, cell space, apparent  $d$ , and resistance to abrasion as full-scale coke made from the same coal at the same temperature of carbonisation, and was characterised by a lower shatter index; reasons are advanced for this difference. It is believed that shatter and tumbler tests will give relative values for comparing different coals and coal blends in the experimental apparatus. The first coal examined gave the strongest coke, as judged by shatter and tumbler tests, at carbonising temperatures of 800–900°; the second coal

gave the strongest coke at 600°, and it is suggested that mid- and low-temperature processes, respectively, are therefore suitable for these two coals. Using this method of carbonisation, the carbonising properties of a coal may be correlated with its constitution etc., and results of the following tests on the two coals examined are given: softening and decomposition temperatures, agglutinating value, plastic range, low-temperature assay, oily and solid bitumen contents.

C. B. MARSON.

**Comparison of acetylene black with gas black and lampblack.** T. R. DAWSON and, in part, N. H. HARTSHORNE (Trans. Inst. Rubber Ind., 1930, 5, 48–70).—Analyses of samples of the three blacks are given and comparisons made as to their colour, pigmenting power, bulkiness, sp. gr., and particle size. The acetylene black is chemically the purest and the lampblack the least pure. All three types of black retard the vulcanisation of a rubber-sulphur mixture. In tensile strength and reinforcing power, although possibly not in permanent set and resilience, gas and acetylene blacks approach one another more closely than they do lampblack, this resemblance extending even to the abnormal discontinuous course of vulcanisation observable with mixtures containing either of the former blacks, together with hexamethylenetetramine. The degree of variability in acetylene black is of the same order as the difference between acetylene black and gas black, but is markedly smaller than the difference between acetylene black and lampblack.

D. F. TWISS.

**Composition of carbonised molasses waste.** W. KILP (Z. Spiritusind., 1930, 53, 305).—Carbonised molasses waste consists essentially of potassium carbonate, chloride, and sulphate, sodium carbonate, and insoluble matter, with a small proportion of moisture. The composition varies according to the stage to which carbonisation has been taken, and black-coloured char may contain over 60% of insoluble matter and approx. 25% of soluble alkali salts, whereas one incinerated more thoroughly may yield over 80% of soluble alkali salts of which 90% consists of potassium salts. The char from beet molasses has a higher content of potassium salts than that from sugar cane, but the latter contains a higher percentage of insoluble matter, owing to its high content of phosphate, silicate, and lime.

C. RANKEN.

**Avoidance of naphthalene troubles [in coal gas].** F. SCHUSTER (Gas- u. Wasserfach, 1930, 73, 1009–1015).—The development of the use of tetralin for removal of naphthalene from coal gas is reviewed, and a system for introduction of tetralin vapour into the gas is described. This is achieved either by supply from a boiler or by atomisation of the liquid. In both cases sufficient tetralin is carried in the gas to prevent deposition of solid; liquid condensate is removed at intervals. The process costs 1–2 pf. for 100 m.<sup>3</sup> of gas, and completely eliminates blockage of services.

R. H. GRIFFITH.

**Formation of copper carbide in acetylene cylinders.** G. F. KOMOVSKI (J. Appl. Chem., Russia, 1930, 3, 401–411).—Pure, dry acetylene does not combine with copper or its alloys. Appreciable quantities of cuprous acetylide are formed only on prolonged contact of



compressed technical acetylene with pure copper; it decomposes in air at 120–123°, in acetylene at 130–150°, or when heated in air at 50° and then introduced into acetylene. In acetylene at 4–5 atm. decomposition occurs at 200–251°. CHEMICAL ABSTRACTS.

**Causes of explosion [in gas holders].** H. WOLF (Chem.-Ztg., 1930, 54, 796).—Deposits of iron sulphide produced by the action of the sulphur compounds in the gas on the metal of the gas holders may ignite spontaneously on contact with air, and constitute a source of danger if the holder contains an explosive mixture. Thin aluminium foil or aluminium bronze powder in contact with air or oxygen may be ignited by the action of a bright light. A. B. MANNING.

**Problems relating to coal tar.** W. J. CHADDER and H. M. SPIERS (Gas J., 1930, 192, 161–166).—Tars produced in a wide range of coal-carbonising systems have very different properties for road-making purposes. By blending suitable light and heavy fractions from various tars, it is possible to prepare mixtures which conform to the specification, and thus to utilise material which would otherwise be rejected. Calculations can be made of the proper proportions of tars or tar fractions which are required to give a product of stated consistency, and nomograms are shown which make rapid determinations possible. R. H. GRIFFITH.

**Free carbon of bituminous coal tar.** J. MARCUSON (Chem.-Ztg., 1930, 54, 795–796).—The benzene-insoluble constituents of vertical- and horizontal-retort tars, amounting to 7% and 24%, respectively, consisted of oxy-acids 8.6%, 0.5%; pyridine-soluble resins 73%, 16.3%; pyridine-insoluble resins 18.4%, 32%; and partly-coked material 0%, 51.2%. No actual free carbon was present. The resins could be readily nitrated or sulphonated. Neither they nor the oxy-acids can be regarded as deleterious constituents of the tars, and it appears preferable therefore to consider the "free carbon" as including only the partly-coked material. This may be determined as follows: the tar (5 g.) is treated with 15 c.c. of fuming sulphuric acid (20% SO<sub>3</sub>), and the mixture, with thorough stirring from time to time, is set aside for 2 hrs. It is then diluted with 100–150 c.c. of water, warmed for 15 min. on the water-bath, and the precipitate separated by centrifuging. A. B. MANNING.

**Resin formation in benzols. III. Continuation of tests on production of stabilised benzols on the technical scale—storage and road trials.** W. H. HOFFERT and G. CLAXTON (Fuel, 1930, 9, 476–481; cf. B., 1930, 1137).—The general running of the engine in road trials on a coke-oven distillate stabilised by the addition of 0.03% of tricresol was as good as, if not better than, with National benzol mixture. Even after six months' storage this distillate gave no weighable amounts of deposit in the induction system and only a trace of soft carbon on the inlet valves. A similar distillate, which, however, contained some free sulphur, gave considerable deposits in the induction system. There is evidence that the lack of agreement between the results of the oxidation and storage tests with some benzols is due to the presence of free sulphur, which

forms non-volatile material from other constituents of the fuel during refluxing. With such fuels the measurement of the rate of oxygen absorption, instead of weight of non-volatile gums formed, might be a better guide to its behaviour on storage. A further cause of discrepancy between the results of the oxidation and storage tests is that in the former the residue corresponds with the non-volatile products which would be formed after a definite period of storage, e.g., 6 months, but gives no indication of the behaviour of the oil for intermediate periods. Tests of the benzols in a variable-compression engine indicate that the anti-knock value of the unsaturated hydrocarbons in the stabilised benzols does not differ appreciably from that of the aromatics. Fleet trials carried out by the Gas Light & Coke Co. on a stabilised benzol-petrol mixture gave satisfactory results.

A. B. MANNING.

**Phenols of petroleum.** E. HOLZMANN and S. VON PILAT (Brennstoff-Chem., 1930, 11, 409–413).—The phenols isolated from the crude naphthenic acids obtained from the distillates of two Polish petroleums (Borislav and Bitkow) have been examined; the phenol content of the crude acids amounted to 30% and 3.1%, respectively. The absence of phenol itself from the Borislav naphthenic acids has been established; all three cresols, however, together with *m*-5- and *m*-4-xenols and  $\beta$ -naphthol were isolated therefrom, and the presence of *o*-4-xenol and trimethylphenols was shown to be probable. No phenols were found having long side-chains or containing methoxy-groups. The phenols, but not the naphthenic acids, could be detected in the original crude petroleum (cf. von Kozicki and von Pilat, B., 1916, 526). A. B. MANNING.

**Oil emulsions.** A. C. BROWNE (Cal. Dep. Agric., Mon. Bull., 1930, 19, 389–405).—Analytical data of numerous oil types are recorded. No correlation is possible between viscosity, sulphonation tests, distillation ratios, or rates of evaporation. A. G. POLLARD.

**Breaking crude oil emulsions.** S. VUISHETRAVSKI (Azerbaid. Neft. Choz., 1930, No. 6, 83–98).—Water should be added at the moment when the emulsion leaves the well; demulsifying agents may be added at the same time. Factors affecting the formation of the emulsions were also investigated.

CHEMICAL ABSTRACTS.

**Motor spirit containing alcohol. III—VI.** W. SWIENTOSLAWSKI (Przemysl Chem., 1930, 14, 385–391, 409–412, 433–436, 457–461; cf. B., 1930, 976).—III. [With J. PFANHAUSER.] The fraction of azeotropic mixtures of gasoline, alcohol, and benzene, used as motor fuel, b.p. below 78.3°, is greater the higher is the benzene content; thus 92% of a mixture containing 30 vol.-% of benzene distils over below 78.3°, the b.p. of the residue then rising sharply to 140–180°. In general, this sharp rise in b.p. is indicative of the phenomenon of azeotropism, and corresponds with the exhaustion of the azeotropic mixture.

IV. [With J. PFANHAUSER and B. KARPIŃSKI.] The vapour pressure-temperature curves of a number of motor fuels containing alcohol show that at temperatures above 25° the vapour pressures of these fuels and of ordinary motor spirit are approximately equal, whilst



below 25° the mixtures examined are less volatile, showing that they are not well adapted to use in a cold motor.

V. [With H. STARCZEWSKA.] The b.p., condensation points, and other tonometric factors of a number of motor-fuel mixtures, containing (apart from petrol) alcohol, ether, benzene, solvent naphtha, or oleic acid, are given.

VI. [With H. STARCZEWSKA and J. KRZYKIEWICZ.] The heats of combustion of a number of motor mixtures containing alcohols have been determined in a Junker's calorimeter, and using Starczewska's bomb calorimeter. The former method gives a constant relative error, the results obtained by the latter method being 1.02 times as great. R. TRUSZKOWSKI.

**Analysis of the gradual oxidation prior to ignition of fuels in internal-combustion engines and the relation of such oxidation to detonation.**

W. M. ZAIKOWSKY, H. B. HOLROYD, and V. M. SOKOLOV (Physical Rev., 1929, [ii], 33, 264).—The change in oxidation of gasoline-air mixtures prior to ignition produced by the admixture of 0.25% of lead tetraethyl has been studied. The untreated mixture undergoes greater oxidation, but no corresponding increase of the compression pressure was observed, indicating that oxidation is not a homogeneous reaction. Lead tetraethyl and other anti-knock compounds reduce the rate of oxidation of the explosive mixture in the gaseous phase for a short time only, whilst oxides deposited on the tube walls remain as effective anti-oxidising agents. Lead oxide acts as a positive surface catalyst for carbon. The effectiveness of anti-knock compounds is attributed to their temporary elimination of oxygen molecules activated sufficiently to react directly with hydrocarbons. L. S. THEOBALD.

**Determination of lead tetraethyl in gasoline.**

L. J. CATLIN and J. E. STARRETT (Refiner Nat. Gasol. Mfr., 1930, 9, No. 7, 155).—To 100 c.c. is added a 10% solution of bromine in carbon tetrachloride 5 c.c. in excess of the volume required to produce a precipitate, decanting after 5 min. After washing with carbon disulphide and removing the solvent, the precipitate is boiled with 15% nitric acid, filtered, and the filtrate evaporated to fuming with sulphuric acid. After dilution the lead sulphate is allowed to deposit, and is then dissolved in ammonium acetate solution and titrated with ammonium molybdate, using tannin as external indicator. CHEMICAL ABSTRACTS.

**Gas-analysis apparatus.** JARRIER.—See I. **Adsorptive power of carbon for rubber.** DITMAR and PREUSZE.—See XIV. **Protection against toxics.** TURNER.—See XXIII.

**PATENTS.**

**Coal-washing apparatus.** A. WITZAL, A. G. RABEL, and H. F. STEVENS (U.S.P. 1,760,293, 27.5.30. Appl., 12.1.27).—The apparatus consists of a tank with two compartments, in the first of which the coal is separated from the slate and other refuse by means of an upward current of water flowing through a screen upon which the refuse collects, and whence it is discharged through an opening connecting the two compartments; it is finally removed from the tank by an endless conveyor. The water in the second compart-

ment is maintained in a more or less quiescent state; the height of the water level therein varies with the resistance which the material on the screen offers to the flow of water, and therefore with the amount of refuse which collects on the screen. The rate of discharge of the refuse from the screen can be varied by altering the angle of slope of a hinged plate at the discharge outlet, and the angle of the plate is controlled automatically by the height of the water in the second compartment in such a manner that the rate of discharge adjusts itself in accordance with the amount of refuse in the coal being washed. A. B. MANNING.

**Coke oven.** C. OTTO (U.S.P. 1,760,770, 27.5.30. Appl., 20.1.28. Ger., 16.11.26).—Each of the heating walls of a coke oven comprises a pair of runner walls and a series of parallel transverse binder walls extending between the runner walls and forming a series of vertical hairpin heating flues. The alternate binding walls, which extend vertically to the oven cover, constitute supporting columns and are made of materially greater thickness than the maximum thickness of the runner walls, in order to support the major part of the weight of the cover and loads resting thereon, and so enable the thickness of the runner walls to be decreased to permit quicker transmission of heat to the charge. A. B. MANNING.

**Coking retort ovens.** J. BECKER, ASSR. to KOPPERS Co. (U.S.P. 1,764,065 and 1,764,496—7, 17.6.30. Appl., [A] 22.5.22, [B] 18.11.20, [C] 17.11.20. Renewed [A] 18.6.26).—(A) A coking retort oven has the horizontal or bus flue, which connects the tops of the vertical flame flues, so placed as to lie in the top structure above the coking chambers and heating walls. The ducts connecting the vertical flues to the horizontal flue are insulated from the heating walls, and the horizontal flue is insulated from those parts of the top of the battery which lie directly over the coking chambers. Overheating of the upper part of the coking chamber and undue cracking of the volatile distillation products are thereby avoided. (B) A coke-oven battery which can be heated either by producer gas or coke-oven gas is provided with a main at one side alternatively operable for feeding producer gas or waste gas, and having reversing valve connexions to alternate pairs of regenerators. Another main on the same side of the battery supplies either coke-oven gas to the heating walls or air to the pairs of regenerators between the alternate pairs referred to above. The coke-oven gas can be fed alternately to the flues on opposite sides of the battery. Reversing valves are provided between the second main and the regenerators with which it is connected, and means are provided also for regulating the air supply through the main when producer gas is used as the heating medium. When coke-oven gas is used it is burned in an atmosphere containing both air and a neutral diluent, e.g., waste gas; the latter is supplied through the first main, and the air through flow pipes connected directly to the atmosphere. (C) In a further modification a pair of air mains is provided on the side of the battery opposed to that having the producer (or waste) gas and coke-oven gas mains, the air mains being connected to alternate pairs of regenerators. Means are provided for permitting air to flow



alternately into each of the two air mains, and for regulating the volume of flow therein in accordance with the gas requirements.

A. B. MANNING.

**Retort [or combustion device].** W. F. RIEGER, Assr. to STOKER MATTIC CORP. (U.S.P. 1,763,442, 10.6.30. Appl., 27.3.28).—A combustion device or burner for an underfed furnace comprises a mouthpiece to which the fuel is conveyed by means of a screw, and an air chamber below the mouthpiece and communicating therewith through a number of tapered air pipes. The edges of the mouthpiece rest on tubular metal pipes through which water is circulated.

A. B. MANNING.

**Manufacture of [activated] carbonaceous material.** A. OBERLE (U.S.P. 1,763,101—2, 10.6.30. Appl., [A] 3.11.23, [B] 31.1.25).—(A) Petroleum coke, or similar material, is heated in the presence of water and lime, or other calcareous material, to a temperature, preferably 480–510°, at which the water and volatile impurities are vaporised. The evolved vapours are distilled off under increased pressure, *e.g.*, 300 lb./in.<sup>2</sup>, which is subsequently reduced until the material, while still hot, is under a vacuum. Finally a current of air, carbon dioxide, or flue gas, etc. is passed through the material to remove any remaining volatile impurities. If desired, the activated carbon so produced may be further purified by treatment with an acid or with an organic solvent. (B) Petroleum coke is mixed with sawdust and acid sludge, and the mixture is set aside to permit the acid to disintegrate the sawdust. The mass is then treated with highly superheated steam, and, after being moulded if desired, may be carbonised further.

A. B. MANNING.

**Manufacture of fuels.** GEN. CARBONALPHA Co. (B.P. 313,889, 18.6.29. Holl., 18.6.28).—Gaseous, liquid, or solid fuels for use in internal-combustion engines are improved by the addition of carbon produced by the autocatalytic decomposition of carbon monoxide. This carbon has a low ignition point, *e.g.*, about 200°, and, in contradistinction to hydrogen, burns smoothly in the cylinder owing to its relatively lower speed of combustion. The speed of combustion of the mixed fuel may be controlled by the proportion of carbon added.

A. B. MANNING.

**Manufacture of liquid products from coal or other carbonaceous material by heating under pressure in the presence of hydrogen or gases or vapours containing hydrogen.** N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 335,386 and 335,513, [A] 3.10.29, [B] 31.5.29. [A] Holl., 7.11.28).—(A) The process is carried out in the presence of a catalyst consisting of a mixture of "Luxmass," pretreated as described in B.P. 314,859 (B., 1930, 1012) or in (B), and molybdenum or its compounds, *e.g.*, ferric molybdate or molybdenum sulphide. (B) Another suitable catalyst is prepared by dissolving "Luxmass" as far as possible in nitric acid, precipitating the solution with ammonia, and drying.

A. B. MANNING.

**Obtaining light hydrocarbons (benzines and petrols) during the low-temperature carbonisation of solid fuels.** A. MEIRO (B.P. 317,857, 22.8.29. Belg., 24.8.28).—The finely-crushed fuel is conveyed through a series of retorts in which it is subjected to

successively higher temperatures. The volatile products of distillation are fractionated, and the heavier hydrocarbons and part of the uncondensed gases are returned continuously to the retorts in order to undergo further decomposition into light hydrocarbons. The retorts are of metal, and are arranged horizontally one above the other in a setting heated by the gases from a furnace in which a part of the distillation gases is burned. Each retort is provided with a worm conveyor, and the material passes from one retort to the next lower one by gravity. Each retort has a gas outlet which is connected to the condenser in which the oils are fractionated, and is arranged immediately above the setting. Below the distillation retorts is an unheated inclined retort up which the semi-coke is conveyed to a closed storage bin, its lower end communicating through a siphon with a tar receiver.

A. B. MANNING.

**Gas generator.** W. G. PAQUETTE (U.S.P. 1,764,974, 17.6.30. Appl., 5.3.29).—A generator for the production of gas by the vaporisation of a liquid fuel comprises a closed compartment, another compartment open at the top and containing a movable bell, and a carburettor arranged below both compartments. A constant water level is maintained in the two compartments. A jet of water carries a current of air into the closed compartment, and thence to the carburettor, into which the liquid fuel is fed simultaneously. The carburetted air collects in the bell, the movement of which automatically shuts off the supplies of water (and therefore air) and liquid fuel when the bell has risen to a predetermined height, and re-establishes the connexions when the bell has again fallen.

A. B. MANNING.

**Gas generator.** F. W. STEERE, Assr. to SEMET-SOLVAY ENG. CORP. (U.S.P. 1,764,992, 17.6.30. Appl., 7.4.23).—A water-gas generator has a closed ash-pit below the grate through which alternate blasts of air and steam are passed. A number of pokers attached to a spider, which can be raised and lowered by hydraulic means, serve to break up and remove clinker and ash from the fuel bed. The grate may consist of a series of alternately fixed and movable rings, the latter being given a limited up-and-down movement by the motion of the pokers. In an alternative arrangement a single centrally located poker of relatively large diameter is used; the poker is hollow and is provided with perforations through which the air and steam may pass to the fuel bed. The grate may consist of a series of rotatable bars so designed as to crush any clinker caught between them and discharge it into the ash-pit.

A. B. MANNING.

**Producer gas generators, and apparatus for cleansing and cooling producer gas for automobiles etc.** A. E. HODGSON, N. C. JONES, and MOTOR OWNERS' PETROL COMBINE, LTD. (B.P. 334,888—9, 10.6.29).—(A) A generator for supplying gas to internal-combustion engines, particularly for automobiles, trucks, etc., and (B) a cooling and scrubbing apparatus for such gas, are described.

A. B. MANNING.

**Manufacture of mixed oil gas and water-gas.** HUMPHREYS & GLASGOW, LTD., Asses. of H. G. TERZIAN (B.P. 335,495, 29.1.30. U.S., 11.7.29).—The blast gases from a generator are burned with secondary air in a



carburettor and superheater. Oil and steam are then introduced into the carburettor and the lightly cracked vapours and oil gas are passed through the superheater and thence through a wash-box, wherein the hot gases take up more water vapour, and a condenser. The oil gas and remaining water vapour are then passed through the fuel bed of the generator, whereby the oil gas is cracked to re-form gas of lower calorific value and sp. gr., and the excess carbon released, which is deposited on the fuel, reacts with the water vapour to form water-gas.

A. B. MANNING.

**Production of combustible gas.** W. BERTELSMANN and A. C. BECKER (B.P. 335,228, 18.3.29).—A non-poisonous combustible gas is produced by freeing ordinary coal gas from carbon monoxide, *e.g.*, by conducting it over catalysts in the presence of hydrogen ( $3\text{H}_2 + \text{CO} = \text{CH}_4 + \text{H}_2\text{O}$ ), or by separation by compression and cooling, etc., and the gas is then restored to its original sp. gr., calorific value, and speed of ignition by the suitable addition of hydrogen, carbon dioxide, and/or nitrogen. The hydrogen and carbon dioxide may be derived from the carbon monoxide by passing it with steam over a heated catalyst. Any excess of carbon dioxide may be removed by washing. The hydrogen required may be obtained by electrolysis, utilising current produced during the periods when the external demand is small. The oxygen produced at the same time may be used for the continuous production of water-gas.

A. B. MANNING.

**Desulphurisation of gases and vapours.** N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 335,394, 8.10.29. Holl., 23.10.28).—The gases are passed over a contact mass containing thorium, preferably as oxide, at a raised temperature, *e.g.*, 650°. Small quantities of promoters, *e.g.*, copper, nickel, magnesium oxide, or lead oxide, may be added to the thorium oxide.

A. B. MANNING.

**Storage of acetylene.** F. ROFFEY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 335,820, 28.11.29).—Dry acetylene is stored under pressure in a cylinder containing absorbent solid (kieselguhr, kapok, etc.) impregnated with a solvent for acetylene, preferably of b.p. above 150°. Suitable solvents are  $\beta$ -ethoxyethyl acetate, glycerol diacetate,  $\beta\gamma$ -diethoxy-*n*-propyl acetate, glycol diacetate, glycerol diethyl ether, etc.

C. HOLLINS.

**Tar-separating apparatus.** H. HENNEBUTTE and E. GOUTAL, Assrs. to SOC. ANON. "LA CARBONITE" (U.S.P. 1,760,058, 27.5.30. Appl., 28.11.27. Fr., 2.12.26).—The hot gases and vapours obtained from the carbonisation of wood, peat, lignite, etc. are led into a receptacle through a central, stationary, conical conduit arranged therein. A rapidly rotating truncated cone to which blades or stirrers are attached is mounted within the lower part of the conical conduit, and serves to bring the incoming gases and vapours into intimate contact with the liquid in the receptacle. Curved tubes mounted on the walls of the stationary conduit, and having their ends immersed in the liquid, bring about a constant circulation of the liquid within the receptacle. The fixed gases, together with the water vapour if the apparatus is operated at a suitably high temperature, are

withdrawn from the top of the receptacle, while the condensed liquid is withdrawn through an overflow pipe which maintains a constant level within the vessel.

A. B. MANNING.

**Distillation of tar.** A. A. MACCUBBIN and J. ZAVERNIK, Assrs. to BARRETT CO. (U.S.P. 1,759,816, 20.5.30. Appl., 11.9.28).—The tar is pumped continuously through a pipe coil wherein it is heated under pressure, but without objectionable decomposition, and passes thence to a vapour box wherein the oil vapours are separated from the pitch residue, and are withdrawn to a condenser. The pitch residue is passed continuously to a vacuum box wherein it undergoes further distillation with or without additional heating. Increased oil yields and pitches of high m.p. are obtained.

A. B. MANNING.

**Conversion of olefines into other hydrocarbons.**

J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 336,234, 4.5.29).—Olefine-containing gases, not necessarily freed from hydrogen sulphide, are passed at atmospheric or higher pressures over a silicon catalyst contained in a tinned chromium-nickel (V2A) steel reaction vessel at 400–600°. Higher olefines or aromatic substances are produced, according to the velocity of the gas over the catalyst. At lower pressures higher temperatures are required. *E.g.*, ethylene, present in a gas mixture, was converted into methane, ethane, butylene, benzene, and toluene. T. A. SMITH.

**Conversion of hydrocarbon oils.** PETROLEUM

CONVERSION CORP., Asses. of A. P. SACHS (B.P. 336,380, 19.9.29. U.S., 6.10.28).—When hot inert gas is used as a direct heating medium in the cracking of oils or similar processes, it removes sulphur compounds in the form of hydrogen sulphide, and has to be freed from this before recycling. This can be effected by extraction with water at a point in the system where the gas is sufficiently cool.

T. A. SMITH.

**Cracking of liquid hydrocarbons.** O. D. LUCAS

(B.P. 336,269, 10.4.29).—Oil is heated to cracking temperature in heat exchangers by means of hot diphenyl or diphenyl oxide. The heating fluid is circulated through a pipe heater, and does not undergo any decomposition. Those parts of the plant in which the cracking stock is heated to decomposition temperature are arranged so that they can be readily freed from carbon.

T. A. SMITH.

**Purification of benzol.** C. F. HAGEN (U.S.P. 1,758,661, 13.5.30. Appl., 13.8.28).—A blended distillate suitable for motor fuel is obtained by mixing discoloured commercial benzol with a heavier hydrocarbon oil and distilling the mixture until a clear distillate is obtained, rejecting the first runnings and stopping the distillation when tar oils begin to come over.

T. A. SMITH.

**Purification of light oils extracted from coal**

gas. KOPPERS CO., Asses. of A. R. ALBRIGHT (B.P. 335,155, 15.4.29. U.S., 10.10.28).—The crude light oils are washed first with acid and then with a solution of sodium phenoxide in order to free them from sulphonic acids. The phenoxide solution used is that obtained in the process of removing the phenols from



the gas liquor, the latter being washed with a suitable transfer medium which is subsequently extracted with an alkali.

A. B. MANNING.

**Absorption process [for removal of light hydrocarbons from natural gas].** C. D. COULTER, Assr. to SOUTH WESTERN ENG. CORP. (U.S.P. 1,759,346, 20.5.30. Appl., 16.8.26).—Natural gas is commingled and washed with a heavy absorbing oil at a predetermined temperature, and under pressure such that only hydrocarbons condensable at that temperature are absorbed. The pressure on the oil is then released without appreciable change in temperature, liberating thereby some of the lighter hydrocarbons. The remainder of the laden absorbing oil is heated and the absorbed hydrocarbons and a portion of the oil are vaporised, cooled, and fractionally condensed. The condensed absorbing oil is returned to the main portion of oil in the system after it has left the absorption operation and before it reaches the heating stage. The vapours are cooled and fractionally condensed again, leaving the still lighter hydrocarbon vapours in vaporous form, the condensing action being carried on so that the gasoline condensate is not forcibly mixed with the non-condensed vapour.

H. S. GARLICK.

**Motor fuel. Liquid fuel.** C. O. JOHNS, Assr. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,757,837—8, 6.5.30. Appl., 21.5.24).—In the production of anti-knock fuels, the addition of a first small portion of lead tetraethyl has more effect than have the successive quantities added. Additional anti-knock effect is obtained by the addition of benzol and/or alcohols, instead of lead tetraethyl. Examples of suitable mixtures are: gasoline 80%, benzol 20%, lead tetraethyl 1.5 c.c./gal. of mixture; gasoline 95%, ethyl alcohol 5%, lead tetraethyl 1.5 c.c./gal. of mixture.

T. A. SMITH.

**Preparation of [anti-knock] motor fuels.** F. A. HOWARD, Assr. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,758,898, 13.5.30. Appl., 21.7.22).—The heavier fractions of gasoline are vaporised and mixed with a limited amount of air. The mixed gases, containing not more than 4 mols. of oxygen per mol. of hydrocarbon, are passed through a tube heated at 230–370°. The products are condensed, treated to remove gummy matter by steam-distillation or dilute soda washing, and remixed with the gasoline.

T. A. SMITH.

**Rectification of lubricating oil.** M. CZARNY and F. C. FROLANDER (B.P. 336,202, 6.6.29).—Lubricating oil from an internal-combustion engine is pumped through a cloth filter to remove solid matter and then through a small vessel containing a wick through the upper end of which a current of air is drawn. Any light constituents in the oil are drawn up the wick and evaporated in the current of air. Oil not drawn up the wick is returned to the engine.

T. A. SMITH.

**Manufacture of lubricants.** ALOX CHEM. CORP. (B.P. 335,152, 10.4.29. U.S., 18.12.28).—The saponifiable products of controlled oxidation of paraffins obtainable by the processes of B.P. 303,560 and 309,382 (B., 1929, 219, 467) are added, with or without "mould oils," to lubricating oils in order to decrease internal

friction and enhance the lubricating power. The amount added is preferably 0.25–0.75%. The crude oxidation products may be extracted with 95% alcohol; preferably they are saponified with caustic alkali and the soap solution, after dilution and clearing, is acidified, the acid precipitate being finally steamed to remove volatile products.

C. HOLLINS.

**Apparatus for pulverising coal and like substances.** W. J. DRUMMOND, Assr. to ASHLINGTON COAL CO., LTD. (U.S.P. 1,781,572, 11.11.30. Appl., 4.1.30. U.K., 24.4.29).—See B.P. 328,437; B., 1930, 750.

**Carbonising process.** W. E. TRENT, Assr. to TRENT PROCESS CORP. (U.S.P. 1,781,613, 11.11.30. Appl., 27.2.25).—See B.P. 261,954; B., 1927, 99.

**Gas-reversing device for regenerative coke ovens.** COLLIN & Co. (B.P. 338,040, 20.12.29. Ger., 27.12.28).

**Burner for heavy oil.** D. PIZZI (B.P. 337,983, 11.11.29).

**Liquid-fuel burners.** S. M. PYANKOW (B.P. 337,787, 6.8.29).

**Filters or strainers for petrol or other liquids [in pumps].** A. G. FENN, and ANGLO-AMERICAN OIL CO., LTD. (B.P. 337,539, 27.9.29).

**Crushing of solids** (B.P. 336,730). **Hydrogenation** (B.P. 336,569). **Deposition of dust** (B.P. 336,201). **Separation of dust from air etc.** (B.P. 336,221).—See I. **Gases containing hydrogen** (B.P. 336,944).—See VII. **Lubricating grease** (U.S.P. 1,749,251).—See XII. **Synthetic resins** (B.P. 314,810).—See XIII.

### III.—ORGANIC INTERMEDIATES.

**Thermal requirements and operating characteristics of the urea autoclave.** K. G. CLARK and U. L. GADDY (Ind. Eng. Chem., 1930, 22, 1084–1087).—Investigations were made on the continuous synthesis of urea from liquid ammonia and liquid carbon dioxide, using an autoclave working at 100 atm. and 183° with a conversion of 35–40%. In the experimental plant used, the urea was separated from unconverted ammonia-carbon dioxide compounds by steam-distillation of the latter. By the use of a series of thermocouples and blank experiments it was established that the reaction is exothermic, the heat of reaction being about 120 kg.-cal./kg. of urea. A heat balance is given and it is considered that with well-designed large-scale equipment the steam consumption in operating this process should be negligible. A series of corrosion tests on samples placed within the autoclave are given in the form of loss in weight/unit surface/24 hrs. They indicate the suitability of high-silicon chromium-nickel steel as a structural material.

C. IRWIN.

**Phenols from petroleum.** HOLZMANN and VON PILAT.—See II. **By-products of saccharin manufacture.** HERZOG.—See XII. **Detection and determination of p-hydroxybenzoic acid.** WEISS.—See XIX.

PATENTS.

**Manufacture of condensation products from diolefines and hydrocarbons.** I. G. FARRENIND.



A.-G. (B.P. 335,512, 5.7.29. Addn. to B.P. 315,312; B., 1930, 1103).—The following additional examples are given. Isoprene in toluene, heated with sodium at 160° for 1 hr., gives  $\epsilon$ (or  $\gamma$ )-phenyl- $\gamma$ (or  $\epsilon$ )-methyl- $\Delta^{\beta}$ -amylene, b.p. 218—222°.  $\beta\gamma$ -Dimethylbutadiene similarly yields  $\epsilon$ -phenyl- $\beta\gamma$ -dimethyl- $\Delta^{\beta}$ -amylene, b.p. 235—238°, whilst from butadiene in tetrahydronaphthalene a butenyltetrahydronaphthalene, b.p. 270—272°, is obtained. C. HOLLINS.

**Manufacture of alcohols [from olefines].** H. D. ELKINGTON. From N.V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 335,551, 23.5. and 4.6.29).—Ethylene (or other olefine, diolefine, etc.) is led with steam over the following catalysts: platinum, gold, silver, copper, iron, nickel, cobalt, chromium, tantalum, vanadium, tungsten, molybdenum, manganese, or compounds of these, especially oxalates, carbonyls, etc. which yield the metal under the conditions of reaction; or bismuth vanadate, copper vanadate, or copper phosphate, or mixtures of these. Examples are platinised pumice at 150°, copper phosphate on pumice at 155°, tungsten trioxide and ferric oxide on silica gel at 300°, copper oxide and tungsten trioxide on active carbon at 300°. C. HOLLINS.

**Manufacture of methanol [methyl alcohol, from carbon monoxide].** H. DREYFUS (B.P. 335,962, 5.6.29).—Carbon monoxide and hydrogen at 50—100 atm. are passed over zinc oxide or mixed zinc-chromium oxides at 300—450° and then, after removal of methyl alcohol, over a copper or manganese catalyst or other catalyst sensitive to sulphur-poisoning. Several vessels of each catalyst may be used in series. C. HOLLINS.

**Manufacture of oxygenated organic compounds [pyrolysis of methyl alcohol].** H. DREYFUS (B.P. 335,631, 2.7.29).—Methyl alcohol vapour is converted into ethyl alcohol and other products when passed, preferably with 1—2 vols. of nitrogen, at 500—600° over oxides, hydroxides, or alkoxides of sodium, potassium, barium, or calcium in a copper tube. C. HOLLINS.

**Purification of synthetic *n*-butyl alcohol.** R. RILEY, S. W. ROWELL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 335,683, 13.8.29).—The material is purified by electrolytic reduction; *e.g.*, it is stirred vigorously with dilute sulphuric acid or bisulphite solution in the cathode cell with a mercury cathode, the current density being 0.05 amp./cm.<sup>2</sup>, and the alcohol layer is subsequently distilled. C. HOLLINS.

**Manufacture of urea.** W. W. TRIGGS. From A. B. LAMB (B.P. 335,913, 27.6.29).—Liquid ammonia and liquid carbon dioxide are separately fed into an autoclave at 150° and the equilibrium mixture (40% of urea) is withdrawn from the top of the autoclave and transferred at atmospheric pressure to a still, where ammonia and carbon dioxide are removed by heat, dried, and returned separately to the liquefiers; the still liquid is an aqueous solution of urea. C. HOLLINS.

**Manufacture of thiourea [thiocarbamide].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 336,111, 19.10.29).—Calcium cyanamide is treated with hydrogen sulphide in absence of liquids or in presence of only sufficient liquid to form pastes with the solid. The product may be extracted with organic solvents, *e.g.*,

alcohol-ether, alcohol-carbon tetrachloride, benzene, with or without previous removal of water.

C. HOLLINS.

**Production of substances which may be used as treatment agents in the textile, leather, and allied industries.** H. T. BÖHME A.-G. (B.P. 317,039, 26.6.29. Ger., 9.8.28).—Wetting, cleaning, foaming, and dispersing agents are produced by cold sulphonation, in presence of acetic anhydride or acetyl chloride, of the alcohols derived from fatty acids by replacement of CO<sub>2</sub>H by CH<sub>2</sub>OH (cf. B.P. 308,824; B., 1930, 809). The alcohol from oleic acid, *e.g.*, is sulphonated with concentrated sulphuric acid in acetic anhydride. C. HOLLINS.

**Production of emulsions.** G. SCHICHT A.-G., and E. ULBRICH (B.P. 314,072, 17.6.29. Czechoslov., 22.6.28).—Esters of aminoalcohols are used as emulsifying agents for oil-in-water emulsions, *e.g.*, the dihydroxypropylaniline ester of castor oil acids, or *N*- $\beta$ -hydroxyethylpiperidine ester of phthalic acid. The agents described in B.P. 309,842, 295,024, and 306,116 (B., 1929, 590; B., 1930, 467, 809) are disclaimed. C. HOLLINS.

**Manufacture of polymerisation products of unsaturated ketones.** I. G. FARBENIND. A.-G. (B.P. 335,185, 15.3.29. Ger., 16.3.28. Cf. B.P. 307,936; B., 1930, 1016).—Ketones containing an aliphatic chain having only one double linking are polymerised in the form of aqueous emulsions; *e.g.*, the methylene derivative of methyl ethyl ketone is agitated for 8 days at 60° with 1% aqueous sodium oleate. C. HOLLINS.

**Catalytic [reduction] process.** E. I. DU PONT DE NEMOURS & Co. (B.P. 312,043, 18.3.29. U.S., 18.5.28).—In the hydrogenation of ketones or aldehydes to alcohols in presence of an oxide catalyst (oxides of zinc, magnesium, aluminium, silicon, chromium, vanadium, iron, etc.) having a dehydrating as well as a hydrogenating activity, the dehydrating effect is suppressed by addition of an oxide, hydroxide, carbonate, or other weak-acid salt of an alkali or alkaline-earth metal, *e.g.*, potassium or sodium carbonate. The reduction of acetone to isopropyl alcohol in presence of zinc chromite and potassium carbonate, and of aldol to  $\alpha\gamma$ -butylene glycol, is described. C. HOLLINS.

**Manufacture of (A, B) esters [vinyl acetate] and (C) di-esters of carboxylic acids [ethylidene diacetate].** CANADIAN ELECTRO PRODUCTS Co., LTD., Assees. of F. W. SKIRROW and G. O. MORRISON (B.P. 308,169, 308,170, and Addn. B.P. 335,223, 18.3.29. U.S., [A, B] 17.3.28).—Acetylene is passed into acetic acid to which has been added a catalyst precipitated by adding below 45° acetylsulphuric acid or a solution of sulphur trioxide in acetic acid to a solution of mercuric oxide in acetic acid. (A, B) For vinyl acetate the temperature of reaction with the acetylene should be 30—35°, and vinyl acetate should be removed as formed by means of large excess of acetylene. For ethylidene diacetate the optimum temperature is 80°. (C) The yield of diacetate is increased by condensing the vinyl acetate in the exit gases and returning it to the reaction. C. HOLLINS.

**Manufacture of glycidic acids.** A. A. KAUFMANN



(B.P. 335,391, 8.10.29).— $\alpha\beta$ -Unsaturated aldehydes are treated with alkali and either alkali hypobromite or hydrogen peroxide. Cinnamaldehyde, aqueous sodium hydroxide, and sodium hypobromite or hydrogen peroxide give phenylglycidic acid; methylglycidic acid, m.p.  $84^\circ$ , is obtained from crotonaldehyde.

C. HOLLINS.

#### Sulphonation of pyridine or its homologues.

I. G. FARBENIND. A.-G. (B.P. 335,817, 26.11.29. Ger., 26.11.28).—Pyridines are sulphonated with oleum in presence of mercuric sulphate.  $\alpha$ -Picoline and pyridine at  $225^\circ$  yield 3-sulphonic acids.

C. HOLLINS.

**Manufacture of 3-hydroxy- $\alpha$ -picoline.** I. G. FARBENIND. A.-G. (B.P. 335,818, 26.11.29. Ger., 26.11.28).— $\alpha$ -Picoline-3-sulphonic acid is fused with caustic alkali at  $210$ – $240^\circ$  to give 3-hydroxy- $\alpha$ -picoline, m.p.  $164$ – $166^\circ$ .

C. HOLLINS.

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

A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 335,547, 19.4.29).—Formaldehyde is condensed in acid medium with 2 mols. of a mixture of phenols comprising at least 1 mol. of a *p*-halogenophenol; or a non-hydroxylated aromatic aldehyde is similarly condensed with 2 mols. of a mixture of phenols comprising at least 1 mol. of a *p*-alkylated or *p*-halogenated phenol. The phenols must contain a free *o*-position and no salt-forming groups as substituents. Sulphuric acid, sulphuric and acetic acids, aluminium chloride, or ferric chloride may be used as condensing agents. The products are hydroxylated di- and tri-arylmethanes and are useful for mothproofing. Examples are: *o*-sulphobenzaldehyde with 2:4-dichlorophenol containing 2% of *p*-chlorophenol and 5% of 2:6-dichlorophenol, or with chlorinated crude *m*- and *p*-cresol mixture; formaldehyde with *p*-fluorophenol containing phenol.

C. HOLLINS.

**Manufacture of optically active 1-phenyl-2-methylaralkylaminopropanols-1 and 1-phenyl-2-methylaminopropanol-1 [ $\beta$ -aralkylmethylamino- and  $\beta$ -methylamino- $\alpha$ -hydroxy-*n*-propylbenzenes].** I. G. FARBENIND. A.-G. (B.P. 318,488, 29.8.29. Ger., 3.9.28).—*dl*- $\beta$ -Benzylmethylamino- $\alpha$ -hydroxy-*n*-propylbenzene, m.p.  $72$ – $73^\circ$ , prepared by reduction of  $\beta$ -benzylmethylaminopropiophenone (cf. Can. P. 291,001), is resolved by means of *d*-tartaric acid. The less soluble *d*-tartrate of the *d*-base, m.p.  $101$ – $102^\circ$ , and the *l*-base, m.p.  $49$ – $50^\circ$ , are described. Catalytic hydrogenation of the *l*-base or its hydrochloride removes the benzyl group as toluene and gives *l*- $\beta$ -methylamino- $\alpha$ -hydroxy-*n*-propylbenzene, m.p.  $39$ – $40^\circ$  (hydrochloride, m.p.  $215$ – $216^\circ$ ,  $\alpha_D$   $-35^\circ$ ); the *d*-base similarly gives the *d*-compound. Resolution of the *dl*-benzyl derivative with *l*-tartaric acid gives first the *l*-tartrate of the *l*-base.

C. HOLLINS.

**Manufacture of 2:4-di-(3'-nitrophenyl)-6-hydroxytriazine-1:3:5 [6-hydroxy-2:4-di-*m*-nitrophenyl-1:3:5-triazine].** I. G. FARBENIND. A.-G. (B.P. 335,783, 30.10.29. Ger., 31.10.28).—6-Hydroxy-2:4-diphenyl-1:3:5-triazine, m.p.  $283^\circ$ , is dinitrated in *m*-positions by mixed acid at  $10$ – $15^\circ$ ; the product has m.p.  $280$ – $281^\circ$ .

C. HOLLINS.

**Manufacture of mercapto[thiol]benzthiazole.**

IMPERIAL CHEM. INDUSTRIES, LTD., and K. H. SAUNDERS (B.P. 335,567, 26.3.29).—A solution of sulphur in carbon disulphide mixed with aniline is caused to flow upwards through a cylindrical pressure vessel at  $200$ – $275^\circ$ , reaction being completed during the time of passage, and overflows into an inner cylinder from which the product is drawn off. Hydrogen sulphide is blown off through a valve at the top of the vessel, and reduces the pressure to any desired degree. The process is continuous, and the apparatus may be used for other liquid-phase reactions under pressure.

C. HOLLINS.

**Manufacture of secondary aromatic amines [dinaphthylamines].** GOODYEAR TIRE & RUBBER CO., ASSEES. OF A. M. CLIFFORD (B.P. 310,871, 20.3.29. U.S. 2.5.28).— $\alpha$ - or  $\beta$ -Naphthol, or an equimolecular mixture, is heated with anhydrous ammonia (1.5 mols.) in a closed vessel at  $300$ – $310^\circ$ . The pressure initially is about 43 atm.

C. HOLLINS.

**Production of aromatic amino-derivatives from monoazo compounds, and of aminoazo compounds containing a  $\beta$ -naphthol component simultaneously therewith.** BRIT. RES. ASSOC. FOR THE WOOLLEN AND WORSTED INDUSTRIES, and A. T. KING (B.P. 335,645–6, 6.7.29).—(A) Monoazo compounds, other than Fast Acid Blue RH and others known to be reducible with sulphur dioxide, which do not contain a  $\beta$ -naphthol residue, are reduced with a sodium sulphite of the composition  $\text{Na}_2\text{SO}_3 \cdot 2\text{NaHSO}_3$  in alcohol or pyridine under reflux. (B) Secondary disazo compounds in which the end component is  $\beta$ -naphthol or a derivative thereof are reduced at  $15^\circ$  with aqueous sodium hydrogen sulphite, preferably with addition of pyridine, whereby only the azo grouping remote from the  $\beta$ -naphthol residue is reduced.

C. HOLLINS.

**Manufacture of dye intermediates [1-chloro-anthraquinone-2-carboxylic acid].** W. SMITH, J. PRIMROSE, and SCOTTISH DYES, LTD. (B.P. 335,232, 15.4.29).—2-Methylanthraquinone is chlorinated in weak oleum at  $0^\circ$ , whereby the proportion of by-products may be used in the direct production of 1-chloroanthraquinone-2-carboxylic acid from 2-methylanthraquinone or from 2-*p*-toluoylbenzoic acid.

C. HOLLINS.

**Halogenation of isocyclic compounds.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 335,948, 3.4.29).—Aromatic or hydroaromatic compounds are chlorinated or brominated by treatment with hydrogen chloride or bromide in oleum or chlorosulphonic acid in presence of carriers. Dibenzanthrone with hydrogen bromide, oleum, and antimony at  $60$ – $65^\circ$  gives a dibromo-compound (navy-blue vat dye). The preparation of di-, tri-, and tetra-bromopyranthrones (orange to orange-red vat dyes), bromo-1:2:6:7-dibenzpyrene-3:8-quinone (yellow-orange vat dye), bromotoluene, b.p.  $138$ – $140^\circ/15$  mm., bromobenzanthrone, and bromo-iodoanthanthrone (blue-red vat dye), is also described.

C. HOLLINS.

**Storage of acetylene (B.P. 335,820. Converting olefines into hydrocarbons (B.P. 336,234).—See II. Naphthenates (B.P. 335,863).—See VII. Lactic and acetic acids (B.P. 335,596). 2:3[ $\beta\gamma$ ]-butylene glycol by fermentation (B.P. 335,280).—See XVIII.**



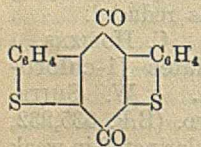
## IV.—DYESTUFFS.

## PATENTS.

**Manufacture of sulphurised dyes.** G. B. ELLIS. From CHEM. WORKS FORMERLY SANDOZ (B.P. 335,297, 6.7.29).—Hydroxylated phenazines are thionated in presence of molybdenum compounds. 3:7-Aminohydroxyphenazine, dissolved in sodium hydroxide solution, is heated with a solution of sodium polysulphide and molybdic acid at 110° for 30–32 hrs. to give a clear bluish-bordeaux-red sulphide dye. 3-Amino-7-hydroxy-2-methylphenazine similarly with ammonium molybdate or phosphomolybdic acid gives a dark red, as does its 8-carboxylic acid or 8-chloro-derivative; etho-safranone yields a bluish-red. C. HOLLINS.

**Manufacture of stable reduction compounds of [indigoid] vat dyes.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 334,919, 11.5.29. Addn. to B.P. 334,878; B., 1930, 1144).—A 6:6'-dihalogeno-4:4'-dimethylthiindigo is reduced with alkali and a reducing agent (hyposulphite) other than alkali hydrogen sulphide, and the solution is treated with acid, or preferably with carbon dioxide. Alcohol is added with advantage during reduction. C. HOLLINS.

**Manufacture of vat dyes of the dithionaphthylenequinone [benzbisthionaphthenequinone] series.** I. G. FARBENIND. A.-G. (B.P. 314,527, 19.6.29. Ger., 29.6.28).—3:3'-Dicarboxy-2:2'-dithionaphthylene ketone, obtainable from *o*-thiolphenylglyoxylic acid and  $\alpha\gamma$ -dichloroacetone, is converted by heating with acetic anhydride into an anhydro-compound, m.p. 272–273°, which at 300° passes into benzbisthionaphthenequinone (annexed formula), m.p. 295°. The same product may be obtained from the 3-mono-carboxylic acid, m.p. 268–269°, prepared by condensing *o*-phenylglyoxylic acid with chloroacetone and causing the resulting 2-bromoacetyl-3-thionaphtheneic acid to react with *o*-thiolbenzaldehyde. The quinone is a golden-yellow vat dye for wool. C. HOLLINS.



**Manufacture of [vat] dyes of the dibenzanthrone series.** SOC. CHEM. IND. IN BASLE (B.P. 314,903, 4.7.29. Switz., 4.7.28. Addn. to B.P. 262,774 and 294,486; B., 1928, 517; 1930, 9).—4:4'-Dibenzanthronyl is heated at 140–150° in trichlorobenzene with addition of ferric chloride to give a blue vat dye fast to washing. C. HOLLINS.

**Preparation of indanthrone dyes.** NEWPORT CO. (B.P. 314,803, 28.6.29. U.S., 2.7.28. Addn. to B.P. 297,692; B., 1928, 847).—Halogenated indanthrones are dehalogenated to any desired degree by heating with a primary arylamine and basic copper acetate, with or without sodium acetate. 3:3'-Dichloro- or -dibromo-indanthrone, refluxed with aniline and basic copper acetate, gives a pure, brilliant indanthrone. C. HOLLINS.

**Manufacture of monoazo [acid] dyes for wool.** I. G. FARBENIND. A.-G. (B.P. 308,830, 28.3.29. Ger., 30.3.28).—Sulphonic acids of  $\beta$ -naphthylamine or 2:8-aminonaphthol, or *N*-derivatives thereof, are coupled with diazotised 5-nitro-2-aminodiphenylsulphone- $\alpha$ -sul-

phonic acids. Examples are: 5-nitro-2-aminodiphenylsulphone-3'-sulphonic acid  $\rightarrow$  methyl- $\beta$ -naphthylamine-7-sulphonic acid (reddish-blue), or  $\beta$ -naphthylamine-7-sulphonic acid (reddish-violet), or  $\gamma$ -acid (greenish-blue). Similar dyes are obtained from 4'-methyl and 4'-methoxy-derivatives of the diazo component.

C. HOLLINS.

**Manufacture of an azo [acid] dye for wool.** I. G. FARBENIND. A.-G. (B.P. 335,705, 30.8.29. Ger., 4.10.28. Cf. B.P. 15,163 of 1913; B., 1914, 784).—3:3'-Dichloro-4:4'-diamino-5:5'-dimethyltriphenylmethane, obtainable from benzaldehyde and 3-chloro-*o*-toluidine, is tetrazotised and coupled with 1-(2-chloro-5-sulphophenyl)-3-methylpyrazolone to give a yellow acid wool dye fast to light. C. HOLLINS.

**Manufacture of [metal complex compounds of azo] dyes.** SOC. CHEM. IND. IN BASLE (B.P. 335,523, 23.4.29. Addn. to B.P. 307,705; B., 1930, 983).—Chromium, or other metal or metals having at. wt. between 45 and 59, is introduced into dyes obtained by coupling the dye J-acid  $\rightarrow$  J-acid (acid-coupled) with a diazotised *o*-aminophenol, or with 2 mols. of a diazotised anthranilic acid, or with 1 mol. of a diazotised *o*-aminophenol and 1 mol. of another diazo component, always excluding components having a salicylic acid grouping. [Stat. ref.] C. HOLLINS.

**Manufacture of chromium compounds of azo dyes forming metallic compounds.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 335,169, 16.5.29).—Chromable azo dyes containing at least one carboxylic or sulphonic acid group are prechromed in presence of organic bases, e.g., dimethylamine, aniline, cyclohexylmethylamine, pyridine. The process is applicable to the production of chromed dyes hitherto only obtainable in acid media, or not previously obtainable. Examples are: 4-chloro-*o*-aminophenol-6-sulphonic acid  $\rightarrow$  1-phenyl-3-methyl-5-pyrazolone (red) or 2:4-dihydroxyquinoline (bordeaux-red); 5-nitro-*o*-aminophenol  $\rightarrow$  Laurent acid (green); 1:2:4-aminonaphtholsulphonic acid  $\rightarrow$  1:8-naphtholsulphonic acid (blue), or phenylmethylpyrazolone (bordeaux-red), or 1-*p*-sulphophenyl-3-methyl-5-pyrazolone (bordeaux-red); anthranilic acid  $\rightarrow$  Schäffer acid (brown-red), 2:4-dihydroxyquinoline (yellow-orange), 1-phenylpyrazolone-3-carboxylic acid (red-yellow), or 1-(2-chloro-5-sulphophenyl)-3-methyl-5-pyrazolone (yellow); 5-sulphoanthranilic acid  $\rightarrow$  phenylmethylpyrazolone (yellow); 4-chloroaniline-3-sulphonic acid  $\rightarrow$  salicylic acid (yellow).

C. HOLLINS.

**Manufacture of insoluble azo dyes [ice colours and pigments].** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 335,555, 21.6.29).—A 2:3-hydroxynaphthoic arylamide is coupled in substance or on the fibre with a diazotised amine of the type Ar·NH·Ar·X·Ar·NH<sub>2</sub>, where Ar is an aromatic residue and X a direct linking or an azo group, or of the type Ar·NH·P·NH<sub>2</sub>, where P is a condensed aromatic system. Examples are: 4-amino-4'-anilindiphenyl  $\rightarrow$  5-chloro-*o*-toluidide or *p*-chloroanilide (claret, becoming reddish dark blue on removal of nitroso-group with phenylhydrazine-*m*-sulphonic acid); 4-amino-4'-anilinoazobenzene  $\rightarrow$  4- or 5-chloro-*o*-toluidide,  $\alpha$ - or  $\beta$ -naphthylamide,



anilide, 4- or 5-chloro-*o*-anisidide (garnet, becoming black by removal of nitroso-group). C. HOLLINS.

**Acid wool dyes of the anthraquinone series.** G. KRÄNZLEIN, F. ROEMER, and E. KRONHOLZ, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,780,976, 11.11.30. Appl., 19.12.28. Ger., 23.12.27).—See B.P. 302,928; B., 1930, 502.

**Halogenated isocyclic compounds** (B.P. 335,948).—See III.

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

**Ageing of cotton contained in rubber goods.** G. BARR (Trans. Inst. Rubber Ind., 1930, 5, 31—47).—Dyed, rubbered balloon fabric on summer exposure may actually increase in strength for the first month or so on account of the "locking" effect of the perished rubber on the fibres. The practice of dyeing yellow the outer cotton ply of a doubled-rubbered fabric aids, however, the preservation not only of the rubber, but also of the cotton. Ultra-violet light causes preferential deterioration of the cotton, whereas tropical sunshine acts more rapidly on the rubber and sulphur, the difference being so great that it is impracticable to use the mercury lamp to forecast the value of any scheme of protection for tropical service. Aluminium finishes protect by virtue of their reflecting power, which excludes not only actinic light, but also heat rays. The portion of the solar spectrum most active in inducing deterioration in cotton appears to coincide with that producing maximum effect with silver bromide, viz., the violet and near ultra-violet. The deterioration of cotton, like that of rubber, on exposure to sunlight is essentially a process of oxidation; practically no change occurs in an evacuated glass tube. Traces of acid in cloth are of serious importance, particularly in view of the temperatures attained during vulcanisation; the rate of loss of strength is approximately proportional to the weight of acid in the cloth and also is doubled for a rise of about 8°.

D. F. TWISS.

**Determination of crude fibre by use of a new kind of filter ["Cella" filter].** K. FEIST and E. KUNTZ (Z. Unters. Lebensm., 1930, 59, 480—483).—The filtration of fibrous residues after treatment with sulphuric acid or alkali may be conveniently performed by means of "Cella" filters, composed of pure cellulose and having pores 0.5—3.0  $\mu$ . Repeat experiments with new filters gave very concordant results, but the re-use of filters causes an apparent increase in the amount of crude fibre and is not recommended. Employment of small-size filters with fine pores is not advantageous, owing to the increase in time required for filtration.

H. J. DOWDEN.

**Variations in the lignin content of pine wood.** P. KLASON (Svensk Pappers-Tidn., 1929, 32, 527—530; Chem. Zentr., 1930, i, 3773).—The average lignin content of samples of pine wood from northern and southern Sweden was 28.1%, with variations of 1% only. Hence climate does not appear to affect the lignin content. On the assumption that lignin consists of 70% of coniferylparaldehyde and 30% of coniferyl alcohol, wood should contain 4.1% OMe; the observed value was 4.3%.

A. A. ELDRIDGE.

**Determination of mechanical wood pulp [in paper].** A. NOLL and F. HÖLDER (Papier-Fabr., 1930, 28, 700—702).—The bone-dry paper (4 g.) is disintegrated (special apparatus described) with 200 c.c. of hot water for 2 min., and the disintegrator washed down with a further 25 c.c. of water. The pulp is kept in a water-bath at 25° for 30 min., in order to obtain regular swelling of the fibres, 100 c.c. of *N*-potassium permanganate are added, and the mixture is kept at 25° for 1 hr., with continued stirring, and filtered. Then 30 c.c. of 0.1*N*-oxalic acid are added to 50 c.c. of water, brought to the b.p., acidified with sulphuric acid, and to this are added 10 c.c. of the filtrate. The excess of oxalic acid is titrated with permanganate. A table is given relating permanganate consumption with mechanical wood-pulp content.

T. T. POTTS.

**Determination of water in paper, pulp, and half-stuff.** M. KNOPFF (Woch. Papierfabr., 1930, 61, 416—418; Chem. Zentr., 1930, i, 3506).—A discussion of the methods available.

A. A. ELDRIDGE.

**Moisture-proof determination of waxed papers.** C. A. THOMAS and H. J. REBOULET (Ind. Eng. Chem. [Anal.], 1930, 2, 390—391).—An apparatus is described for rapid determination (2 hrs.) of water-vapour transmission through waxed papers. Pinholes and imperfections in the paper before waxing cause large increases in transmission, whilst normal handling and creasing have not much effect.

J. LEWKOWITSCH.

**Photoelectric process control.** STYER and VEDDER; McMASTER.—See XI. **Determination of crude fibre in cacao.** KÜRSCHNER and HANAK.—See XIX.

## PATENTS.

**Purified [vegetable] fibre.** R. A. GORTNER and W. F. HOFFMAN, Assrs. to NORTHWEST PAPER CO. (U.S.P. 1,757,768, 6.5.30. Appl., 30.1.26).—Such fibres, particularly wood fibres in chip or pulp form, are freed from mineral impurities (other than traces of silica) and colloidal organic colouring matter by electrolysis a 5—10% aqueous suspension of the material, using 220 volts *D.C.* Graphite electrodes are preferably used and are separated from the pulp by, e.g., fabric diaphragms. Hot or cold water may be used, optionally in conjunction with suitable reagents, and provision is made for continuously renewing the electrolyte in the electrode compartments. The purified material contains less than 0.05% of ash and does not discolour when heated at 60—70° for 100 hrs. Oxycellulose and hemicelluloses are not removed by this treatment.

D. J. NORMAN.

**Compressed [fibrous] products.** HALIZITE CORP., Asses. of A. HAWERLANDER (B.P. 336,754, 20.9.29. U.S., 13.3.29).—Sawdust, wood shavings, etc., after being moistened with a volatile liquid (petrol), are treated with sufficient binding material (phenol-formaldehyde condensation product) to cover the surface of the fibrous particles and are finally subjected to heat and pressure.

F. R. ENNOS.

**Preparation of mercerised wood-fibre tissue.** M. O. SCHUR and R. H. RASCH, Assrs. to BROWN CO. (U.S.P. 1,749,003, 4.3.30. Appl., 5.1.27).—High- $\alpha$ -cellulose wood-fibre tissue is treated with caustic soda solution of mercerising concentration containing an oxidising



agent (hypochlorite, permanganate), washed with hot water, treated with an acid and reducing agent (sulphurous acid) to neutralise the last traces of alkali and oxidant, and finally washed free from the reaction products; the high-grade cellulose produced is suitable for nitration to yield artificial silk, films, lacquers, etc.

F. R. ENNOS.

**Colloided cellulose and indurated fibre.** F. G. HAHN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,757,755, 6.5.30. Appl., 8.2.28).—Cellulose in woven, non-woven, or sheet form is gelatinised without dissolution by treating it with an aqueous solution of a fixed caustic alkali containing 2–11 mols. of alkali per 100 mols. of water at a temperature between the f.p. of the reagent and about 20° above it. When the required degree of swelling is attained the material is compressed to the desired shape and the swelling agent removed by, e.g., washing with water.

D. J. NORMAN.

**Production of cellulose fatty acid esters.** K. WERNER (B.P. 336,349, 15.8.29).—Water is removed from air-dried cellulose before esterification by intimately mixing in a ball or roller mill at temperatures up to 50° with sufficient fatty acid and fatty acid anhydride to complete the esterification, but with an amount of catalyst insufficient to promote the reaction (0.02–0.05% of sulphuric acid or 0.05% of sulphuryl chloride calc. on the wt. of cellulose); esterification is subsequently carried out by gradual addition of a weak catalyst (sulphuryl chloride, phosphorus oxychloride), the temperature being allowed to rise to 60–70°.

F. R. ENNOS.

**Manufacture of (A) highly esterified acetylnitrocellulose, (B) artificial silk.** N.V. FABR. VAN CHEM. PROD., Assees. of J. N. ELGERSMA (B.P. 317,854–5, 21.8.29. Holl., 24.8.28).—After acetylating partly nitrated cellulose with acetic acid and acetic anhydride in the presence of sulphuric acid or a derivative thereof as catalyst, the excess of anhydride is removed by addition of the equivalent amount of water or an alcohol, and the whole is kept at 30–50° until the cellulose sulphate present has been transformed into acetate. The solution is then (A) precipitated with water and the highly esterified acetylnitrocellulose dried, dissolved in a suitable solvent, and worked up into the desired form; or (B) treated with sufficient sodium acetate to render the free sulphuric acid inactive, filtered, and spun into a bath containing aqueous acetic acid.

F. R. ENNOS.

**Cellulose ether compositions.** E. I. DU PONT DE NEMOURS & Co. (B.P. 312,309, 15.4.29. U.S., 24.5.28).—A cellulose ether is dissolved in a mixture of an alcohol which is a solvent for the ether, e.g., butyl alcohol, with an aliphatic hydrocarbon or hydrocarbon mixture which is a non-solvent or a poor solvent therefor, e.g., gasoline, the two liquids being in such proportions that the mixture has a greater solvent power than the combined solvent power of the separate liquids.

F. R. ENNOS.

**Solvent for cellulose nitrate.** J. P. TRICKEY, Assr. to QUAKER OATS Co. (U.S.P. 1,756,228, 29.4.30. Appl., 3.8.28).—The use of esters of tetrahydrofurfuryl alcohol is claimed, these being water-white, stable in colour, free

from objectionable odour, compatible with gums and resins, and miscible with other solvents used in nitrocellulose lacquers.

D. J. NORMAN.

**Reducing the viscosity of nitrocellulose.** (A) G. H. TOZIER, and (B) P. C. SEEL, Assrs. to EASTMAN KODAK Co. (U.S.P. 1,757,491 and 1,757,481, 6.5.30. Appl., 6.11.26).—(A) Nitrocellulose is digested for at least 8 hrs. with water under pressure in an acid-resisting autoclave at above 110° until the desired viscosity is reached. The nitrocellulose should remain completely immersed during the whole operation, and the steam used for heating is injected below the water level. The water thus introduced, together with any wash water which may be added to the system to facilitate the removal of decomposition products, flows away through a heat exchanger and a steam trap. An external circulating pump is provided. (B) The use of an acid-resisting autoclave is obviated by neutralising the acid as it is formed by the controlled introduction of alkali, e.g., 5% caustic soda solution, into the circulatory system.

D. J. NORMAN.

**Apparatus for preparation of cellulose xanthate.** M. DASSONVILLE (B.P. 337,149, 26.9.29. Fr., 1.7.29).—A form of jacketed rotating digester is described, which has a smooth interior and can be opened by separating into two halves.

B. M. VENABLES.

**Manufacture and working up of artificial materials.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 336,250, 4.6.29).—A true sulphonic acid or sulphionate of a derivative of an aliphatic or cycloaliphatic hydrocarbon containing at least 8 carbon atoms (sulphonated oleic acid) is incorporated with artificial masses made from or containing cellulose or its derivatives, or with solutions or baths for their production.

F. R. ENNOS.

**Manufacture of [cellulosic] sheets, films, or the like.** CELLULOID CORP. (B.P. 315,840, 19.7.29. U.S., 19.7.28).—A solution of cellulose or of a cellulose derivative is poured on to the surface of a forwardly moving inert liquid medium, e.g., mercury or a low-melting alloy, which is heated to evaporate the solvent, and the partially-dried sheet, after being stripped off, is passed over one or more drying drums and thence to the winding device.

F. R. ENNOS.

**Manufacture of films, ribbons, and sheet-like solids.** SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 318,250, 26.8.29. Fr., 31.8.28).—Liquid films are formed from solutions or pseudo-solutions by surface tension between two or more solid threads or plates serving as supports, and are then coagulated; the supports, which may be of silk, wire, or of the same composition as the film, may be removed from the manufactured product or not, as desired.

F. R. ENNOS.

**Rotary dewatering sieves for cellulose and the like.** J. STRINDLUND (B.P. 336,879, 27.12.29. Nor., 11.6.29).—A cylindrical sieve which rotates while partly immersed in the pulp mixture has a number of chambers below the sieve cloth, each of which is provided with an outflow pipe which is bent backwards with respect to the direction of rotation so that the outflow end remains immersed in the water passing into the interior of the



sieve until the layer of pulp on the sieve has moved out of the mixture. The dewatering is effected by the difference in pressure due to the level of the pulp mixture being higher than that of the outflowing water in the interior of the cylinder. F. R. ENNOS.

**Manufacture or treatment of materials made of or containing organic derivatives of cellulose.** H. DREYFUS (B.P. 336,217, 28.3.29).—In order to improve their tensile strength, extensibility, or elasticity, such cellulose materials in the finished condition or continuously with their production by dry- or semi-dry-spinning processes, are treated with neutral organic solvents in relatively high concentration, *e.g.*, 25–65% aqueous or alcoholic solutions of diacetone alcohol, ethyl lactate, etc., while being stretched or allowed to shrink as desired; the excess of solvent is subsequently removed from the product and the remainder allowed to dry therein. F. R. ENNOS.

**Desulphurisation of viscose artificial silk.** BRIT. ENKA ARTIFICIAL SILK CO., LTD., Assees. of N.V. NEDERL. KUNSTZIJDEFABR. (B.P. 317,438, 20.7.29. Holl., 16.8.28).—The silk is treated in baths or sprays of ammonium sulphide solution, in which the proportion of polysulphide is maintained below a predetermined limit by means of a cyclic process, part of the liquor being distilled under pressure to regenerate ammonium sulphide, which is returned for re-use. F. R. ENNOS.

**Sheet material and its manufacture.** A. E. H. FAIR, Assr. to A. D. LITTLE, INC. (U.S.P. 1,757,010, 6.5.30. Appl., 1.7.29).—An all-wood paper suitable for electric condensers is made from a mixed furnish containing  $\alpha$ -pulp and 10–60% of sulphate pulp. The pulps are beaten separately and require prolonged and careful treatment, the object being in the case of the  $\alpha$ -pulp to reduce the fibres without cutting them or inducing appreciable hydration, and in the case of the sulphate pulp to fibrillate and at the same time to hydrate the fibres. D. J. NORMAN.

**Treatment of waste spun or woven textile material.** A. L. BURLIN (U.S.P. 1,781,317, 11.11.30. Appl., 26.10.29. U.K., 2.11.28).—See B.P. 322,608; B., 1930, 138.

**Washing of spinning cakes of artificial silk.** BRIT. BEMBERG, LTD., Assees. of J. P. BEMBERG A.-G. (B.P. 337,590, 25.10.29. Ger., 9.11.28).

**Continuous machines for dehydration [by suction] of the pulp in the manufacture of artificial leather and of papers from a pulp difficult to drain.** C. CARCANO (B.P. 337,346, 22.7.29).

**[Winding device for] production of artificial threads and filaments [by the dry-spinning process].** J. E. PEDDER, and COURTAULDS, LTD. (B.P. 336,718, 21.8.29).

**Lactic and acetic acids** (B.P. 335,596).—See XVIII. Nitrocellulose (B.P. 336,235).—See XXII.

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

### PATENTS.

**Production of fast tints [ice colours] on wool.** Soc. Chim. Ind. in BASLE (B.P. 310,758, 29.4.29. Switz., 29.4.28).—If the amount of alkali in the paddling bath is

increased to 0.6–1.8 g. of sodium hydroxide per litre, ice colours of enhanced brightness are obtained on wool by the use of 2:3-hydroxynaphthoic arylamides (containing no amino-, sulphonic, sulphonamide, or carboxyl groups) with diazo components free from hydroxyl, sulphonic, or carboxyl group. An example is the anilide with 5-chloro-*o*-toluidine (scarlet).

C. HOLLINS.

**Mixture for transforming [reducing depth of] dyed colours in fabrics etc.** A. G. BLOXAM. From AUSTRALINE COLOR TRANSFORMERS PROPRIETARY, LTD. (B.P. 317,410, 13.3.28).—Dyed material is bleached to any desired degree by treatment with a mixture of a stripping agent (sodium hyposulphite, carbonate, hydrogen carbonate, or sulphate) and an oxidant capable of bleaching the dye (a mixture of sodium acetate, sodium thiosulphate, and hydrogen peroxide).

C. HOLLINS.

**Dyeing and printing [with soluble leuco-vat dye esters].** DURAND & HUGUENIN SOC. ANON. (B.P. 313,407, 10.6.29. Ger., 9.6.28).—Over-oxidation when using a copper salt as developer for "Indigosols" is prevented by addition of a thiocyanate to the developing bath.

C. HOLLINS.

**Printing on materials made of or containing cellulose esters or ethers.** BRIT. CELANESE, LTD., and J. A. WAINWRIGHT (B.P. 335,206, 19.6.29).—Fabrics of cellulose acetate etc. are capable of reproducing fine differences of light and shade when printed with plates or rollers engraved to a large number of gradations of depth.

C. HOLLINS.

**Treatment of textile and other materials made of or containing cellulose esters.** BRIT. CELANESE, LTD. (B.P. 309,377, 8.4.29. U.S., 7.4.28. Addn. to B.P. 304,596; B., 1929, 1012).—Instead of being added from time to time, the whole of the alkali necessary to effect the required degree of saponification, together with soap and a salt of a strong base with a weak acid, is added initially to the saponifying bath, which is in bulk 130–160 times the wt. of the cellulose ester in the material.

F. R. ENNOS.

**Delustring [cellulose ester or ether silk] fabrics.** C. DREYFUS and H. PLATT, Assrs. to CELANESE CORP. OF AMERICA (U.S.P. 1,756,941, 6.5.30. Appl., 27.12.28).—The material, particularly cellulose acetate silk fabric, is impregnated with a zinc salt in the presence of a swelling agent (zinc chloride, acetone, formic or acetic acid), set aside for several hours, then passed through a solution of a sulphide (*e.g.*, sodium sulphide), rinsed, soaped, and dried; the zinc sulphide deposited within the fabric reduces its lustre permanently and also gives the fabric an affinity for direct dyes. The process may also be applied to partly hydrolysed acetate silk.

A. J. HALL.

**Composition of matter and its employment. [Rendering balloon fabric impermeable to inflating gas.]** C. M. CARSON, Assr. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,750,460, 11.3.30. Appl., 6.1.27).—The fabric is coated with a mixture of 1 pt. of gelatin and fibrin with at least 3 pts. of rubber latex, together with glycerin and a hygroscopic electrically conductive salt (potassium chloride) which is chemically inert to





the other ingredients; after partly drying, the film is sprayed with formaldehyde, which tans the gelatin, and is subsequently dried.

F. R. ENNOS.

**Dye vats or jiggers and machines employing immersion rollers.** F. HEWITT (B.P. 337,894, 12.9.29).

**Application of liquids to [travelling] yarns or threads.** BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 337,433, 30.5.29).

**Machine for mercerising fabrics.** O. HOFFMANN (B.P. 337,712, 10.3.30).

**Clarifying liquids** (B.P. 336,865).—See I. **Treatment agents for textiles** (B.P. 317,039). **Mothproofing agents** (B.P. 335,547).—See III. **Treatment of cellulose derivatives** (B.P. 336,217).—See V.

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

**Loss of nitrous products owing to degradation in the lead-chamber [sulphuric acid] process.** A. SANFOURCHE and L. RONDIER (Bull. Soc. chim., 1930, [iv], 47, 952–958).—Experiments were carried out by passing a gaseous mixture of oxygen, nitrogen, sulphur dioxide, and nitric oxide into a vessel containing sulphuric acid, the temperature and conditions being those which prevail in the lead chambers. The reduction of the nitrous substances (to nitrous oxide and nitrogen) passes through a maximum as the temperature rises, this maximum increasing as the acid concentration decreases and moving towards the region of high temperature as it increases. The reduction becomes more pronounced with increasing sulphur dioxide concentration, except at high temperatures, when the reduction is virtually constant. The results indicate that the reaction takes place in sulphuric acid solution.

S. K. TWEEDY.

**Nitric acid produced from ammonia by modified pressure system.** G. FAUSER (Chem. and Met. Eng., 1930, 37, 604–608; cf. B., 1928, 601).—Experiments on the industrial scale showed that in the oxidation of ammonia, the conversion is greater at atmospheric than at higher pressures, and that the rate of oxidation of nitric oxide increases as the pressure is raised. In a plant in which the former reaction is carried out at atmospheric pressure, the absorption apparatus, working under pressure, consists of a series of horizontal cylindrical vessels, cooled by water on the outside, in which the gases bubble through nitric acid or water on the countercurrent principle. Data in connexion with the design of the absorption system are given.

D. K. MOORE.

**Ammonium sulphate from gypsum.** B. WAESER (Brennstoff-Chem., 1930, 11, 396–398, 418–420).—Recent developments in the production of ammonium sulphate are briefly discussed, reference being made particularly to Baud's process (B., 1928, 12), to the Bayer cement process (B., 1926, 271), and to other developments in the manufacture of sulphuric acid and cement from gypsum (cf. Diehl, B., 1921, 506 A; Neumann, B., 1927, 106). The cost of converting ammonia into ammonium sulphate is estimated at 24 pf. per kg. of nitrogen, by direct combination with sulphuric acid, or 13.3 pf./kg., by the gypsum process. These costs may

actually be higher than the cost of production of the ammonia itself by synthetic methods.

A. B. MANNING.

**Extraction of magnesia from dolomite.** L. CAMBI (Giorn. Chim. Ind. Appl., 1930, 12, 438–441).—The technical possibility of Twynam's process (B., 1929, 851) is considered. Using a dolomite containing MgO 20.78, CaO 31.2, ( $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ ) 0.43,  $\text{CO}_2$  46.82, insoluble matter 0.19%, and sieving and levigating the milk prepared from the calcined dolomite in order to separate the heavy granules containing undecomposed dolomite and a large part of the impurities, it was found possible to prepare magnesia which, after washing and calcining, had the composition (%): MgO 98.91, CaO 0.39, ( $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ ) traces. The economic side of the process is not discussed.

T. H. POPE.

**Preparation of cupric acetate.** Z. A. IOFA and L. L. KLYACHKO-GURVICH (J. Appl. Chem., Russia, 1930, 3, 361–365).—Cupric oxide is agitated for 1–2 hrs. with concentrated acetic acid and the acetate (containing 1–2% CuO) is stirred at 80° with the solution obtained from a previous crystallisation; after filtration the solution is diluted with solution remaining from the first operation and cooled to crystallise.

CHEMICAL ABSTRACTS.

**Dust control in phosphate rock grinding.** W. H. GABELER (Ind. Eng. Chem., 1930, 22, 1077–1080).—The Davison Chemical Co.'s plant at Baltimore for grinding phosphate rock consists of 12 Raymond mills, with an average capacity of 4 tons per hr. each. To overcome the dust nuisance, Draco two-compartment filters with cotton-napped bags were installed, each designed to handle the air from two mills. The air volume passing through one unit was 900 cub. ft. per min. from each vent, i.e., 1800 cub. ft. per min. The static pressure at the intake to the filter was 1 in., and the suction at the exhaust 2 in. The average load was 2 h.p. A recovery of 32 lb. of pulverised rock per hr. was obtained from two mills, equivalent to 1 ton per day and representing 0.2% of the capacity. This amounted to a dust concentration, assuming 99% efficiency, of 2.05 grains per cub. ft. Comparative screen tests on material from the mills and the filter showed, respectively, 91.9 and 99.9% through 100-mesh, 81.9 and 99.5% through 150-mesh, 67.0 and 96.5% through 200-mesh, and 51.3 and 83.9% through 300-mesh. The power consumption for the whole system does not exceed 15 h.p.-hrs., and the only labour costs are for inspection and lubrication; maintenance charges over 6 months were negligible.

W. J. WRIGHT.

**Change of the water-soluble phosphoric acid content of superphosphate during storage in bulk.** III. **Relation of the variety of raw phosphate and of the conditions of its manufacture to the decline of the water-soluble phosphoric acid content.** T. SHŌJI, E. SUZUKI, and E. NANAI (J. Soc. Chem. Ind., Japan, 1930, 33, 418–419 B; cf. B., 1930, 903).—The change of the water-soluble phosphate content during storage at the ordinary temperature and at 60° of superphosphates made from phosphate rock from various districts has been studied: only in a few cases is the water-soluble content preserved by storage at



the higher temperature. Variations of procedure during the manufacture of superphosphate, such as alteration of the quantity and concentration of the sulphuric acid used and of the degree of fineness of the mineral, are without influence on the diminution of the water-soluble content.

H. F. GILLBE.

**Reversion of the citrate-soluble phosphate in Rhenania phosphate.** K. SCHARER (Landw. Versuchs-Stat., 1930, 111, 1—10).—Samples of Rhenania phosphate, after storage in corked bottles for a year, showed slight decreases in the proportion of citrate-soluble phosphate. These were, however, within the limits of error officially allowed in this test. A. G. POLLARD.

**Soluble sulphurs [polysulphide sprays].** R. P. TUCKER (Cal. Dep. Agric., Mon. Bull., 1930, 19, 422—429).—The atomic structure of alkali polysulphides is examined.

A. G. POLLARD.

**Determination of small quantities of oxygen in gases.** H. R. AMBLER (Analyst, 1930, 55, 677—680).—The combustion of hydrogen in excess of oxygen in the presence of platinum wire at red heat is examined with a view to the determination of oxygen in excess of hydrogen. Ammonia was not formed in the presence of nitrogen and hydrogen in sufficient quantities to introduce error, and the method was found suitable for all proportions of oxygen up to the explosion limit (about 4%) and for higher proportions if the pipette is strong enough. The accuracy of the method may be reduced to 0.1% of the total gas if oxides of carbon are present.

D. G. HEWER.

**A Western nitrogen-fixation plant.** G. N. WESTBY (Ind. Eng. Chem., 1930, 22, 1099—1103).—A description is given of the plant and arc process used by the American Nitrogen Products Co., at La Grande, Washington, prior to the destruction of the factory by fire. The product contained 96.5—99.0%  $\text{NaNO}_2$ , according to the market requirements. With a production of 200 tons per month, the cost of the nitrite was 4.55 cents per lb.

W. J. WRIGHT.

**"Dry-ice."** D. H. KILLEFFER (Ind. Eng. Chem., 1930, 22, 1087—1091).—The development of the solid carbon dioxide ("Dry-ice") industry in America is recorded. For preserving perishable goods, Dry-ice shows a marked economy compared with ice, the available refrigeration per lb. being about double. Refrigerators can maintain a temperature of 5° for ten days without recharging. For cooling large quantities of materials, as distinct from maintaining a low temperature, Dry-ice is uneconomical. Suitable methods of storage and distribution of large quantities of the material during the winter or summer are described.

W. J. WRIGHT.

**Carbonised molasses waste.** KILP. **Determination of lead tetraethyl in gasoline.** CATLIN and STARRETT.—See II. **Magnesite.** DWORZAK.—See VIII. **Photoelectric process control.** STYER and VEDDER; McMASTER.—See XI. **Potash shale as fertiliser.** HEATH. **Magnesium salts as fertilisers.** DIX and BISCHOF.—See XVI. **Liquor aluminii acetico-tartrici and liquor aluminii acetici.** HOLDERMANN.—See XX. **Protection against toxics.** TURNER.—See XXIII.

## PATENTS.

**Manufacture of nitric acid.** M. BATTEGAY, Assr. to CALCO CHEM. Co., INC. (U.S.P. 1,756,532, 29.4.30. Appl., 3.2.27. Fr., 19.10.26).—Nitrosylsulphuric acid is circulated downwards through narrow towers filled with Raschig rings and heated electrically at the lower end at 60—110°, while a current of substantially pure oxygen is passed upwards through the towers. The addition of small quantities of ceric sulphate, ammonium metavanadate, or chromic acid to the acid liquid materially accelerates the rate of oxidation of the nitrogen peroxide therein.

A. R. POWELL.

**Manufacture of nitric acid.** W. W. TRIGGS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 336,233, 27.3.29).—In the catalytic oxidation of ammonia, the gases leaving the oxidiser at 700—900° pass first to a cooler in which their temperature is reduced to about 250°, and thence to a condenser in which they are rapidly cooled to about 20°, water vapour being thereby condensed and oxidation of nitric oxide to nitrogen peroxide begun. The condensate flows back in countercurrent to the ascending hot gases, and by careful regulation a condensate containing the minimum amount of nitric acid may be drawn off and conducted to the absorbers. The gases from the condenser pass to an oxidiser in which they are completely converted into nitrogen peroxide, oxidation being accelerated under the pressure maintained throughout the process, and thence reach the absorbers where they are bubbled through a countercurrent of successively more dilute acid. Nitric acid of 69% concentration may be obtained. [Stat. ref.]

W. J. WRIGHT.

**Manufacture of phosphoric acid and hydrogen.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 337,109, 24.8.29).—Phosphorus or phosphorus acids of a lower stage of oxidation than phosphoric acid are heated at 250—500° under pressure with anhydrous phosphoric acid.

W. J. WRIGHT.

**Synthetic production of ammonia.** R. S. RICHARDSON, Assr. to CHEM. ENG. CORP. (U.S.P. 1,750,649, 18.3.30. Appl., 12.3.27).—After the gases leave the converter, part of the ammonia is condensed and removed before fresh gases are introduced, being thus obtained in a pure state, and after admission of the gases the remainder of the ammonia is condensed, thereby absorbing any impurities from the gases or the system.

W. J. WRIGHT.

**Wire-gauze catalysts [for the oxidation of ammonia].** C. TONIOLO, and "AZOGENO" SOC. ANON. PER LA FABR. DELL'AMMONIACA SINTETICA E PROD. DERIV. (B.P. 337,680, 6.1.30. It., 25.7.29).—The catalysts comprise two or more metal (e.g., platinum) wire gauzes of different mesh laid one above the other so that the wires of the different gauzes lie at different angles to one another.

A. R. POWELL.

**Manufacture of anhydrous sodium sulphide.** A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 336,251, 10.6. and 6.8.29).—Sodium sulphate is reduced by hydrogen or other gaseous reducing agent at 500—600°, the mass being maintained in the solid state during the reaction. Reduction is accelerated if a catalyst is employed, such catalyst consisting of sodium sulphide



or caustic soda, or metals or mixtures containing them, *e.g.*, the crude melt from the sodium sulphide manufacture.

W. J. WRIGHT.

**Manufacture of ammonium carbamate and the carbamates and carbonates of the alkali metals and of magnesium.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 336,206, 1.7.29).—The reaction of ammonium bicarbonate with liquid ammonia is effected with 2–3 times the amount of ammonia theoretically required, so that conversion takes place at ordinary temperatures. The process may also be carried out with gaseous ammonia, which, under the pressure employed, forms a solution with part of the salt.

W. J. WRIGHT.

**Manufacture of ammonium sulphate.** F. G. LILJENROTH, ASSR. to KUNSTDÜNGER-PATENTVERWERTUNGS A.-G. (U.S.P. 1,758,449, 13.5.30. Appl., 24.12.28).—A mixture of finely-ground calcium cyanamide and calcium sulphate is heated under pressure with water and carbon dioxide to produce ammonium sulphate and calcium carbonate. The latter is collected and dissolved in nitric acid to obtain calcium nitrate and carbon dioxide under pressure for use in the first stage of the process.

A. R. POWELL.

**Inhibiting the corrosive effect of brines towards iron and steel.** O. V. MARTIN, ASSR. to MARTIN-COLVIN Co. (U.S.P. 1,750,761, 18.3.30. Appl., 26.10.27).—The brine is treated with milk of lime sufficient to liberate about 0.05% of magnesium hydroxide from the magnesium chloride present in the brine. The  $p_H$  of the solution is thus reduced to a value at which the dissolved salts cease to promote the corrosion of the metal.

A. R. POWELL.

**Production of alimentary salt.** P. WOOG (B.P. 319,203 and Addn. B.P. 336,279, [A] 5.7.29, [B] 8.7.29. Fr., [A] 17.9.28, [B] 18.2.29).—(A) An alimentary saline preparation, which contains all the salts in sea water without alteration, is obtained by concentrating the sea water until salts separate in a crystalline form, and then evaporating it at not above 105°, so that it does not become superheated; an agglomeration of crystals, which hinders evaporation, is prevented by constant stirring. (B) To the mixture obtained as described an insoluble magnesium salt or finely-divided magnesium metal may be added. The magnesium salt may consist of partly hydrated magnesium carbonate, which may be prepared by adding an alkali carbonate to the paste.

W. J. WRIGHT.

**Making magnesium chloride from chlorine and a magnesium base.** T. GRISWOLD, JUN., and R. M. HUNTER, ASSRS. to DOW CHEM. Co. (U.S.P. 1,756,247, 29.4.30. Appl., 2.7.28).—Magnesium oxide, hydroxide, or carbonate is treated with chlorine and sulphur dioxide in the presence of water to obtain a solution of magnesium chloride and sulphate from which the latter is separated by evaporation and crystallisation. The magnesium chloride mother-liquor is evaporated to obtain the dry salt for the production of metallic magnesium by fused electrolysis, the chlorine liberated in this process being used in the preparation of fresh quantities of the chloride.

A. R. POWELL.

**Refining and purifying barium sulphate.** H. V. FARR, ASSR. to MALLINCKRODT CHEM. WORKS (U.S.P. 1,752,244, 25.3.30. Appl., 4.3.26).—The powdered sulphate is heated at 130° with a concentrated solution of a salt of another alkaline-earth metal capable of forming a complex barium salt. The product is hydrolysed with water, and the extremely fine barium sulphate produced is purified by washing. Various references to such double salts are given.

W. J. WRIGHT.

**Production of naphthenates of heavy and alkaline-earth metals.** I. G. FARBENIND. A.-G. (B.P. 335,863, 16.1.30. Ger., 22.2.29).—Naphthenic acid is boiled with aqueous sodium hydroxide, a solution of a salt of a heavy metal or alkaline-earth metal is added directly to the boiling liquid, and the precipitate, washed by hot decantation, is maintained molten at about 130° until dry; the whole operation is conducted in the same vessel. The preparation of lead manganese and cobalt zinc naphthenates is described.

C. HOLLINS.

**Manufacture of [crystalline] zinc sulphide.** E. C. GASKILL (U.S.P. 1,758,741, 13.5.30. Appl., 17.12.26).—Zinc sulphide ores mixed with carbonaceous material are heated by electrical resistance to 1000° in a vacuum or in a current of nitrogen or steam free from oxygen, whereby pure zinc sulphide volatilises and condenses in settling chambers and bag plant as fine crystals which can be ground to yield a dense white pigment.

A. R. POWELL.

**Production of basic lead chromate.** A. STEWART, ASSR. to U.S. CHEM. PRODUCTS Co., INC. (U.S.P. 1,751,295, 18.3.30. Appl., 9.12.27).—An aqueous suspension of lead sulphate is treated at 70° with a soluble alkali dichromate and sufficient soluble alkali hydroxide to give a product containing about 55%  $PbCrO_4$  and 45%  $PbO$ . The basic lead chromate, after settling, is washed, filtered, and dried at 120°.

W. J. WRIGHT.

**Manufacture of zeolites.** PERMUTIT Co. (B.P. 319,746, 26.9.29. U.S., 27.9.28).—Solutions of aluminium sulphate and sodium silicate are mixed, and to this mixture is added, before gelation, a solution of aluminium sulphate. The proportions of the reagents are such that (a) the total alkali content of the silicate and aluminate solutions exceeds that required to form neutral sulphate with the sulphuric acid of the aluminium sulphate, and (b) the ratio  $Na_2O:Al_2O_3$  in the final mixture is 1:1.

W. J. WRIGHT.

**Obtaining pure glauconite [for water-softening].** H. KRIEGSHEIM and W. VAUGHAN, ASSRS. to PERMUTIT Co. (U.S.P. 1,757,374, 6.5.30. Appl., 4.2.28).—Crude greensand is thoroughly washed with water to remove clay and fine particles, carefully graded into sizes, and vigorously agitated with hot dilute sodium hydroxide solution to remove humus. The purified sand is then passed through a hot solution of sodium silicate ( $d$  1.02) to restore silica removed by the previous treatment, agitated with very dilute aluminium sulphate solution to remove free alkali, and finally scrubbed thoroughly with warm water.

A. R. POWELL.

**Production of elemental sulphur.** R. C. BENNER and A. P. THOMPSON, ASSRS. to GEN. CHEM. Co. (U.S.P.



1,751,066—8, 18.3.30. Appl., [A—C] 8.9.26).—(A) A mixture of sulphide ore and carbonaceous material is subjected to the action of an oxidising gas in counter-current, and the gaseous products at 350° are passed over a catalyst, such as bauxite, to complete the reduction to elemental sulphur. (B) Sulphide ore is treated with a carbonaceous material comprising a considerable amount of hydrocarbons, and the gaseous products are treated with an oxidising gas and passed over a catalyst as in (A). (C) In the processes described in (A, B), reduction is carried out at temperatures above 1200°, so that the slag may be removed in a fused condition.

W. J. WRIGHT.

**Apparatus for purifying sulphur and obtaining carbon disulphide and sulphur oxides.** G. JAKOVAMERTURI (B.P. 310,972, 3.5.29. Fr., 4.5.28).—The lower portion of a furnace is divided vertically into three compartments in each of which are set two superimposed cast-iron retorts, all of which can be put into communication by means of pipes and valves; the centre ones also communicate with an upper chamber. Temperatures of 800–900°, 450–500°, and 130°, respectively, may be attained in the three compartments. In the upper portion of the furnace, in addition to the chamber referred to, is a second one in which are placed horizontally three water-cooled, sheet-steel cylinders, open at one end, and communicating by a pipe with a cooled receiver. In manufacturing carbon disulphide, two retorts in an end compartment are loaded with brown coal and the centre ones with sulphur, the volatilised sulphur passing into the former pair and combining with the coal. For the production of roll sulphur the first two retorts are cut off and sulphur vapour from the centre pair is condensed in the last two, from which the sulphur is run off. To obtain flowers of sulphur, only the centre retorts are utilised, the sulphur vapour passing through the upper chamber into the cooled cylinders, and any oxides of sulphur being condensed in the receiver. Sulphur ore may be used instead of sulphur, arrangements being then made for dehydrating and the removal of tar.

W. J. WRIGHT.

**Separation and recovery of gases [sulphur dioxide from smelter gases].** R. L. HASCHE, ASST. to AMER. SMELTING & REFINING CO. (U.S.P. 1,758,398, 13.5.30. Appl., 17.8.27).—The gases, freed from dust and fume, are passed through silica gel or active charcoal at 25°, whereby the sulphur dioxide is absorbed. The adsorbed gas is liberated by passing steam through the adsorbent and the greater part of the steam is condensed at as high a temperature as possible. The gases then pass through a water-cooled condenser to remove most of the remaining water and are finally dried by means of sulphuric acid before passing to the refrigerating apparatus for the production of liquid sulphur dioxide.

A. R. POWELL.

**Production of phosphorus oxychloride.** J. S. DUNN, F. BRIERS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 337,123, 6.9.29).—Natural calcium phosphates are pretreated at 300° with phosgene to decompose carbonate, and, after removal of the calcium chloride by washing, the product is dried and treated with phosgene.

W. J. WRIGHT.

**Purification of triaryl phosphates.** W. GIBSON and C. R. HENSHAW, ASSES. to IMPERIAL CHEM. INDUSTRIES, LTD. (U.S.P. 1,781,225, 11.11.30. Appl., 23.10.29. U.K., 24.10.28).—See B.P. 322,057; B., 1930, 95.

**Vessels proof against chemicals etc.** (B.P. 316,134).—See I. **Smelter-furnace by-products** (U.S.P. 1,755,845). **Utilisation of dolomite etc.** (U.S.P. 1,749,210—1).—See X. **Complex iron compounds** (B.P. 335,965).—See XX. **Breathing cartidges** (B.P. 337,170).—See XXIII.

## VIII.—GLASS; CERAMICS.

**Ultra-violet window glazing.** H. E. BECKETT (Dept. Sci. Ind. Res., Building Res. Bull. No. 8, 1930, 13 pp.).—The literature dealing with the therapeutic value of ultra-violet radiation and the behaviour of ultra-violet light-transmitting glasses is reviewed, and the amount of radiation received by a window facing north is examined.

M. PARKIN.

**Russian and Slovakian magnesite.** E. DWORZAK (Feuerfest, 1930, 6, 145–150).—Chemical analyses of a number of magnesite bricks of Austrian, Slovakian, and Russian origin are given, and a comparison is made of the colour, composition, and physical properties both of the bricks and of the granular, calcined material. Russian magnesite bricks have a higher magnesia content (> 92% MgO) than the Slovakian, with consequent greater refractoriness and refractoriness under load. A lower flux content makes them less vitreous and more porous, and, owing to the lower firing temperature, the sp. gr. is somewhat lower than that of the Slovakian bricks. In spite of the higher porosity, the Russian bricks are equally resistant to slag attack. The chemical composition of the Russian calcined magnesite is the same for all granulations, viz., about 91% MgO and 3% Fe<sub>2</sub>O<sub>3</sub>. A grain size of 0–5 mm. gives the most satisfactory degree of vitrification both with the Russian and Slovakian material, and if larger grades are used a certain proportion of fines must be added to act as a bond. In steel works up to 15% of open-hearth furnace slag, with a grain size of about 5 mm., may be added to the Russian magnesite to assist vitrification.

F. SALT.

**Effect on boiler-furnace walls of gases from bagasse and coffee-pod fuels.** F. W. FREISE (Feuerfest, 1930, 6, 150–152).—A study was made of the injurious effect of gases rich in potassium on the walls of furnaces fired with sugar-cane residues or with coffee-pod waste. Most of the potassium was present as carbonate, which readily decomposes. A special test furnace was constructed, in which different types of refractories were tested. Bricks containing less than 50% Al<sub>2</sub>O<sub>3</sub> lasted for short periods only. Furnaces exposed to vigorous attack should be built of bricks with not less than 60% Al<sub>2</sub>O<sub>3</sub>. Zirconia bricks conferred no further advantages, but a paste made up with ground zirconia and water-glass gave good results either as a mortar or as a coating for those parts of the walls directly exposed to the gases.

F. SALT.

**Friction of dry solids in vacuo.**—SHAW and LEAVEY.—See I. **Photoelectric process control.** STYER and VEDDER; McMASTER.—See XI.



## PATENTS.

**Tunnel kilns.** V. LASTOVICKA (B.P. 336,171, 23.12.29).—A tunnel kiln for firing bricks has a longitudinal recess extending along one side from the chimney, near the entrance end of the tunnel, to a point near the middle, opposite which is the firebox. The kiln is surmounted by a drying tunnel, vents being provided in the kiln roof. F. SALT.

**Manufacture of enamel ware.** W. E. DOUGHERTY (B.P. 336,797, 17.10.29).—Titania is a suitable opacifier for glasses free from chlorine, or for glasses containing chlorine if a suitable salt of the calcium group is present. If traces of iron are present, relatively small amounts of tin oxide or zirconia, or both, added to the titania, will remove the yellowish cast in the enamel. Titanium may be used in this way as an opacifier also in the ground coat. F. SALT.

**Semi-porcelain bodies for manufacture of pottery.** J. E. TAMS (B.P. 336,172, 27.12.29).—A semi-porcelain body, either for casting or jollying, contains asbestos powder, ball clay, ground stone, and/or ground felspar, roughly in the proportions of 50, 40, 10, and 10 pts., respectively. F. SALT.

**Manufacture of ceramic material.** A.C. SPARK PLUG Co. (B.P. 315,196, 8.7.29. U.S., 7.7.28).—An aluminium silicate is fused in the presence of fluxes, and the fused mass is subjected to sudden cooling in a stream of water, if necessary under pressure. Crystal growth is thus arrested, and porcelain bodies made with this fused product as the non-plastic ingredient have greater mechanical strength owing to continued and additional crystalline growth during the firing of the porcelain. The process is particularly valuable in the case of sillimanite, mullite, and andalusite. F. SALT.

**Methods and apparatus for feeding molten glass.** L. MELLERSH-JACKSON. From BALL BROTHERS Co. (B.P. 337,988—9, 12.11.29).

**Feeding the glass batch to a glass furnace.** J. E. POLIAK. From HAZEL-ATLAS GLASS Co. (B.P. 337,950, 24.10.29).

**Heating apparatus for making safety glass etc.** DU PONT VISCOLOID Co. (B.P. 337,547, 3.10.29. U.S., 27.10.28).

## IX.—BUILDING MATERIALS.

**The rotary kiln in cement manufacture.** III—VII. W. GILBERT (Cement, 1930, 3, 561—566, 699—704, 946—953, 1196—1206, 1472—1484).—The description and results of a 6-day process test on a typical wet-process rotary kiln are given. The kiln was 8.5 ft. in diam. and 202 ft. long, with an inclination of 1 in 25. During the test 263.8 tons of coal (dry) produced 928.9 tons of clinker. The total heat loss from the shell of the kiln was calculated to be equivalent to approx. 327 lb. of coal per hr., and from the cooler 67 lb. per hr., the figures representing conditions on a quiet day, and with a partly roofed kiln. By substituting a double layer of firebrick, 9 in. in total thickness, for the usual 6-in. lining, 76.7% of this loss might be saved, though the increased first cost and maintenance must be offset. The test indicated 29.6% of excess air in

the kiln and 74% efficiency of the coal as fired. The latter figure might be improved by reducing within limits the water content of the slurry by cutting down the excess air, or by otherwise reducing the temperature of the exit gases. C. A. KING.

**Iron and steel used in cement manufacture.** ANON. (Cement, 1930, 3, 1305—1315).—The heavy wear on the mechanical parts of cement plant has suggested a description of the characteristics and mechanical properties of the different forms of iron and steel as applied to operating conditions. Although the production of wrought iron has decreased considerably by reason of its supersession by mild steel, the superior resistance of the former to shock, heat, and oxidising conditions is noted. C. A. KING.

**Accelerated soundness test [for concrete aggregates].** H. F. KRIEGER (Civil Eng., 1930, 1, 120—122).—Considerable variation in the results of the sodium sulphate immersion test will be obtained unless the temperature is controlled closely, due to the effect of temperature of saturation of sodium sulphate; e.g., between 20° and 30° the concentration of the solution changes from 20% to above 41% Na<sub>2</sub>SO<sub>4</sub>. For sewage-disposal media at least 20 pieces should be taken; for concrete aggregates at least 50 pieces 1—2 in. in diam., and for finer materials the sample should consist of 1 kg. carefully screened. A solution of 400 g. of sodium sulphate per litre is prepared at 30°, and set aside for 12 hrs.; at all times excess crystals should be present. The sample, dried at 105° and cooled, is immersed in the solution for 19 hrs. at 30°, examined, dried, and the loose fragments are separated. The test should be run for 5 cycles on concrete aggregates, and for 20 cycles on material for sewage-filtering purposes. An individual specimen breaking into three or more pieces or losing more than 20% of its weight by chipping is considered to have failed, and a sample having 20% or more of its constituent pieces failing is unsound; 15—20% indicates "doubtful soundness" and necessitates a second trial. C. A. KING.

**Sinkage [of pulp wood].** III. Changes in the water-gas system in logs during seasoning and flotation. G. W. SCARTH and R. D. GIBBS (Canad. J. Res., 1930, 3, 80—93; cf. Gibbs, A., 1930, 1322).—Observations on the production of gas in logs by fermentation during seasoning and flotation are recorded. C. W. GIBBY.

**Sinkage studies.** IV. Mechanism of absorption of water by wood blocks. G. W. SCARTH (Canad. J. Res., 1930, 3, 107—114; cf. A., 1930, 1322).—Studies of the rate of penetration of water into white pine-wood blocks show that the limiting factor in the sinking of logs is the rate of escape of air by dissolution and diffusion. R. K. CALLOW.

**Evaluation of sieve analyses [of cement] and Abrams' modulus of fineness.** A. HUMMEL (Zement, 1930, 19, 355—364; Chem. Zentr., 1930, i, 3478).

**Coal tar.** CHADDER and SPIERS.—See II. Ammonium sulphate from gypsum. WAESER.—See VII.

## PATENT.

**Cement for electric lamp caps** (B.P. 316,866).—See XI.



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

**Metallography of some ancient Egyptian implements.** (Sir) H. C. H. CARPENTER and J. M. ROBERTSON (J. Iron Steel Inst., 1930, 121, 417—454).—Photomicrographs of various ancient iron implements show that the Egyptians were familiar with carburising and water-quenching; the latter process may have been known in 1200 B.C., but tempering was probably not discovered until considerably later. H. F. GILLBE.

**Agglomeration of blast-furnace dust and treatment of iron carbonate ores by the Greenawalt process.** R. CORDONNIER (Rev. Mét., 1930, 27, 467—478).—The material to be treated is mixed with 2—8% of powdered fuel and 5—20% of water. The mixture is fed by a charging carriage on to a mechanical hearth. Combustion is initiated by ignition with blast-furnace gas (or mineral oil) and continues under the action of a fan. The agglomerate resembles a very porous coke in texture, is highly permeable to gases, and may be smelted with great economy. Volatile impurities are removed and the percentage of iron is increased. In treating carbonates the charge is covered with a mixture of calcined ore and powdered fuel, to prevent uneven evolution of gas. E. H. BUCKNALL.

**Influence of liquid as against solid pig-iron additions on the operation of the open-hearth furnace.** F. WEISGERBER (Stahl u. Eisen, 1930, 50, 1489—1494).—Using 20—30% of pig iron in the open-hearth steel process, the addition of the pig iron in the molten state increases the capacity of the furnace by 10%, reduces the quantity of pig iron required for efficient operation by 10%, reduces the fuel consumption by 15%, and slightly increases the consumption of dolomite. The resulting economies amount to about 2 RM. per ton of steel. A. R. POWELL.

**Influence of melting conditions on the micro-structure and mechanical strengths of grey cast irons containing various amounts of carbon and silicon.** A. L. NORBURY and E. MORGAN (J. Iron Steel Inst., 1930, 121, 367—392).—The influence of melting conditions and superheating, of addition of steel, graphite, silicides, and oxide, and of streams of hydrogen, nitrogen, and carbon dioxide on molten mixtures of Swedish iron, Armco iron, and ferrosilicon has been studied. The refining influence of superheating on the graphitic structure has been confirmed, and addition of small quantities of steel has a similar effect; when, however, 40% and 10% ferrosilicons, 60% carbon silicide, and 5% nickel silicide are added to melts of whiteheart malleable iron, grey test bars of good mechanical properties are produced. Nickel silicide and 10% ferrosilicon alloy readily with the iron, but 40% ferrosilicon and calcium silicide alloy more slowly. Nitrogen and hydrogen, bubbled simultaneously or separately through the molten iron, tend to improve the metal by bringing slag to the surface, but are otherwise without influence; carbon dioxide causes the appearance of considerable quantities of slag, consisting probably of silicates. Iron oxide additions cause the removal of manganese, silicon, and carbon by oxida-

tion, without causing appreciable reduction in strength of the cast metal. The strength and hardness of pearlitic irons increase as the total carbon content decreases and as the distance below the eutectic composition increases; areas of supercooled graphite with ferrite lower considerably the strength and hardness of pearlitic irons. In ferritic irons the strength and hardness increase with decreasing size of the graphite particles and with increasing distance below the eutectic, whereas increase of the silicon content lowers the strength and increases the hardness. H. F. GILLBE.

**Pressure-welding of iron.** H. ESSER (Arch. Eisenhüttenw., 1930—1, 4, 199—206; Stahl u. Eisen, 1930, 50, 1500).—Pieces of pure iron and of iron with increasing carbon content were welded by pressure *in vacuo* at various temperatures and the tensile strength of the welds was determined; the shape of the ends welded was made conical so as to ensure fracture taking place at that point. Under a welding pressure of 2 kg./mm.<sup>2</sup> the strength of the welds in pure iron increases with rise of welding temperature to a maximum at 900°, then falls sharply, and finally increases again; at 900° continuous growth of the crystals through the weld is observed, but at 910° a distinct line of demarcation is observable along the joint. The strongest welds are obtained with metal having a very fine-grained structure; the strength of welds made with single crystals varies with the orientation of the crystal surfaces in contact. Highly polished surfaces form the strongest welds at much lower temperatures than roughened surfaces, e.g., maximum strength in the first case is obtained by welding at 970°, but if the surfaces are roughened with grade 1G emery maximum strength of the weld occurs with a welding temperature of 1280°. With increasing carbon content the temperature range for obtaining strong welds by pressure becomes more restricted.

A. R. POWELL.

**Tempering of severely quenched special steels.** A. MICHEL and P. BÉNAZET (Rev. Mét., 1930, 27, 501—508).—Previous work on the tempering of austenitic steel (B., 1930, 287) is now extended to a study of the effects of adding chromium, alone or with nickel, tungsten, or cobalt, by means of the Chevenard differential dilatometer. Typical quenched structures have been obtained on cooling steel specimens 4 mm. in diam. in air from 1250° (sometimes 1150°). In chromium steels increase in the Cr : C ratio renders such structures more easily obtainable and more difficult to destroy by tempering. This result is explained on the basis of formation of Cr<sub>3</sub>C<sub>2</sub> and a ferrite rich in chromium. Steels containing 5% W in addition to chromium exhibit a part of the  $\gamma$ — $\alpha$  transformation on heating. This is an effect intermediate between the two types of behaviour previously observed (*loc. cit.*). Tempering after severe quenching produces a fine dissemination of the carbides. Such treatment may be employed prior to hardening alloy steels of commerce. E. H. BUCKNALL.

**Pearlitic rustless cast steel.** V. ZSÁK (Giesserei, 1930, 17, 339—342; Chem. Zentr., 1930, i, 3348).—Steel is rendered rustless by 12—18% Cr; Acl is shifted to about 800°, and the eutectoid is at 0.3% C. For good incorrodibility the carbon content should be



0.1—0.3%. Steel containing 0.25—0.3% C and 18% Cr is recommended for thin-walled castings; manganese (0.4%), silicon (0.5%), phosphorus and sulphur (0.03%), and nickel (0.4%) have little effect. A. A. ELDRIDGE.

#### Properties of some steels containing chromium.

A. R. PAGE and J. H. PARTRIDGE (J. Iron Steel Inst., 1930, 121, 393—415).—The mechanical strength and toughness and the resistance to oxidation and scaling at temperatures up to 900° have been determined for various chromium steels. Increase of the chromium and silicon contents from 4.7% and 0.16% to 6% and 0.31% hardly influences the mechanical properties of the steel. Steels containing about 5.6% Cr cannot be air-hardened if more than 2% Si is present, and although silicon up to 3.5% tends to make the steel brittle without impairing its strength at the ordinary temperature, at higher temperatures the strength is considerably reduced; up to 5.8—8% Cr and up to 1.6—3.1% Si produce steel of poor strength above 700°, although of greater strength than low-chromium steels at the ordinary temperature. At 900° steels containing 1.0—1.5% Si are markedly deficient in ductility. Nickel-chromium austenitic steels are stronger and less plastic when hot than the silicon steels, but there is little advantage in increasing the nickel and chromium contents from 9% to 13%. The elongation and reduction of area under load are fairly constant for nickel-chromium steels at all temperatures, whereas with silicon steels both diminish as the temperature rises. The impact values show that all the steels are relatively brittle and that heating increases the toughness; steel containing 5.99% Cr, 0.31% Si, and 0.57% C shows a remarkably high impact value at about 400°. Chromium steels containing 1.5% Si are highly resistant to oxidation at a red heat and the scale formed is adherent. All the silicon-chromium steels examined have about the same coefficient of expansion, whilst the nickel-chromium steels have a relatively high coefficient, and in all cases the coefficient increases with rise in temperature.

H. F. GILLBE.

#### Alloys for use at high temperatures. Nickel-chromium and complex iron-nickel-chromium alloys.

I. W. ROSENHAIN and C. H. M. JENKINS. II. C. H. M. JENKINS, H. J. TAPSELL, C. R. AUSTIN, and W. P. REES (J. Iron Steel Inst., 1930, 121, 225—236, 237—314).—Alloys and steels containing up to 60% Cr together with nickel and, in some cases, other elements have been examined with regard to their endurance under load at 800° or 650°. Under conditions of stable equilibrium a second constituent appears besides the nickel-rich solid solution in binary chromium-nickel alloys at 800° when the chromium content reaches 40%, and the mechanical strength diminishes simultaneously. Alloys containing 50—60% Cr possess poor mechanical properties; at 800° the 30% chromium alloy has the maximum endurance, but at 650° a lower chromium content is preferable. A 90/10 alloy containing a little carbon is stronger than the 80/20 alloy. Sand-casting appears to produce a stronger material than chill-casting. The majority of the nickel-chromium-iron alloys investigated consist of nickel-austenitic iron solid solution, but some are influenced by the  $\gamma$ — $\alpha$  transformation and exhibit very high values of the

mechanical properties at the ordinary temperature. Although the behaviour of these alloys is satisfactory at 650°, at 800° and above the binary nickel-chromium alloys are preferable. Deoxidation prior to casting, preferably with magnesium, is essential with the nickel-chromium alloys, and when iron is present in addition the rate of pouring and the temperature of the molten metal are also of great importance. An improvement of the mechanical properties of certain of the ternary casting alloys occurs if carbon, silicon, tungsten, titanium, or molybdenum is present. Thus an alloy containing Cr 30%, Ni 30%, Fe 33.5%, W 4%, C 1.5%, and Si 1% has at 800° a life of 50 days under a stress of 6 tons/in.<sup>2</sup> The strongest of such alloys has a high ratio between the tensile strengths at 800° and at the ordinary temperature. Additions to the ternary alloys increase the hardness and reduce the ductility at high temperatures, and rolling or forging becomes practically impossible, but when such processes were possible the resulting metal had a shorter life than the cast; the ratio of tensile strengths at 800° and the ordinary temperature is relatively low for the rolled alloys. Brinell tests and microscopical examination of various alloys after slight straining and annealing indicate that, in general, cast material is stronger at high temperatures than are the rolled alloys, provided that the temperature of the test approaches the recrystallisation temperature of the metal; at lower temperatures the wrought material is usually the stronger. Investigation of the recrystallisation processes in binary nickel-chromium alloys indicates that, after thorough rolling, only the 70/30 alloy hardens appreciably on re-annealing, whereas the cast alloys all harden markedly when annealed after slight deformation. The recrystallisation temperature of slightly deformed and annealed alloys is 50—100° below that of the cast alloy. Discussion of the difference of endurance of the cast and wrought alloys shows that the cause cannot yet be fully explained. H. F. GILLBE.

**Determination of oxygen in steel.** G. THANHEISER and C. A. MÜLLER (Mitt. Kaiser-Wilh.-Inst. Eisenforsch., Düsseldorf, 1929, 11, 87—94; Chem. Zentr., 1930, i, 3581).—The silica tube must be carefully cleaned before each experiment. After outgassing the graphite crucible and its content of 30—40 g. of electrolytic iron, the blank value is determined; the sample is added after the fused mass has been cooled nearly to the f.p., and the blank value is redetermined after the experiment. The fused mass must not be used repeatedly. A. A. ELDRIDGE.

**Determination of silicon in steel and iron.** H. WOLF and R. HEILINGÖTTER (Chem.-Ztg., 1930, 54, 878—879).—Complete separation of the silicon from steel cannot be effected by a single evaporation with hydrochloric acid or with sulphuric acid by any of the published methods. At least four evaporations are required with intermediate filtrations. Tests on a steel containing 4.12% Si showed that the results after one evaporation were uniformly 0.12% low, whatever modifications in the dissolution of the sample and evaporation of the solution were used.

A. R. POWELL.

**Electrometric titration of chromium in steel and ferrochromium.** F. SPINDECK (Chem.-Ztg., 1930,



54, 890).—The steel (0.5–3 g.) is dissolved in 16% sulphuric acid and the chromium oxidised with persulphate and silver nitrate, any permanganic acid destroyed by boiling with a little 1:1 hydrochloric acid, and the cold solution titrated electrometrically with ferrous ammonium sulphate solution after addition of manganese sulphate. Fischer's type of stirring apparatus is used together with two platinum electrodes and an Emich filter tube filled with a solution made by mixing solutions of 2.926 g. of ammonium vanadate and 4.9 g. of ferrous ammonium sulphate with 50 c.c. of 1:5 sulphuric acid and diluting to 500 c.c. A. R. POWELL.

**Etching figures in iron and steel.** V. N. SVETCHNIKOV (Rev. Mét.: 1930, 27, 512).—In an additional note (cf. B., 1930, 1113), the author discusses work by Portevin (B., 1923, 781 A). Special reference is made to effects of coring in solid solutions on the regularity of etching figures. E. H. BUCKNALL.

**Economical scaling of sheet iron.** E. DWORCZAK (Przemysl Chem., 1930, 14, 361–373).—For cleaning 1 ton of sheet iron, 1 mm. thick, with sulphuric acid, 40 kg. of 15% acid are required, and 2.3% of the metal is dissolved; the addition of such preparations as naphthalenesulphonic acid or "Vogel's economiser" inhibits the reaction. In the presence of the latter reagent only 17 kg. of sulphuric acid are required, and only 0.78% of iron is dissolved. Scaling at the optimum temperature (30°) is complete in 30 min.

R. TRUSZKOWSKI.

**Chemical methods of protecting metals and alloys from corrosion.** J. CURNOT and J. BARY (Rev. Mét., 1930, 27, 479–485).—A review of known methods. E. H. BUCKNALL.

**Activation by copper sulphate in the flotation of blende.** M. MORTENSON (Tidsskr. Kjem. Berg., 1930, 10, 118–119).—The time required for the activation of blende by copper sulphate solution depends on the iron content of the mineral, being longest for blende rich in iron (marmatite). The rate of activation also increases with temperature and with the presence of acid in the liquid. The electrical conductivity of blendes varies greatly with the iron content; a pure iron-free blende from Spitsbergen showed a resistance of  $6 \times 10^6$  ohms per cm.<sup>3</sup>, whilst that of marmatite from Røros containing 15.4% Fe was only  $8 \times 10^{-1}$  ohm.

H. F. HARWOOD.

**Refining of Alagirski distilled zinc.** N. P. ASEEV, V. V. DOLIVO-DOBROVOLSKI, and B. F. GRASCHTSCHENKO (Coloured Metals, 1930, pp. 40).—The most suitable temperature for the liquation of distilled zinc (Fe 0.09–0.193, Pb 1.13–2.12, Cd 0.024–0.231%) from the Alagirski works is about 450°, the period of heating depending on the degree of impurity. The minimum content of lead in the purified zinc was 0.83%, which is considerably below the limits established by Spring and Romanoff (1.5%) or Rössler and Edelmann (1.7%). Metallographic investigation of the zinc to be liquated shows that, with 0.06% Fe or more, crystals of FeZn<sub>7</sub> are developed, and that solid solutions of iron in zinc are practically absent. This result is in discord with the earlier view, that solid solutions containing up to 0.7% Fe exist, but is confirmed by the work of Ogawa and

Murakami (A., 1929, 141). Micrographs taken after liquation show a characteristic development and accumulation, both of crystals of FeZn<sub>7</sub> and of lead alloys. Repeated liquation yields no noteworthy results. Redistillation of the Alagirski zinc yields a highly pure metal containing 0.03–0.05% Pb, but only when definite conditions of temperature, pressure, amount of distillate, condensation, etc. are observed. Electrolytic refining of the zinc is not practicable, owing to the lack of cheap electrical energy. T. H. POPE.

**Influence of temperature on the toughness of aluminium alloys.** T. KOBAYASHI (Mem. Ryojun Coll. Eng., 1930, 3, 145–153).—The variations with temperature of the toughness of a number of commercially important aluminium alloys has been determined by an impact method for the interval 20–550°.

H. F. GILLBE.

**Rapid determination of tin and antimony in alloys with a high copper content.** A. M. BELOUSOV (J. Appl. Chem., Russia, 1930, 3, 437–439).—The sample is dissolved in nitric acid, the solution being treated at the b.p. with 3% ammonium nitrate solution, boiled, and kept on the water-bath for 1 hr. The mixture is centrifuged, the washings and the sediment being heated with hydrochloric acid; either tin or antimony is determined volumetrically. Low results are obtained for antimony in the absence of tin; hence addition of a known quantity may be necessary.

CHEMICAL ABSTRACTS.

**Determination of cadmium in mill and smelter products.** W. E. KECK, G. L. OLDRIGHT, and F. K. SHELTON (Univ. Utah Tech. Paper, 1930, No. 12, 15 pp.).—The sample is dissolved in a mixture of nitric acid and potassium chlorate, the residue being evaporated with hydrochloric acid. The residue from this is dissolved in dilute sulphuric acid and treated with coarse iron filings. After boiling, filtering, and washing the residue, hydrochloric acid is added to the filtrate and cadmium is precipitated by boiling with ammonium sulphide; it is redissolved and reprecipitated, finally being redissolved and reprecipitated as cadmium ammonium phosphate and weighed as the pyrophosphate.

CHEMICAL ABSTRACTS.

**Electrodeposition of chromium.** V. P. ILINSKI, N. P. LAPIN, and L. N. GOLTZ (J. Appl. Chem., Russia, 1930, 3, 309–320).—Increase in the concentration of sulphate ions above 3 g. of chromic sulphate per litre decreases the current efficiency, improves the lustre and probably the hardness of the deposited metal, and widens the permissible limits of current density.

CHEMICAL ABSTRACTS.

**Electrolytic production of antimony.** F. VOGEL (Metallbörse, 1930, 20, 537–538, 650–651; Chem. Zentr., 1930, i., 3349–3350).—Optimal conditions are specified. A solution containing sulphuric acid (100–160 g.), hydrofluoric acid (20 g.), and antimony (200 g. per litre) may be used. The anodes (95–97% Sb) may contain lead, tin, iron, nickel, zinc, copper, bismuth, and the noble metals as impurities. A. A. ELDRIDGE.

**Determination of bismuth in lead ores by internal electrolysis.** E. M. COLLIN (Analyst, 1930, 55, 680–682).—The method described previously (B., 1930, 773)



has been modified so that it may be used in the case of ores without preliminary smelting, the difference being in the pretreatment of the ore to effect its decomposition and dissolution. The ore is dissolved in concentrated hydrochloric acid, with or without the help of zinc, and, after evaporation to dryness, the residue is taken up with dilute (1:1) nitric acid, hot water is added, and the liquid is filtered. After the addition of potassium permanganate (until no longer decolorised) and hydroxylamine hydrochloride, the solution is electrolysed and the combined bismuth and copper deposit is separated as before; the bismuth is then dissolved in 1:1 nitric acid and the solution electrolysed. If less than 1 mg. Bi is present, it is determined colorimetrically. D. G. HEWER.

**Friction of dry solids *in vacuo*.** SHAW and LEAVEY. —See I. **Coke fines [for zinc smelting].** DAMM and WESEMANN. —See II. **Magnesite.** DWORZAK. —See VIII. **Iron and steel in cement manufacture.** ANON. —See IX. **Photoelectric process control.** STYER and VEDDER; McMASTER. —See XI.

## PATENTS.

**Smelting of ores [in a blast furnace] and recovering by-products therefrom.** F. T. SNYDER (U.S.P. 1,755,845, 22.4.30. Appl., 8.6.25).—The ore is smelted with bituminous coal in a blast furnace under such conditions that the volatile distillation products of the coal are recovered by passing the gases through suitable condensers and scrubbers. To prevent caking in the upper zones a large part of the uncondensed gases from the scrubbers is passed back through the upper zones of the furnace so as to remove therefrom the more readily condensable distillates of the coal. A recovery of over 100 lb. of ammonium sulphate per ton of coal used is claimed, owing to the presence of lime in the charge.

A. R. POWELL.

**Manufacture of iron and steel.** H. HAGEMANN (B.P. 336,954, 18.7.29).—Pig iron is blown until the carbon is reduced to 0.02%, tapped into the ladle, and treated with an alloy of iron with 18–22% Al and 40–50% Si until completely deoxidised. Just prior to casting, manganese low in carbon is added to bring the composition of the cast metal up to 0.5% Mn, 0.02% C, 0.09% Cu, and only traces of silicon.

A. R. POWELL.

**Production of slags for use in the manufacture of wrought iron.** A. M. BYERS Co., Assees. of A. H. BEALE, H. A. BRASSERT, and F. WILLE (B.P. 315,858, 20.6.29. U.S., 21.7.28).—Slag for use in the Aston process of making wrought iron is prepared by melting tap cinder and iron roll-scale under non-reducing conditions in a hearth furnace, the charge being so proportioned that the product contains 60–75% FeO, 5–15% Fe<sub>2</sub>O<sub>3</sub>, and 10–12% SiO<sub>2</sub>. A. R. POWELL.

**Moulding of liquid or plastic iron, steel, and other metals and alloys difficult to mould.** P. MÜLTHAUP (B.P. 336,688, 26.7.29).—The moulding appliances consist of cast, sintered, or pressed tungsten.

A. R. POWELL.

**Manufacture of magnetic [iron-nickel] alloys.** W. S. SMITH, H. J. GARNETT, and W. F. RANDALL (B.P. 336,948, 22.6.29).—Alloys of iron with 30–50% Ni,

up to 10% Cr or Mn, and free from carbon are annealed completely, cooled, subjected to a straining operation, e.g., winding on a conductor, and reannealed at a temperature between 500° and 700° at least 50° above the magnetic transformation point. A. R. POWELL.

**Chromium or chromium-nickel steel alloys.** F. KRUPP A.-G. (B.P. 337,349, 25.7.29. Ger., 26.6.29).—Alloys containing 0.07–1% C, 18–25% Cr, 7–12% Ni, and more than 2% V and/or Ti are claimed. The amount of vanadium and/or titanium added is at least twice the carbon content, the function of these metals being to combine with the carbon and thus stabilise the austenitic structure of the steel. A. R. POWELL.

**Production of titanium steel.** W. and H. MATHE-SIUS (B.P. 337,715, 12.3.30. Ger., 16.3.29).—Molten steel after decarburisation and deoxidation is tapped in a strong stream from one ladle into another containing briquettes of a titanium thermic mixture.

A. R. POWELL.

**High-speed tool-steel alloy.** G. R. COULS (U.S.P. 1,756,282, 29.4.30. Appl., 1.9.27).—Steel containing substantially 20% W, 1% Cu, 4% Cr, 1.5–2.5% V, and 73% Fe is claimed.

A. R. POWELL.

**Electrodes or rods for welding [non-rusting steel].** J. H. ATTCHISON, H. BULL, and L. JOHNSON (B.P. 337,589, 25.10.29).—The rod is prepared by drawing down a billet consisting of a shell of chromium steel containing 20% Cr and 0.1% C surrounding a core of nickel, the weight of the core being about 10% of that of the steel. When it is desired to introduce other elements into the weld, e.g., tungsten, molybdenum, or manganese, the nickel core is provided with a sleeve of steel containing a high percentage of these elements.

A. R. POWELL.

**Protection of iron and steel. Cementation processes for ferrous metals.** A. FOLLIET and N. SAIN-DERICHIN (B.P. 337,562 and 337,635, [A] 10.10.29, [B] 28.11.29).—(A) Iron or steel articles are heated at 850–1050° in a mixture of fine-grained aluminium, granular carborundum, 2–5% of the chloride of chromium, nickel, or manganese, and 1–2.5% of ammonium chloride, whereby a coating of aluminium containing chromium, nickel, or manganese is produced which resists scaling up to 1500°. (B) The steel is heated in a mixture of chromite, granulated aluminium, and chromic chloride, whereby the outer layer is converted into a chromium steel. The chromate may be replaced by ilmenite, calcined pyrolusite, garnierite, or wolfram for the production of cemented layers of titanium, manganese, nickel, or tungsten steels, respectively.

A. R. POWELL.

**Inhibitor material [for use in steel pickling].** L. J. CHRISTMANN, Assr. to AMER. CYANAMID Co. (U.S.P. 1,755,812, 22.4.30. Appl., 18.9.29).—Tetramethyldi-aminodiphenyl disulphide is claimed as an inhibitor in sulphuric acid pickling baths. A. R. POWELL.

**[Inhibitor for use in baths for the] cleaning and protection of metal surfaces.** J. C. VIGNOS, Assr. to RUBBER SERVICE LABS. Co. (U.S.P. 1,750,651, 18.3.30. Appl., 19.11.27).—An inhibitor for sulphuric acid pickling baths comprises a sulphonated arylthiazole com-



pound, *e.g.*, the aldol condensation product of aniline hydrochloride, ethylene glycol, and a sulphonated mercaptobenzthiazole. A. R. POWELL.

**Production of coated metal bodies [galvanised iron].** F. B. DEHN. From AMER. ROLLING MILL CO. (B.P. 337,399, 29.7.29). The galvanising bath contains zinc alloyed with 3–20% Al, preferably 8%, and is floated on a lead bath through which the pickled steel sheets are passed first. After passing through the galvanising bath the treated sheets are heated at a temperature just above the m.p. of the coating to produce a finely-crystallised structure in the coating. A. R. POWELL.

**Manufacture of coated iron and steel articles.** E. M. JONES, ASSR. to PARKER RUST-PROOF CO. (U.S.P. 1,750,270, 11.3.30. Appl., 13.6.27).—The articles are pickled and boiled in a bath containing acid phosphates of iron, zinc, or manganese, together with finely-divided silica in suspension; a layer containing phosphate and silicate of iron is formed. A. R. POWELL.

**Coated metal [iron] and its manufacture.** F. W. DE JAHN and J. G. DELY, ASSRS. to CHEM. RES. & DESIGNING CORP. (U.S.P. 1,755,686, 22.4.30. Appl., 10.10.29).—Sheet iron or steel is passed through a bath of molten lead containing 0.05–0.5% Bi, 0.05–0.2% Cu, up to 0.1% Hg, and 3–8% Sn. At the point of entry of the metal into the bath the latter is covered with a layer of fused zinc chloride and at the lowermost portion of the bath the coated metal is subjected to the forcible impingement of jet currents to dislodge adhering particles of foreign matter. A. R. POWELL.

**Manufacture of [steel] articles hardened by nitrogenisation.** F. KRUPP A.-G. (B.P. 312,349 and 337,404, 29.4.29. Ger., 2.5.28).—Articles of steel containing (A) 0.4–0.5% C, 0.25–0.35% Si, 0.5–0.6% Mn, 1.2–1.5% Cr, 0.3% Mo, and 0.5% V, or (B) 2% C, 12% Cr, 0.8% W, and up to 1% Si, Mn, V, and/or Mo, are heated at 500° in ammonia for 70 hrs. A. R. POWELL.

**Separation of zinc, tin, lead, bismuth, cadmium, antimony, arsenic, and similar elements from sulphide ores or secondary products by means of blast-smelting.** E. J. KOHLMAYER (B.P. 336,670, 19.7.29).—A mixture of zinc or lead sulphide ore with pyrites is melted in a rotating drum-furnace the length of which is equal to its diameter, so that the heating flame passes forward and backwards through the furnace. The slag is skimmed off and the charge blown with air, whereby the zinc or lead is oxidised and volatilised, leaving a residue of ferrous sulphide. By continuing the air blast and occasionally adding small quantities of lime the iron may be completely converted into calcium ferrite, which is suitable for smelting in the blast furnace for the recovery of its iron content. The method is also applicable to the recovery of tin, bismuth, cadmium, arsenic, and antimony from pyritic ores. Addition of a small quantity of iron to the charge after removing the slag accelerates the removal of the volatile metal. A. R. POWELL.

**Reduction of zinciferous materials.** E. H. BUNCE and G. T. MAHLER, ASSR. to NEW JERSEY ZINC CO. (U.S.P. 1,749,126—7, 4.3.30. Appl., 4.1.28).—(A) Roasted

zinc ore is reduced with carbon in the usual way and the gases and vapours are passed through a briquetted mass of zinc oxide and carbon or through a tower packed with fireclay and/or carbonaceous material, at such a temperature that the lead and other impurities in the zinc vapour are condensed and entrapped in the porous mass while the zinc vapour passes to the usual condensing system. (B) The operation is carried out in a single continuous operation by charging the briquetted mass of zinc oxide and carbon into the top of a vertical retort, maintaining the temperature of the upper layers at 800–900° so that the lead is entrapped therein, and removing the purified zinc vapours from a point adjacent to the charging door. A. R. POWELL.

**Treatment by volatilisation of zinc ores and mixtures thereof.** A. FOLLIET and N. SAINDERICHIN (B.P. 337,636, 28.11.29. Fr., 19.10.29).—Crushed zinc sulphide ore is passed in a thin layer through a furnace into which is blown a blast of air at 650–800°; in the first zone oxidation and removal of the sulphur is effected, and in the second carbon is added to cause reduction and volatilisation of the zinc. A. R. POWELL.

**[Zinc] alloy for bearing surfaces.** H. C. HALL (B.P. 336,881, 30.12.29).—The alloy comprises zinc with 8–18% Al, 0–1% Cu, and 0–1% Ni, and is suitable for use up to 200°. A. R. POWELL.

**Utilisation of dolomite or magnesian limestone for production of magnesium etc.** H. H. DOW and E. O. BARSTOW, ASSRS. to DOW CHEM. CO (U.S.P. 1,749,210—1, 4.3.30. Appl., [A] 30.10.26, [B] 8.11.26).—(A) Powdered dolomite made into a slurry with water is treated with the chlorine produced in the electrolytic magnesium cell and with sulphur dioxide, whereby a solution of magnesium chloride and a residue of calcium sulphate are obtained. (B) Chlorine from the cell is caused to react with sulphur dioxide in the presence of water and the resulting solution of hydrochloric and sulphuric acids is treated with calcium chloride to remove the latter acid. The resulting hydrochloric acid solution is neutralised with dolomite and heated with sufficient calcined dolomite to precipitate all the magnesium as hydroxide, which is washed free from calcium compounds and dissolved in a further portion of the hydrochloric acid to obtain magnesium chloride. The calcium chloride produced is returned to the first stage of the operation. A. R. POWELL.

**Refining of magnesium and its alloys.** I. G. FARBENIND. A.-G. (B.P. 336,498, 13.12.29. Ger., 2.1.29).—Iron may be removed from magnesium and its alloys by stirring into the molten alloy at 900° about 1.5% of finely-powdered manganese and keeping the mixture at 700° for 2 hrs., whereby the iron and manganese settle to the bottom of the pot and the purified magnesium may be decanted off; it contains less than 0.02% Fe. A. R. POWELL.

**Improving the mechanical properties, especially the compression yield point, of articles made from magnesium and its alloys.** I. G. FARBENIND. A.-G. (B.P. 337,706, 20.2.30. Ger., 11.3.29).—The metal or alloy is extruded into rods at 300–350° at a rate between



half the normal rate and the full normal rate and the metal is quenched immediately it issues from the press.

A. R. POWELL.

**Casting of magnesium and its alloys.** J. E. HOY, Assr. to DOW CHEM. CO. (U.S.P. 1,751,038, 18.3.30. Appl., 3.7.26).—The air in the mould is replaced by sulphur dioxide and sulphur vapour by igniting a quantity of sulphur in the sprue just prior to casting the metal.

A. R. POWELL.

**Light metal [aluminium] alloy.** H. T. TILLQUIST and J. HÄRDÉN (B.P. 337,099, 17.8.29).—Aluminium-chromium alloys are produced by melting aluminium and chromium in hydrogen or in a vacuum in a high-frequency induction furnace. The alloys are suitable for introducing small quantities of chromium into aluminium or other aluminium alloys.

A. R. POWELL.

**Light-metal [aluminium] alloy.** BIRMINGHAM ALUMINIUM CASTING (1903) CO., LTD., and P. PRITCHARD (B.P. 337,558, 9.10.29).—An alloy of aluminium with 3.5% Mg and 0.5% Mn is claimed. When subjected to anodic oxidation a dense, strong, and adherent film is produced which is highly resistant to corrosion.

A. R. POWELL.

**Casting of molten metals or alloys [of lead].** H. HARRIS (B.P. 336,727, 27.8.29).—The molten lead or lead alloy is fed from an overhead melting vessel through a series of nozzles into the space between two parallel horizontal drums revolving in opposite directions in such a way that the metal is in a plastic semi-solid state as it is squeezed between the drums, and issues therefrom in the form of a sheet.

A. R. POWELL.

**Treatment of complex ores of copper, cobalt, and nickel.** R. S. CARRERAS (B.P. 336,584, 10.7.29).—The ore is dissolved anodically in dilute sulphuric acid containing 5% of sodium chloride and the solution is electrolysed for the recovery of copper, using insoluble anodes. The electrolyte is then evaporated fractionally for the recovery of cobalt and nickel sulphates from which the metals are recovered by electrolysis.

A. R. POWELL.

**Smelting of [chromium, manganese, and vanadium] ores.** ELECTRO METALLURG. CO., ASSEES. OF F. M. BECKET (B.P. 309,594, 10.4.29. U.S., 13.4.28).—Ferro-alloys of the above metals are obtained by the silicothermic reduction of their oxides or alkaline-earth salts in a self-propagating reaction mixture of such a composition that the slag formed contains a relatively high percentage of the desired metal which is subsequently recovered by reduction with carbon in an electric furnace. *E.g.*, a mixture of vanadium pentoxide, ferro-vanadium-silicon, lime, steel scrap, and sodium nitrate is ignited to obtain ferrovanadium containing 35% V, 7% Si, and <0.3% C, and a slag containing about 10% V. The slag is then smelted with carbon to produce the ferrovanadium-silicon alloy for the first operation.

A. R. POWELL.

**Working up of chromium ores.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 336,970, 22.7.29).—Chromite is mixed with 1–2% of sodium nitrate and calcined in air at 800–1100° for 30 min. prior to the usual roasting treatment with lime and sodium carbonate.

A higher extraction of chromium is obtained in a shorter time after the pretreatment.

A. R. POWELL.

**Manufacture of hydrocarbon-containing metals and alloys.** BAYERISCHE METALLWERKE A.-G. (B.P. 316,103, 22.7.29. Ger., 20.7.28).—Tungsten, chromium, vanadium, or titanium, or their alloys or oxides are heated below 2500°, preferably at 1400–1600°, in an atmosphere of hydrocarbon vapour, *e.g.*, acetylene, benzene, or naphthalene, containing 1–2% of ammonia, whereby a hard surface layer is produced suitable for metal-cutting or wire-drawing.

A. R. POWELL.

**Manufacture of tungsten.** J. GRAY. From A. PACZ (B.P. 337,160, 3.10.29).—A solution of sodium tungstate is mixed with silver nitrate and the mixture is poured into hydrochloric acid to obtain a mixture of tungstic acid and silver chloride. After washing and drying, the powder is mixed with thorium nitrate and reduced in hydrogen. The resulting metal powder is pressed into bars which are sintered in hydrogen under such conditions that the whole of the silver is removed by volatilisation. The ingots are then swaged and drawn into wire as usual. The silver used in the process should be 0.5–10%, and the thorium about 1.5%, of the tungsten used.

A. R. POWELL.

**[Tungsten-cobalt] alloy for cutting tools.** B. E. FIELD, Assr. to HAYNES STELLITE CO. (U.S.P. 1,750,796, 18.3.20. Appl., 29.8.25. Renewed 11.1.29).—The alloy contains at least 35% W and 30% Co together with up to 12% Cr and 0.20–1.25% B.

A. R. POWELL.

**Dressing of residues containing metal.** HIRSCH, KUPFER- U. MESSINGWERKE A.-G. (B.P. 319,327, 13.9.29. Ger., 20.9.28).—The material is ground and washed or air-flegated to remove dross etc. and leave a residue of fairly clean metal which is melted in an induction furnace. The dross etc. is smelted separately or treated chemically to recover its content of valuable metal.

A. R. POWELL.

**Casting of metals.** I. SUGIMURA, K. MAGARISAWA, and M. YASUMOTO (B.P. 337,286, 8.1.30).—Two immiscible metals, such that one has a lower m.p. and higher  $d$  than the other, are melted together, stirred, and allowed to settle until the lighter metal floats in a liquid mass just below the surface of the heavier. On casting the mixture the heavier metal forms a tightly adherent sheath around a core of the lighter metal. Examples of such pairs of metals are cast iron-copper, cast iron-lead, zinc-lead, and aluminium-lead; the first-named metal in all cases forms the core of the casting.

A. R. POWELL.

**Production of age-proof welds by the fusion-welding method.** F. KRUPP A.-G. (B.P. 316,555, 25.7.29. Ger., 30.7.28).—The welding wire used is an alloy of iron with 0.1% Al and 0.6% Mn or other deoxidising agents, 1% Ni, about 1% Cr, and 0.4% Mo or 1% W. The wires are preheated at 800–1000° in hydrogen to eliminate oxygen, sulphur, carbon, and phosphorus.

A. R. POWELL.

**Welding of bronze.** L. J. TIBBENHAM (B.P. 336,402, 1.10.29).—The welding rod is coated with a mixture of 49% of boric acid, 9% of borax, 13% of iron oxide, 17% of cupric oxide, and 12% of sodium silicate solution.

A. R. POWELL.



**Sealing or joining of metal objects to insulating material.** S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 337,491, 12.8.29).—The metal objects comprise ring- or disc-shaped parts composed of an alloy of 50–90% Fe with one or more of the elements tungsten, molybdenum, tantalum, niobium, cobalt, or silicon, and up to 5% Cr, Al, or Mg. The composition of the alloy is such that its coefficient of expansion is within  $\pm 20\%$  of that of glass.

A. R. POWELL.

**Electrodeposition of chromium.** C. H. PROCTOR, Assr. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,749,443, 4.3.30. Appl., 9.3.29).—A bath containing (per gal.) 8–48 oz. of chromium trioxide, 0.08–0.5 oz. of sulphuric acid, and 0.25–1.25 oz. of "finely-ground chrome-iron ore" is claimed.

A. R. POWELL.

**Stripping of chromium plating.** TERNSTEDT MANUFG. CO., Assees. of J. F. McCULLOUGH (B.P. 336,532, 11.2.30. U.S., 6.3.29).—The articles are treated anodically at 12 volts in a 6–12% solution of oxalic, citric, or tartaric acid at 25–30°.

A. R. POWELL.

**Electrolytic production of light metals [magnesium].** E. O. BARSTOW, Assr. to DOW CHEM. CO. (U.S.P. 1,755,380, 22.4.30. Appl., 9.11.25).—The electrolysis is conducted in a steel vessel which acts as the cathode and is provided with a centrally disposed carbon anode surrounded by a fireclay diaphragm. Outside this diaphragm is a rectangular box the end walls of which are submerged below the surface of the electrolyte, whilst the side walls extend well above the surface. The box is provided with two transverse partitions, one extending well below the other, and outside the longer partition a propeller is fixed so that its motion draws the surface layers of the electrolyte through the box in such a way that the suspended particles of magnesium are trapped between the two partitions in the box and there caused to coalesce to a mass of molten metal.

A. R. POWELL.

**Tilting gear for crucible furnaces.** J. H. LUDLOW, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 338,013, 25.11.29).

**[Sand-moulds for] casting of stainless steel.** W. E. MARTIN and J. A. BERLYN (B.P. 337,413, 29.7.29).

**Catalysts for ammonia oxidation** (B.P. 337,680). **Non-corrosive brine** (U.S.P. 1,750,761). **Magnesium chloride** (U.S.P. 1,756,247). **Sulphur dioxide from smelter gases** (U.S.P. 1,758,398).—See VII. **Enamel ware** (B.P. 336,797).—See VIII.

## XI.—ELECTROTECHNICS.

**Electrical method and instrument for measuring liquid consistency.** C. E. FAWKES (Amer. Paint & Varnish Manufs.' Assoc., Oct., 1930, Circ. 370, 543–547).—The consistency load impressed on a submerged rotor impeller turning in the liquid under standard conditions is electrically indicated on a visual dial. From the readings obtained and the sp. gr. of the liquid, viscosity in c.g.s. units can be calculated.

S. S. WOOLF.

**Process control with the "electric eye" [photoelectric cell].** C. A. STYER and E. H. VEDDER (Ind. Eng. Chem., 1930, 22, 1062–1069).—Various types of

photoelectric cells are described. These are suitable, when used in conjunction with the necessary current-amplifying devices, for, *e.g.*, indicating and recording the density of smokes, controlling the turbidity of thickener overflow in the concentration of metallurgical slimes, counting such objects as hot ingots or bags of salts etc. discharged from a conveyor, or for use in the paper industry. Other examples given, with quantitative data, include the automatic recording of concentration of copper sulphate solution, by colour changes, and of sulphuric acid, by refractive index. C. IRWIN.

**Photoelectric cells in chemical technology.** A. J. McMASTER (Ind. Eng. Chem., 1930, 22, 1070–1073).—The characteristics of vacuum and gas-filled photoelectric cells are described (*cf.* preceding abstract). The latter are the more sensitive and the former the more constant. Relations of cell currents and light units are given, together with the variation of effect with varying wave-length of light. Relays are also referred to. Whenever cells are used for quantitative measurements, regular calibration from a standard is necessary. They may be used in the measurement of optical properties of materials, or the determination of the light-transmissive power, reflecting power, or absorption factor of metals, glass, tiles, other ceramic materials, paints, varnishes, cellulosic products, etc.

C. IRWIN.

**Gas-analysis apparatus.** JARRIER.—See I. **Nitrogen-fixation plant.** WESTBY.—See VII. **Titration of chromium in steel and ferrochromium.** SPINDECK. **Chromium.** ILINSKI and others. **Antimony.** VOGEL. **Bismuth in lead ores.** COLLIN.—See X. **Electrical properties of rubber.** CURTIS and others.—See XIV. **Conductometer for sugar factories.** ŠANDERA.—See XVII. **Universal thermostat.** FINK. **Quartz lamp and wine analysis.** HEIDUSCHKA and MÖHLAU.—See XVIII.

## PATENTS.

**Electric-furnace resistor.** F. A. FAHRENWALD (U.S.P. 1,755,350, 22.4.30. Appl., 19.5.24).—A cast-metal resistor consisting of a sinuous grid having straight legs of ribbed section, connected at alternate ends by integral loops, some of which have outwardly-extending prongs, is claimed.

J. S. G. THOMAS.

**Metal-arc and metal-vapour lamps. Electric-discharge tubes.** S. G. S. DICKER. From N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 336,208 and 336,222, 5.7.29).—(A) In an electric-discharge tube containing a mixture of metals or an alloy, *e.g.*, cadmium amalgam, which preferably forms an electrode of the tube, a channel separated from the discharge space is provided to return the condensed metal to the part of the tube containing the alloy or mixture and deliver it below the surface thereof. (B) To prevent deposition of metallic vapour on the tube wall, that part of the wall to which metal is liable to adhere is coated with, *e.g.*, calcium fluoride, which prevents such deposition. Alternatively, the tube wall may be coated with potassium chloride, which reacts with deposited metallic vapour, *e.g.*, magnesium, to form products which are volatile or do not absorb the radiation emitted by the discharge.

J. S. G. THOMAS.



**Manufacture of thermionic electrodes of vacuum-electric tube devices.** WESTINGHOUSE LAMP CO., Assees. of M. N. FREDENBURGH (B.P. 315,324, 11.7.29. U.S., 11.7.28).—Alkaline-earth carbonates precipitated in a predetermined minimum time at or above 85°, from an approx. 15% solution of soluble salts, *e.g.*, the nitrates of barium and/or strontium and calcium, the last-named being present to the extent of 10–20% of the total anhydrous salts, by addition of a solution of *N*-ammonium carbonate, are converted into oxides, which are applied to the electrodes. J. S. G. THOMAS.

**[Electrode for] photosensitive cells.** ARCTURUS RADIO TUBE CO., Assees. of S. RUBEN (B.P. 336,397, 30.9.29. U.S., 11.1.29).—A copper electrode, coated with cuprous oxide by heating at about 1000° and etched by a solution of ammonium chloride, is partly encased in an insulating compound, *e.g.*, beeswax and resin. J. S. G. THOMAS.

**Manufacture of coloured electric glow lamps.** I. G. FARBENIND. A.-G. (B.P. 315,313, 5.7.29. Ger., 11.7.28).—Lamps are varnished with a solution of a polymerised vinyl ester in an organic solvent containing a colouring matter, *e.g.*, aminoazobenzene. J. S. G. THOMAS.

**Cement for caps for electric incandescence lamps etc.** GEN. ELECTRIC CO., LTD., Assees. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 316,866, 5.7.29. Ger., 4.8.28).—A fatty drying oil, *e.g.*, linseed or tung oil, which is but little decomposed by heat, together with, if desired, hexamethylenetetramine or other condensing agent for artificial resins, is added to cements consisting of a spirit solution of artificial resins and a filler. J. S. G. THOMAS.

**Alkaline storage battery.** J. F. MONNOT (U.S.P. 1,780,935, 11.11.30. Appl., 31.7.28. U.K., 4.11.27).—See B.P. 297,996; B., 1928, 864.

**Composition of matter. [Insulating material.]** M. DARRIN, Assr. to F. N. BURT CO., LTD. (Re-issue 17,867, 11.11.30, of U.S.P. 1,644,711, 11.10.27).—See B., 1928, 129.

**Luminous electric-discharge tubes.** GEN. ELECTRIC CO., LTD., Assees. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 336,490, 4.12.29. Ger., 13.7.29).

**Light-sensitive devices.** COMP. DES LAMPES (B.P. 377,477, 6.8.29. Fr., 10.4.29).

**Thermostats** (B.P. 337,093). **Measuring humidity** (B.P. 336,855).—See I. **Pure *n*-butyl alcohol** (B.P. 335,683).—See III. **Purified fibre** (U.S.P. 1,757,768). **Sheet material** (U.S.P. 1,757,010).—See V. **Magnetic alloys** (B.P. 336,948). **Welding electrodes for steel** (B.P. 337,589). **Magnesium** (U.S.P. 1,749,210—1 and 1,755,380). **Light metal alloy** (B.P. 337,558). **Treatment of complex ores** (B.P. 336,584). **Metal-insulation joints** (B.P. 337,491). **Chromium** (U.S.P. 1,749,443 and B.P. 336,532).—See X.

## XII.—FATS; OILS; WAXES.

**Determination of butter fat.** E. SCHWEIZER and J. GROSSFELD (Z. Unters. Lebensm., 1930, 59, 494—501).—During the course of three years a number of

samples of butter, margarine, and mixtures of the two have been examined to determine the constancy of the Reichert–Meissl value and the butyric acid value. The mean percentage deviation from the normal values of 27.0 and 20.0 were found to be  $\pm 10.0$  and  $\pm 7.4$ , respectively, for samples of normal butter. The mean of the Reichert–Meissl values (37 samples) was 28.7, which corresponds closely with the van Raalte value of 28.4. The ratio of the butyric acid value to the Reichert–Meissl value was found to exceed in all cases the value 0.66 (mean of 37 samples, 0.713), and thus confirms van Raalte's observation that for pure butter the "xylol percentage number" should exceed 66.0. In addition to these characteristic figures, the values of Juckenack's difference (saponification value — Reichert–Meissl value — 200) and Grossfeld's difference (saponification value — 1.5  $\times$  butyric acid value) have been studied and their significance in the detection of adulteration is exemplified. H. J. DOWDEN.

**Which drying process gives a reliable determination of fatty acids in soap?** J. DAVIDSOHN (Chem. Umschau, 1930, 37, 293—296).—The volatility of the fatty acids of the kernel oils leads to inaccuracy in this determination. Mixtures of dried coconut and tallow fatty acids were studied under various conditions of temperature etc. The losses at 100° are too great to permit the use of this temperature in drying, and are not reduced by the admixture of paraffin wax. Goldschmidt's second method (Z. Deuts. Oel- u. Fett-Ind., 1920, 40, 407), in which the desiccated ethereal extract of the fatty acids is evaporated to constant weight at 60°, gives reliable results, but the treatment with anhydrous sodium sulphate need not be prolonged beyond 10 min. and the process may be shortened by evaporating the last traces of ether at 50° in an air current. E. LEWKOWITSCH.

**Apparent iodine value of elæostearic acid.** J. VAN LOON (Chem. Umschau, 1930, 37, 269—271).—The curve of apparent iodine value of  $\beta$ -elæostearic acid is similar to that obtained for tung oil (B., 1930, 996) and shows a kink at iodine value 180, corresponding to two-thirds saturation of the acid.  $\beta$ -Elæostearic acid appears to undergo slight polymerisation on keeping under alcohol. There is evidence of the presence in tung oil of a hitherto unknown highly unsaturated acid, which is fully halogenated under the conditions studied. E. LEWKOWITSCH.

**Value of by-products of saccharin manufacture to the oil and fat industry.** W. HERZOG (Chem. Umschau, 1930, 37, 296—298).—The products formed by the condensation of *p*-toluenesulphonamide with fatty acid chlorides, *e.g.*, *p*-toluenesulphopalmitamide, m.p. 100–102°, yield salts with alkalis and alkaline-earths that are of value as emulsifying agents. The preparation and properties of chloroamine, chloramine-T, etc., and their use as preservatives for edible fats (cf. G.P. 408,647; B., 1925, 375) or in disinfectant soaps (cf. G.P. 401,011; B., 1924, 987), or possibly for determination of iodine value (cf. Margosches and Frischer, B., 1927, 727) are described. E. LEWKOWITSCH.

**Chinese tung oil seeds.** ANON. (Bull. Imp. Inst., 1930, 28, 267—272).—Tung oil, extracted from one



sample of nuts from *Aleurites Fordii* and from two samples of *A. montana*, had the following respective characteristics: yield (from moisture-free kernels) 58.5%, 62.6%, 59.8%;  $d_{15}^{25}$  0.9428, 0.9381, 0.9405; acid value 0.44, 12.1, 1.5; saponif. value 192.3, 191.5, 192.6; iodine value (Wijs, 3 hrs.) 179.2, 163.1, 170.0; unsaponifiable matter 0.9, 0.6, 0.7%;  $n_{40}^{20}$  1.5155, 1.506, 1.509; heat test (Browne, min.) 11, 24, 20.5; solidif. point of fatty acids 30.0°, 38.2°, 41.1°. The residual respective meals, as prepared from the kernels, had (%): moisture 10.3, 9.7, 12.8; crude proteins 37.9, 37.9, 47.9; fat 0.4, 1.6, 0.7; carbohydrates etc. (by diff.) 35.9, 33.4, 24.7; crude fibre 7.8, 7.1, 6.8; ash 7.7, 10.3, 7.1%; the nutrient ratios were 1:1.0, 1:1.0, 1:0.5; and food units 132, 132, 146. Meals from the whole nuts are also described.

E. H. SHARPLES.

**Blown linseed oil and sulphur-treated linseed and wood oils.** A. HOLLANDER (Farben-Ztg., 1930, 36, 118—120).—The enhanced water resistance of "vulcanised" and blown oils (cf. B., 1930, 382) is illustrated by further data, experimental details being quoted. The gel formation in such films before drying is a combination of the ordinary "oxypolymerisation" with sulphur adsorption by the "molecular valencies" of the double linkings. The latter effect hinders the oxygen absorption due to the "main valencies," and hence retards drying and degradation processes. The drying of these oils (with the exception of "wood oil fraction") is considered to occur mainly by "desolvation."

S. S. WOOLF.

**Perilla oil.** H. P. KAUFMANN (Allg. Oel- Fett-Ztg., 1930, 27, 39—40; Chem. Zentr., 1930, i, 3735).—The following values were obtained: iodine value (Kaufmann) 204, 204; thiocyanogen value 124, 129. Of the total fatty acids 0.69, 0.66% represents unsaponifiable matter; 7.6, 6.7% saturated acids; 3.9, 10.7% oleic acid; 44.3, 33.6% linoleic acid; and 44.2, 49.0% linolenic acid (20.1, 21.3% of which is in the  $\alpha$ -form). The oil contains unsaponifiable matter 0.7, 0.7; saturated acids 7.2, 6.3; oleic acid 3.7, 10.1; linoleic acid 41.9, 31.9; linolenic acid 41.7, 46.4%.

A. A. ELDRIDGE.

**Sunflower seed from Southern Rhodesia.** ANON. (Bull. Imp. Inst., 1930, 28, 272—276).—Six representative samples of varieties of sunflower seed from S. Rhodesia contained 6.6—7.7% of moisture; the oil contents, on moisture-free seeds, and the respective acid values, were 30.7, 0.8; 29.6, 0.9; 29.3, 0.8; 29.2, 0.8; 26.1, 0.6; 31.8, 0.5%. The first and last compare favourably with the quality usually crushed in Europe.

E. H. SHARPLES.

**Dhupa kernels (*Vateria indica*) from India.** ANON. (Bull. Imp. Inst., 1930, 28, 279—281).—Extraction of the kernels of *V. indica* yielded 23.2% of greenish-white fat (on dry kernels) having  $d_{15}^{20}$  0.8585, m.p. (open tube) 34.2°, acid value 1.0, saponif. value 190.3, iodine value (Wijs, 3 hrs.) 45.9, unsaponifiable matter 0.9%,  $n_{40}^{20}$  1.4588, solidif. point of fatty acids 52.7°. The residual meal had moisture 8.8%, ether extract 4.1%, crude proteins 6.2%, crude fibre 5.0%, ash 2.1%, carbohydrates etc. (by diff.) 73.8%, nutrient ratio 1:13.4,

food units 100. The meal was free from cyanogenetic glucosides and alkaloids, but had a bitter taste which would render it unsuitable for use as a feeding-stuff.

E. H. SHARPLES.

**Moringa aptera seed from Egypt.** ANON. (Bull. Imp. Inst., 1930, 28, 276—279).—The kernels (4.9% of moisture) from the seeds (wt. 0.5 g., 51.2% of shell) of *M. aptera* on extraction yield 50% of oil having  $d_{15}^{20}$  0.9151,  $n_{40}^{20}$  1.461, acid value 0.5, saponif. value 188.2, iodine value (Wijs, 3 hrs.) 71.2, unsaponifiable matter 0.5%, solidif. point of fatty acids 28.1°. The residual meal has moisture 8.7%, crude proteins 48.6%, fat 2.6%, carbohydrates etc. (by diff.) 28%, crude fibre 6.6%, ash 5.5%, nutrient ratio 1:0.7, food units 156. The meal is free from cyanogenetic glucosides, but contains a saponin and, probably, a substance of an alkaloidal nature. Both the oil and meal are very similar to those from *M. pterygosperma* (from which Ben oil is derived).

E. H. SHARPLES.

**American cherry-kernel oil.** G. S. JAMIESON and S. I. GERTLER (Oil & Fat Ind., 1930, 7, 371—372, 387).—The expressed oil (32—40%) from the kernels of the sour cherry (cf. Rabak, B., 1917, 159) after refining was pale in colour and had (figures for the crude oil in parentheses):  $d_{20}^{25}$  0.9183 (0.9176),  $n_D^{25}$  1.4740 (1.4742), acid value 0.09 (4.39), iodine value (Hanus) 115.8 (118.7), saponif. value 190.7, Reichert—Meissl value 0.3, Polenske value 0.2, unsaponifiable matter 0.5% (0.66%), saturated acids (corr.) 7.7% (lead salt-ether method), unsaturated acids (corr.) 87%, iodine value 127.9. The composition of the oil is given as: acids (%): oleic 46.85, linoleic 40.11, myristic 0.19, palmitic 4.04, stearic 2.79, arachidic 0.72. The refined oil, which has a bland flavour and good keeping properties, should be suitable for use as a salad oil and in cosmetic manufacture.

E. LEWKOWITSCH.

**Abutilon oil.** L. JOLSON (Masloboino Zhir. Delo, 1929, No. 8, 22—25).—Cultivated abutilon seeds contain moisture 9.7, oil 16.5, crude protein 19.8, cellulose 16.9, ash 5.35, and nitrogen-free extract 31.75%; the oil has  $d_{20}^{20}$  0.9265, iodine value 131.7, saponif. value 190.6, acid value 4.42, unsaponif. matter 0.80%, and is similar to cottonseed and soya-bean oil. The fatty acids of the oil have f.p. 19.9°, iodine value 124.2, neutralisation value 190.3.

CHEMICAL ABSTRACTS.

**Fatty oil of coriander.** G. NORKIN (Masloboino Zhir. Delo, 1929, No. 8, 25—26).—Coriander seed contains, in addition to the essential oil, a fatty oil having  $d_{15}^{25}$  0.919,  $n_D^{26}$  1.4688, acid value 7.84, saponif. value 192.15, iodine value 93.81, Reichert—Meissl value 0.49; the acids have mol. wt. 291.4,  $n_D^{26}$  1.4678, neutralisation value 196.5, iodine value (Hübl) 97.46. The colour reactions of the oil and the properties of the sodium soap are recorded.

CHEMICAL ABSTRACTS.

**Study of the official method of bleaching test of the American Oil Chemists' Society.** C. MA and J. R. WITTHROW (Ind. Eng. Chem. [Anal.], 1930, 2, 374—377).—The results of experiments with varying conditions justified the manipulative details prescribed in the official test; full bleaching can be obtained, however, in 1 min. at temperatures between 92° and 125.°



On either side of these limits the degree of bleaching decreases. No increased bleaching is obtained by increasing the amount of fuller's earth beyond 7%, or prolonging the treatment beyond 5 min. The Hess-Ives tint photometer (cf. Kress and McNaughton, B., 1916, 449) is useful, and the degree (%) of bleach may be satisfactorily expressed as  $100(T-L)/(100-L)$ , where  $T$  and  $L$  are the total percentage luminosities of the bleached and unbleached oils, respectively.

E. LEWKOWITSCH.

**Action of silent [electric] discharge on oils, fats, and fatty acids.** V. Catalytic properties of added substances. I. Y. IWAMOTO (J. Soc. Chem. Ind., Japan, 1930, 33, 247—248 B; cf., *ibid.*, 1929, 32, 93 B).—Oleic acid was subjected for 6 hrs. to the action of the silent discharge in an atmosphere of hydrogen and in the presence of 5% of various metallic and metallic-soap catalysts, and the iodine value, stearic acid content, etc. of the products were determined. Platinum and palladium act as powerful positive catalysts for the hydrogenation; uranium, iron, bismuth, and aluminium oxides are weaker in action. Bismuth oxide and ferric chloride appear to accelerate polymerisation rather than hydrogenation. Copper, zinc, and magnesium oxides, titanium chloride, and cobalt hinder both polymerisation and hydrogenation, whilst silver oxide and zinc chloride entirely inhibit any action of the silent discharge.

E. LEWKOWITSCH.

**Colour reactions of [fatty] oils.** W. H. DICKHART (Oil & Fat Ind., 1930, 7, 391).—10 C.c. of the oil, acidified with 1 c.c. of concentrated hydrochloric acid, are shaken for 1—2 min. with 10 c.c. of amyl alcohol, and the liquids allowed to separate. Soya-bean oil (also admixed with 9 vols. of tung oil) produces a deep blue colour persistent for 24 hrs. Linseed and perilla oils give a blue colour fading in 24 hrs. to yellow and pale blue, respectively. Olive, cottonseed, arachis, sunflower, rape (yellow after 24 hrs.), and tung oil solutions are colourless. Crude cottonseed and palm oils give red colours.

E. LEWKOWITSCH.

**Analysis of olein.** H. P. KAUFMANN [with R. WALTHER] (Allg. Oel-Fett-Ztg., 1930, 27, 4—9; Chem. Zentr., 1930, i, 3735).—A discussion. Normal oleins contain more saturated and higher unsaturated fatty acids than was previously supposed.

A. A. ELDRIDGE.

**Transformation of fatty acids.** STADNIKOV and VOSSCHINSKAJA.—See II. **Waxed paper.** THOMAS and REBOULET.—See V. **Soap in paints.** BALDWIN. **Determination of insolubles and wax in shellac.** STILLWELL.—See XIII. **Colloid chemistry of butter.** KING. **Determination of fat in chocolate.** WISEMAN.—See XIX.

## PATENTS.

**Extraction of fatty oil.** M. SATO and (A) C. ITO, (B) Y. ISHIDA (B.P. 336,273—4, 10.6.29).—The material is extracted with (A) alcohol under pressure at a temperature above the b.p. of the solvent and below 120°, or (B) with a hot (above 65°) mixture of alcohol and light petroleum (and/or benzene) in the proportions, e.g., 9:1, and the oil is separated from the extract by cooling to below 50°. A continuous process is described

in (B), by which soya beans yielded 19.5% of a pale oil and a meal rich in nitrogen. E. LEWKOWITSCH.

**Refining of edible oils.** R. B. HILL, Assr. to BROWN Co. (U.S.P. 1,749,976, 11.3.30. Appl., 30.11.23).—Oils are decolorised by treatment with 0.05—0.15% of a 30—75% (by wt.) solution of sulphuric acid. The oils may be further treated with alkali and the soap-stock coagulated with powdered bentonite (ardmorite).

E. LEWKOWITSCH.

**Manufacture of lubricating grease.** E. N. KLEM-GARD (U.S.P. 1,749,251, 4.3.30. Appl., 17.3.27).—Smooth transparent buttery greases are prepared by heating paraffin oil with 5—25% of (commercial) aluminium stearate at 168—289° until the mixture is dehydrated and the stringy texture destroyed, and then cooling without agitation.

E. LEWKOWITSCH.

**Preparation of emulsions.** K. HATTORI (U.S.P. 1,752,176, 25.3.30. Appl., 31.3.26).—Protalbic acid (e.g., 2—10 pts. per 1000) is used as emulsifier for cod-liver oil (with or without, e.g., lactic acid), camphor, etc.

E. LEWKOWITSCH.

**Soap-making and -forming apparatus.** L. H. NELLES (B.P. 337,430, 2.5.29).

Hydrogenation (B.P. 336,569).—See I.

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

**Metallic soaps.** II. **Effect of soaps in paints—preparation of paints.** J. T. BALDWIN (Amer. Paint & Varnish Manufs.' Assoc., Oct., 1930, Circ. 370, 492—506; cf. B., 1930, 67).—The consistency and settling and weathering properties of a series of paints containing lithopone, zinc oxide, basic carbonate and basic sulphate white lead, and titanox, in vehicles containing a comprehensive range of metallic soaps, were observed, and the results are tabulated.

S. S. WOOLF.

**"Chalking" of paints.** H. WOLFF (Farben-Ztg., 1930, 36, 221—223).—The seriousness of chalking as a paint defect depends on the purpose for which the paint is intended. White paints chalk more than tinted paints, the main cause of chalking being the destruction of the binding vehicle due to the influence of actinic rays; thus admixture of stand oil with a boiled-oil paint reduced chalking considerably. The addition of 0.05% of lampblack to a white paint reduced the extent of chalking by 10% in 6 months and by 30% in 1 year, whilst for 0.1% of tinter the corresponding figures were 47% and 60%. Chalking does not always proceed at a uniform rate: cases are instanced of paints that chalked heavily for 9 months, and then showed a period of 6—9 months in which chalking was much less pronounced before the final disintegration of the film set in. This is explained by the stratification of paint films (previously discussed by Blom and by Wolff; cf. B., 1929, 609, 103), chalking pigments forming a pigment-rich outer layer, whilst the surface layers of paints based on non-chalking pigments have relatively low pigment concentration.

S. S. WOOLF.

**Causes of floating of pigments in paints and enamels.** C. A. HOLKESVIG (Amer. Paint & Varnish Manufs.' Assoc., Oct., 1930, Circ. 370, 477—489).—



Colour floating or flooding is a condition of concentration of colour at the surface of a film, due to presence of certain pigments in true or colloidal solution, or to differences in ease or rate of dispersion of pigments. The use of insoluble pigments or pigments of similar dispersion rates in the particular vehicle employed, and the continuation of grinding until differences of dispersion are equalised, will reduce or eliminate floating.

S. S. WOOLF.

**Synthetic resins in enamels and house paints.**

L. K. SCOTT, E. J. PROBECK, and O. MILETI (Amer. Paint and Varnish Manufs.' Assoc., Oct., 1930, Circ. 370, 437—449).—A description of exposure tests, the results of which indicate the superior durability of "glyptal" enamels and house paints when compared with "4-hr. enamels" and normal house paints, respectively.

S. S. WOOLF.

**The "aridyne"—a standard unit of [paint] drying power.** C. F. CARRIER (Amer. Paint & Varnish Manufs.' Assoc., Oct., 1930, Circ. 370, 510—519).—The "aridyne," a standard for use in evaluation of driers, is defined as the drying power of 0.064 oz. of oil-soluble metallic lead dispersed in a volatile solvent. "Soligen" driers are suggested as a suitable basis for the preparation of standard drying solutions.

S. S. WOOLF.

**Accelerated testing of protective coatings.** M. F. MORAM, JUN. (Amer. Paint & Varnish Manufs.' Assoc., Oct., 1930, Circ. 370, 548—564).—Eighteen paints were exposed to normal weathering and to five different accelerated weathering cycles and the results are tabulated and discussed.

S. S. WOOLF.

**Colour control in the paint plant.** M. R. PAUL and J. P. ST. GEORGE (Amer. Paint & Varnish Manufs.' Assoc., Oct., 1930, Circ. 370, 419—436).—An account of an investigation into the preparation and maintenance of colour standards by spectrophotometric methods. The practical limits for commercial colour matching are indicated.

S. S. WOOLF.

**Selective adsorption by pigments.** C. P. VAN HOEK (Farben-Ztg., 1930, 36, 267—269).—Theories explaining the relative durabilities of exterior paints containing white lead, zinc oxide, lithopone, or titanium white in terms of basicity and tendency to soap formation are summarised and shown to be inadequate. Selective adsorption of fatty acids, and retention of the products of oxidation in the film by pigments, are considered to be significant factors leading to non-hardening films. This must be taken in conjunction with the degree of ultra-violet light absorption of the pigment, with proportionate protection of the binding vehicle, and from this aspect zinc oxide is to be regarded as the most durable pigment of the range, being superior to white lead for resisting severe weather conditions. These views are correlated with the "chalking" qualities of lithopone and titanium white, and other theories of chalking are briefly examined.

S. S. WOOLF.

**Micrography of coloured pigments.** I. SIENNA. ANON. (Farben-Ztg., 1930, 36, 120—121).—A summary of the work of H. Wagner and R. Haug on the examination of eleven samples of Italian and indigenous siennas, including optical properties, physical properties, e.g.,

sp. gr., state of subdivision, oil absorption, etc., chemical composition, microscopical examination, and a scheme of specification.

S. S. WOOLF.

**[Nitrocellulose] solvents.** A. HEINEMANN (Farben-Ztg., 1930, 36, 270; cf. Zimmer, B., 1930, 1119).—Attention is drawn to the effect of the molecular complexity of solvents on the size of the nitrocellulose-solvent complexes formed, and hence on the viscosity of nitrocellulose solutions. The significance of the presence of the hydroxyl group in solvents is also indicated.

S. S. WOOLF.

**Lacquer studies. I. Development of an abrasion test for use with nitrocellulose lacquers.** W. KOCH (Ind. Eng. Chem. [Anal.], 1930, 2, 407—409).—A modification of Sward's abrasion test (B., 1929, 825) is described, in which carborundum powder is driven by a regulated air current against the test film, which is inclined at 45° to the blast, the first appearance of actual holes in the film being taken as the end-point. The "abrasion factor" is recorded as (weight of abrasive used)/(film thickness in cm.  $\times$  1000), a standard lacquer being used for comparison. More than 24 hrs.' ageing of the very thin films used (0.0015—0.0025 cm. thick) has no effect on the abrasion factor.

E. LEWKOWITSCH.

**Blooming of varnish films.** W. KRUMBHAAR (Amer. Paint & Varnish Manufs.' Assoc., Oct., 1930, Circ. 370, 520—532).—The following conclusions are drawn from the behaviour of trial varnishes. To be free from bloom, varnishes should contain sufficient calcium to neutralise free acidity and as much manganese as possible (regarding colour). Zinc, lead, and cobalt driers, stand oils, and blown oils favour bloom. Linseed oil (in the proportion of at least 1½ pts. of oil to 1 pt. of fused resin) gives non-blooming varnishes, as does wood oil in special combination with linseed oil and bodied at not above 210°. The blooming tendencies of resins vary greatly, that of Sierra Leone gum being very low, whilst ester gum and other synthetic resins are unsuitable for use in non-blooming varnishes. The nature and method of addition of solvents used are not significant, with the exception that solvents containing fractions boiling above 221° should be avoided.

S. S. WOOLF.

**Resistance to tackiness of certain paint, varnish, and lacquer coatings on the interior of cedar-lined receptacles.** F. RABAK (Ind. Eng. Chem., 1930, 22, 1136).—Tests on a number of finishes stored in a small cedar-lined safe show that vapours emanating from the cedar lining act as a slow solvent for the vehicles of certain coatings, e.g., oil enamels, varnishes, asphaltum, etc., softening the films to the extent of tackiness. Paints free from or containing relatively little drying oil, e.g., flat wall paints, lacquers, radiator bronze, were satisfactory, showing no stickiness over a period of 18 months.

S. S. WOOLF.

**Determination of wax in shellac.** A. G. STILLWELL (Ind. Eng. Chem. [Anal.], 1930, 2, 387).—Powdered shellac (5 g.) is dissolved by boiling in 150 c.c. of water containing 3 g. of sodium carbonate. After cooling to below 15° and filtering through a cotton-wool and asbestos plug in a specially prepared Soxhlet apparatus, the



precipitate is well washed successively with water and with 50 c.c. of 70% alcohol, dried in an oven, and extracted with carbon tetrachloride. The solvent is evaporated and the extract weighed. Results are obtained within 3 hrs. and agree with those from the official method.

E. LEWKOWITSCH.

**Determination of "insolubles" in shellac.** A. G. STILLWELL (Ind. Eng. Chem. [Anal.], 1930, 2, 420—421).—It has been found that the official extraction method fails to remove from the shellac 0.3—0.5% of a wax (m.p. 93°) which passes into varnishes and should not be included as "insoluble." A more rapid method is described in detail in which the powdered lac (5 g.) is dissolved in 50 c.c. of a mixture of alcohol and carbon tetrachloride (2 : 1): the solution is filtered by siphoning through a wad of cotton-wool, and the insoluble portion is washed with hot alcohol, extracted for 1 hr., and dried. The results agree with those obtained by the standard method when extraction is carried to completion.

E. LEWKOWITSCH.

**Determination of the m.p. of resins.** M. RANGASWAMI (J. Oil Col. Chem. Assoc., 1930, 13, 287—288).—Durrans' "mercury method" (cf. B., 1929, 690) is modified by the use of small amounts, e.g., 0.2 g., of resin and by conducting the test in a crucible in place of a test-tube. Within limits, the amount of mercury used does not affect the accuracy of the determination, if heating be sufficiently slow. Typical results are quoted.

S. S. WOOLF.

**New colour reaction for rosin.** H. C. COHEN (Farben-Ztg., 1930, 36, 121).—1 c.c. of a solution of 1 vol. of chlorosulphonic acid in 5 vols. of chloroform is added to 1 c.c. of a 10% chloroform solution of the material under test. Rosin gives a relatively permanent violet-red coloration, albertol and ester gum a rose-pink, drying oils give no colour. One drop of a 2% rosin solution in linseed oil gives a deep red coloration with 1 c.c. of the reagent.

S. S. WOOLF.

**Measurement of liquid viscosity.** FAWKES. **Photoelectric process control.** STYER and VEDDER; McMASTER.—See XI. **Blown and sulphur-treated oils.** HOLLANDER.—See XII.

#### PATENTS.

**Manufacture of synthetic resins.** I. G. FARBENIND. A.-G. (B.P. 314,810, 1.7.29. Ger., 2.7.28).—Solvent naphtha, freed from phenols and bases, is condensed below 90° with an alkyl ether of a *m*-substituted phenol, particularly of a *m*-cresol or *m*-5-xylenol, in presence of a condensing agent (stannic chloride, ferric chloride, fluoboroacetic acid, sulphuric acid) other than aluminium chloride. The resins (m.p. 148°, 138°, 113°, 133°, and 94°, respectively, in the five examples) are miscible in all proportions with petrol and with drying oils.

C. HOLLINS.

**Decoration of [hard] surface-covering material [e.g., linoleum etc.].** ARMSTRONG CORK CO., Assees. of J. C. MCCARTHY (B.P. 337,924, 8.10.29. U.S., 21.11.28).

**Production of thin plates of artificial resin.** C. A. JENSEN (B.P. 337,530, 13.9.29).

**Azo pigments** (B.P. 335,555).—See IV. **Mercerised wood-fibre** (U.S.P. 1,749,003). **Cellulose ether com-**

**positions** (B.P. 312,309). **Solvent for cellulose nitrate** (U.S.P. 1,756,228). **Reducing the viscosity of nitro-cellulose** (U.S.P. 1,757,491 and 1,757,481).—See V. **Naphthenates** (B.P. 335,863). **Crystalline zinc sulphide** (U.S.P. 1,758,741).—See VII. **Coloured glow lamps** (B.P. 315,313).—See XI.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Fractionally coagulated crêpe [rubber].** LONDON COMMITTEE OF THE CEYLON RUBBER RESEARCH SCHEME (Bull. Imp. Inst., 1930, 28, 294—299).—In fractional coagulation of latex in two successive stages, the first fraction is the richer in acetone-soluble constituents, but the plasticity of both fractions is little different from that of the control sample. Both fractions give vulcanised products of good tensile strength, but vulcanisation is much the more rapid and "flatter" with the first fraction, and the product ages better.

D. F. TWISS.

**Swelling of vulcanised rubber in liquids.** J. R. SCOTT (Trans. Inst. Rubber Ind., 1930, 5, 95—118). The time-swelling curve for vulcanised rubber immersed in an organic solvent consists of an early markedly curved portion representing the initial rapid absorption and "saturation" and an indefinitely prolonged slight slope representing a slow continued absorption, termed the "swelling increment." This increment is probably caused by gradual depolymerisation of the rubber arising from the presence of dissolved oxygen in the swelling liquid; it is greatly increased by addition of depolymerising agents, such as acids or copper salts, or of oxidising agents to the liquid, and decreased by antioxidants and exclusion of oxygen. The swelling maximum is determined by the chemical character of the liquid rather than by its physical properties, but the swelling time (disregarding the "increment") seems to be determined by the physical properties, particularly the viscosity. Increase in temperature and exposure to light increase the swelling maximum. The presence of organic accelerators or of sulphur increases the depolymerisation effect as indicated by the swelling increment, but the swelling maximum is diminished by all factors which reduce the extensibility of the rubber, such as progressive vulcanisation or the presence of reinforcing constituents. A swelling test should be of value for the selection of antioxidant substances and for the detection of injurious ingredients in constructional materials intended for use with rubber.

D. F. TWISS.

**Action of zinc oxide containing iron in rubber mixtures.** R. DITMAR and C. H. PREUSZE (Caoutchouc et Gutta-Percha, 1930, 27, 15,219—15,220).—From the point of view of chemical and mechanical results the presence of iron oxide in zinc oxide, up to 2% on the latter, is completely without modifying influence on the effect of the zinc oxide as a compounding ingredient for rubber. The only observable influence is a tendency to impart a slight colour to otherwise white vulcanisates.

D. F. TWISS.

**Influence of increasing content of different fillers on the calender behaviour of unvulcanised rubber mixtures.** R. DITMAR and C. H. PREUSZE (Caoutchouc et Gutta-Percha, 1930, 27, 15,212—15,214



—The results of observations are recorded of the effect of various proportions of chalk, zinc oxide, carbon black, and silica gel on the ease of calendering to smooth sheet, and the degree of contraction of the sheet in the next 5 min. Adsorbent fillers decrease the contractive effect and facilitate smooth calendering. D. F. TWISS.

**Permeability of rubber mixings.** W. C. DAVEY and T. OHYA (Trans. Inst. Rubber Ind., 1929, 5, 27—30).

—Experiments on the rate of diffusion of hydrogen through sheets of rubber attached to cambric for support, using a constant-flow method and applying an interferometer to the refractometric determination of the diffused hydrogen, show that vulcanisation increases the permeability, but that the presence of an organic accelerator and particularly of an antioxidant or softener improves the result. The presence of mineral fillers in the rubber assists the passage of hydrogen, but the effect is less with finer fillers such as zinc oxide and carbon black. D. F. TWISS.

**Effect of different conditions of storage on the hardness of rubber.** LONDON COMMITTEE OF THE CEYLON RUBBER RESEARCH SCHEME (Bull. Imp. Inst., 1930, 28, 289—293).—Samples of rubber, on storage for 6 months at 0° after their arrival in this country, invariably become hard and frozen, storage at 15° also sometimes leading to the same result; the samples stored at 0°, however, generally require less milling to reach a fixed degree of plasticity. The length of time for which the coagulum remains in the serum on the plantation has no effect on the hardness of smoked sheet rubber, but storage in this country leads to general hardening, the effect being particularly marked in a dried atmosphere. No hardening was observed in an atmosphere of nitrogen. D. F. TWISS.

**Heats of combustion of rubber, gutta-percha, and balata.** T. H. MESSENGER (Trans. Inst. Rubber Ind., 1930, 5, 71—86).—After purification by extraction with water and acetone, respectively, and successive dissolution in benzene, precipitation with alcohol, re-dissolution in chloroform, and re-precipitation with acetone, the dried rubber, gutta-percha, and balata gave substantially the same heats of combustion, viz., 11,000 g.-cal. per g. This result confirms the close chemical similarity between these hydrocarbons and gives no indication of any difference in degree of polymerisation. Disaggregation of rubber by milling or exposure to sunlight also left the heat of combustion unaltered, the alteration effected therefore being presumably physical in character rather than one of real depolymerisation. In an appendix, values obtained by various experimenters for the C:H ratio in rubber, gutta-percha, and balata are compared. D. F. TWISS.

**Effect of temperature, pressure, and frequency on the electrical properties of rubber.** H. L. CURTIS, A. T. MACPHERSON, and A. H. SCOTT (Physical Rev., 1929, [ii], 33, 1080).—The dielectric constant, power factor, and resistivity of rubber depend on the percentage of sulphur used in its vulcanisation, on the temperature and pressure, and, for the first two properties, on the frequency at which measurements are made. L. S. THEOBALD.

**Standardisation of vulcanisation testing [of rubber].** J. R. SCOTT (Trans. Inst. Rubber Ind., 1930, 5, 139—150).—The details of procedure for vulcanisation testing, particularly for the evaluation of raw rubber, are discussed, the following factors receiving consideration: composition and preparation of the test mixture, interval between mixing and vulcanisation, conditions of canisation, interval before tensile testing, character of the tensile tests, and degree of vulcanisation to be adopted for comparative purposes. D. F. TWISS.

**Determination of adsorptive power of amorphous carbon [for rubber] with methylene-blue.** R. DITMAR and C. H. PREUSZE (Gummi-Ztg., 1930, 45, 243—244; cf. Carson and Sebrell, B., 1929, 1024).—To 50 c.c. of a 0.01% solution of methylene-blue at 15° are added small portions from a bulk of 0.5 g. of the carbon black with shaking, until the solution is decolorised; the mixture is then titrated with the methylene-blue solution until faint colour persists, the "adsorptive power" of the carbon black being expressed as the number of parts of methylene-blue adsorbed by 100 pts. The adsorptive power varies widely for different grades of black, and has no simple relation with their relative rates of sedimentation from water or with their appearance in ultra-violet light. Highly adsorptive carbon black retards the rate of vulcanisation of a rubber mixture and also tends to reduce the amount of "overflow" during vulcanisation, so that the sp. gr. of the vulcanisate is greater than for a similar mixture containing a less adsorptive black. The relative ease of wetting by rubber, which is a feature of practical importance in carbon blacks, can be estimated by observing the difference in appearance of two small sheets of masticated crêpe rubber which have been sprinkled very thinly with the two samples of black under comparison and kept at 70° for 45 min. D. F. TWISS.

**Comparison of carbon blacks.** DAWSON and HARTSHORNE.—See II. Ageing of cotton contained in rubber goods. BARR.—See V.

#### PATENTS.

**Preservation of rubber.** J. TEPPEMA, Assr. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,781,306, 11.11.30. Appl., 30.11.26).—See B.P. 281,616; B., 1929, 66.

**Method of uniting rubber and other substances.** L. B. SEBRELL, Assr. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,781,649, 11.11.30. Appl., 26.4.28).—See B.P. 310,461; B., 1930, 472.

**Disintegration of rubber (B.P. 336,828). Vessels proof against chemicals etc. (B.P. 316,134).**—See I. Mercaptobenzthiazole (B.P. 335,567).—See III. Sheet-like solids etc. (B.P. 318,250).—See V.

#### XV.—LEATHER; GLUE.

**Measurement of the swelling of hide by expression of the water.** H. OWRUTSKY (Collegium, 1930, 427—430).—When pelt was swollen in a lime liquor and then weighted, the amount of water removable by pressure (as determined at regular intervals) was found



to diminish as the process was continued. Delimed pelts were next treated with water and with 0.1N-solutions of various acids, the thickness of the swollen pelts was measured, the pelts were submitted to pressure, and the weight of water expressed was noted. It was shown that the swelling of the pelt could be measured by the amount of expressed water, but not by the thickness of the swollen pelt, since the pelt swollen in distilled water was thicker than that swollen in 0.1N-acids. The water-swollen pelt lost most water (14.1%) by expression, and pelt swollen in 0.1N-hydrochloric acid lost least (5.36%), so that the greatest degree of swelling was shown in hydrochloric acid. The water in alkali-swollen pelt is in a state of weaker combination with collagen than is the water in acid-swollen pelt, since it is more readily expressed. D. WOODROFFE.

**Determination of the best conditions of tannage with extracts of *Rhus cotinus*, L., and *Carpinus duinensis*, Scop., at different  $p_H$  values.** A. N. ANFIMOV and A. P. PISSARENKO (Collegium, 1930, 419—427).—Examination of *Rhus cotinus*, L., extract liquors ( $d$  1.0615) and *Carpinus duinensis*, Scop., extract liquors ( $d$  1.028) of different  $p_H$  values showed that the higher was the  $p_H$  value, the greater was the amount of precipitated material. At  $p_H$  8, 75% of *Rhus cotinus* was precipitated. When hide powder was treated with the respective liquors, the maximum amount of material was found to be absorbed by the powder from liquors having  $p_H$  2. Pieces of pelt tanned in these liquors exhibited minimum thickness and swelling and gave the best finished leather when liquors having  $p_H$  5—6 were used. D. WOODROFFE.

**Fluorescence test for [identification of] tanning materials.** C. VAN DER HOEVEN (Collegium, 1930, 414—419).—The fluorescence observed in ultra-violet light on wadding soaked in the tannin solution ( $a$ ) without further treatment, ( $b$ ) with the addition of acid or alkali, respectively, is given in tabular form for 62 tanning extracts and materials, synthetic tannins, and sulphite-cellulose extracts. The most characteristic colours were obtained with mangrove bark, ulmo, sulphite-cellulose extract, and certain synthetic tannins. D. WOODROFFE.

#### PATENTS.

[Rotary] apparatus for tanning skins, hides, etc. E. G. WILSON (B.P. 337,408, 22.7.29).

Treatment agents for leather (B.P. 317,039).—See III.

### XVI.—AGRICULTURE.

**Examination of soils.** K. UTESCHER (Z. Pflanz. Düng. 1930, 18A, 203—217).—The value of chemical analyses, notably the acid-extraction processes of the Prussian Geological Institute, in determining the nature and properties of soils is emphasised. A. G. POLLARD.

**Soil colloids. I. Base exchange and soil acidity. II. Factors influencing the dispersion of soil colloids in water.** A. N. PURI (Mem. Dept. Agric. India, 1930, 11, 1—38, 39—51).—I. Experiments with a number of soils have shown that when a soil is exhaustively treated with 0.05N-hydrochloric acid and washed free from excess the amount of hydrogen ion

retained by it is a characteristic constant for the particular soil. A part of the hydrogen-ion content can be replaced by any other cation by exhaustive treatment with a neutral salt; the free acid simultaneously thereby liberated is then equivalent to the amount of cation entering the soil complex. The "acidoid" or soil completely saturated with hydrogen ions has been studied as a complex acid in a number of reactions characteristic of acids. Experiments on sucrose inversion were carried out on 6 soils from various parts of the world, but they all indicated the same dissociation constant, the value being close to that of acetic acid. The main result of these investigations is to show that the acidoid possesses the characteristics of a true acid, and that the reactions take place in three distinct stages, in which 1, 2, and 3 equivalents of hydrogen ion take part. The first hydrogen ion is replaced by exhaustive treatment with a neutral salt and the suspension is then neutral. Only the first hydrogen ion can invert sucrose, hydrolyse ethyl acetate, decompose zinc sulphide, or show "exchange acidity." The second hydrogen ion can be replaced by heating the suspension with calcium or barium carbonate in a current of air. Normal salt formation by replacing the third hydrogen ion takes place in the presence of a large excess of alkali. The methods of determining lime requirements of acid soils are discussed in the light of the tribasic acidoid theory, and large-scale experiments on liming have shown that neither liming nor manuring alone much improves the soils. A combination of the two is necessary, and the optimum dose of lime is different in the presence of different manures. After the maximum value for the crop yield with any manure, there is a distinct tendency for the yield to fall. The toxicity of soils is confined to the first replaceable hydrogen ion, and a simple method for its measurement is outlined.

II. The dispersion of soils has been studied as nearly as possible under field conditions by leaving the soil in contact with water for 24 hrs. and determining the dispersed clay (0.002 mm.) by the pipette method. This value is termed the "dispersion factor," and the dispersion coefficient is defined as the percentage of the total clay which can pass into suspension on being left in contact with water for 24 hrs. It is proposed to give the name "clayic acid" to the soil completely saturated with hydrogen ions and to call the salts derived therefrom "clayates." The dispersion coefficients of clayic acid and a number of clayates have been determined; the lithium and sodium salts give considerably higher values than any other. All good soils give a comparatively low dispersion coefficient and all barren alkali soils give a high value. Soils rich in exchangeable sodium give a high dispersion coefficient and a very low rate of percolation to water and whilst treatment with gypsum increases the permeability, it lowers the dispersion coefficient. Highly acid soils give a very low dispersion coefficient and a very high rate of percolation. Irrigated soils give a comparatively higher dispersion coefficient than unirrigated soils. E. S. HEDGES.

**Microflora of leached alkali soil.** J. E. GREAVES and J. D. GREAVES (Bot. Gaz., 1930, 90, 224—230).—The restoration of bacterial activities in alkali soils following leaching varied with the concentration and



nature of the salts present, and is probably due to the removal of inhibitory substances by the saline solutions and a subsequent stimulation of the organisms by the remaining salts. In this respect leaching is less effective in the presence of chlorides than of sulphates. In Ashby media inoculated with soils sulphates stimulated nitrogen fixation to a greater extent than any other salts examined. A. G. POLLARD.

**Origin, nature, and isolation of the inorganic base-exchange compound of soils.** E. TRUOG and J. A. CHUCKA (J. Amer. Soc. Agron., 1930, 22, 553—557).—Minute quantities of base-exchange material in soil can be detected by treatment with aqueous magenta (0.5%) and washing with water and alcohol, any such material being coloured. The material, which appears to be formed from feldspars under alkaline conditions of weathering, was separated from bentonite and a silt loam sub-soil. CHEMICAL ABSTRACTS.

**Effect of silicic acid [in soil].** W. KRÜGER and G. WIMMER [with O. RINGLEBEN, O. UNVERDORFEN, J. GRIMM, and H. LÜDECKE] (Z. Ver. deut. Zucker-Ind., 1930, 80, 771—812).—Literature concerning the inter-relationships between soil phosphates and silica in plant nutrition is discussed. The favourable effect of silica on the growth of plants is increased by the presence of ferrous sulphate. The latter in the absence of silica decreases crop yields by reducing the assimilability of phosphates. The bearing of this on the work of Lemmermann is discussed. A. G. POLLARD.

**Determination of the total surface area of soils, clays, and similar substances.** H. KURON (Z. Pflanz. Düng., 1930, 18A, 179—203).—The adsorption of water by soils, their mechanical fractions, sand, and permutit is examined. At lower vapour pressures adsorption follows the Ostwald-Freundlich rule in all cases, and calculations of total surface area of the materials may be made directly. The point of inflexion of drying-out curves of soil and its constituent fractions occurs at the same vapour pressure. The water adsorption of a soil at various vapour pressures can be represented as the sum of the adsorptive values of its constituent fractions. Drying-out curves for permutit are of a similar nature to those of natural zeolites. A. G. POLLARD.

**Simplified method of determining "sticky point" of soils.** A. N. PURI (Agric. J. India, 1930, 25, 206—209).—The method of Keen and Coutts (B., 1928, 905) has been modified to adapt it for use in both the field and laboratory, and comparative results for 48 Indian soils are detailed. E. HOLMES.

**Rapid determination of the nitrogen requirement of soils.** J. PÁZLER (Z. Zuckerind. Czechoslov., 1930, 55, 87—90).—The sample (100 g. of soil passing 2-mm. sieve) is shaken for 1 hr. with 1% potassium sulphate solution in a nearly filled 500-c.c. Stohmann flask. After filling to the mark, the suspension is filtered and 300 c.c. of the filtrate are used for nitrate determination by means of Devarda's alloy. Nitrate contents so determined agree closely with those by Mitscherlich's method for nitrogen-deficient soils. For richer soils

the Mitscherlich values are definitely higher. The presence of calcium carbonate in soils tends to increase the nitrate values recorded by the method described.

A. G. POLLARD.  
**Determination of phosphate and potash requirements of soils by the citric acid method and Sigmond's nitric acid method.** W. JESSEN and W. LESCH (Z. Pflanz. Düng., 1930, 18A, 218—235).—None of the methods of Neubauer, of Lemmermann, or of Sigmond gives a sharp line of demarcation between normal and phosphate-deficient soils. Sigmond's method is simpler and more definite for soils rich in carbonate, but has no advantage for soils of low carbonate content. No relationship exists between the mechanical composition of soils and the solubility of their phosphate contents. All purely chemical methods give similar values for the potash requirement of soils, but in heavy soils these differ from the results of pot experiments and the seedling method. In clay soils rich in hydrochloric acid-soluble potash the assimilation of potash by plants is greater than the total exchangeable potash present, and in these soils chemical methods fail to give definite information as to assimilable potash contents. A. G. POLLARD.

**Determination of the nutrient content and nutrient changes in soils by the citric acid method and other processes.** J. HASENBÄUMER and R. BALKS (Z. Pflanz. Düng., 1930, 9B, 456—473).—The nutrients removed from soil in four successive crops were much greater than the decrease in citric-soluble material in the soil. The loss of citric-soluble nutrients under these conditions approximated to the loss of water-soluble nutrients, and this general agreement was maintained in differently fertilised soils. The loss of citric-soluble phosphate and potash during cropping tended to become less than the loss of root-soluble material (Neubauer) as the clay content of the soils increased. The citric-soluble potash content of soil samples varied considerably with the distance from plant roots at which they were taken. Corresponding variations in the citric-soluble phosphate contents were small. In general, variations in the citric-soluble, water-soluble (Dirks and Scheffer), and root-soluble phosphates in soils were similar, but the values were not strictly proportional. Niklas' method (absorption of potash and phosphate by *Aspergillus*) is less sensitive to differences in soil treatment, but yielded values in general agreement with the above. A. G. POLLARD.

**New microbiological method for determining the fertiliser requirement of soils.** H. NIKLAS, H. POSCHENRIEDER, and J. TRISCHLER (Z. Pflanz. Düng., 1930, 18A, 129—157).—Factors influencing the growth of *Aspergillus niger* in soil cultures are examined and a method is developed for determining the phosphate and potash requirement of soils by means of the weight of the mycelium produced. Addition of 1% of citric acid to the nutrient solution minimises the growth of other soil organisms and maintains a fairly constant  $p_H$  during the test. Chalk present in the soil is initially neutralised by excess of citric acid. Results obtained are closely in agreement with those of Neubauer tests. A. G. POLLARD.



**Influence of different methods of making stall manure on the losses during stacking, and the utilisation of the manure by plants (with special reference to the Krantz hot-fermentation process).** R. SAILER (Landw. Versuchs-Stat., 1930, 111, 63—153).—Comparison is made of open-yard manure and hot- and cold-fermented pit manure. Losses of dry matter and nitrogen during rotting were greatest in yard manure both in summer and winter. Hot- and cold-fermented pit manures suffered similar losses in winter, but in summer the nitrogen loss during hot fermentation was the greater. In all cases summer losses were greater than the corresponding winter losses. Losses in hot-fermenting stacks occurred mainly in the early (loosely stacked) stage. Weed seeds lost their germinative power during hot fermentation, but remained viable in cold stacks. The quantity of drainage liquor was greatest in hot-fermenting stacks and less in yard manure, although the chemical composition of the liquor was practically the same in all cases. In field trials the efficiency of the manures was in the order yard manure < cold-fermented < hot-fermented. Hot-fermented manure showed no superiority in increasing the efficiency of artificial fertilisers. In sterile soils there was little or no utilisation of hot-fermented manure.

A. G. POLLARD.

**Effect of barnyard manure on a calcareous soil.** D. W. PITTMAN (J. Amer. Soc. Agron., 1930, 22, 549—552).—The amount of available phosphorus is largely influenced by the amount of manure applied. Low yield of sugar beet on unmanured soil is due to lack of soluble phosphorus in the early growing season as well as to lack of nitric nitrogen. Farm manure is therefore essential to sugar beet on highly calcareous soil.

CHEMICAL ABSTRACTS.

**Intake of nutrients by plants.** E. HASELHOF, F. HAUN, and W. ELBERT (Landw. Versuchs-Stat., 1930, 111, 11—62).—The yields and mineral contents of a number of crops in various stages of growth in differently fertilised soils are recorded and discussed.

A. G. POLLARD.

**Dependence of the intake of nutrients [by plants] on the reaction of the substrate.** P. DAHM (Ber. deut. Bot. Ges., 1930, 48, 312—316).—No definite relationships were observed between the reaction of the substrate and the assimilation of nutrients in water or in pot cultures. The difficulties of maintaining constant  $p_H$  in nutrient solutions and of determining small quantities of the constituents are greater than are usually supposed. Under the conditions of the experiment lime was more important as a nutrient than as a neutralising agent.

A. G. POLLARD.

**Action of various nitrogenous fertilisers on acid soils.** D. MEYER (Z. Pflanz. Düng., 1930, 9B, 474).—The injurious effects of cyanamide on acid soils previously referred to (cf. Meyer and Obst, B., 1930, 208) are limited mainly to acid soils poor in humus and of low biological activity.

A. G. POLLARD.

**Interpretation of fertilising experiments with cyanamide.** E. J. PRANKE (Amer. Fertiliser, 1927, 67, No. 11, 60—66).—When calcium cyanamide is exposed to the air, 8% may be converted into dicyano-

diamide in 1 year; this form of nitrogen is, however, effective as a fertiliser if it does not exceed 10% of the total quantity present. The reaction of calcium cyanamide with superphosphate and with soil acids is also discussed.

CHEMICAL ABSTRACTS.

**Potash shale as a source of potash for growing plants.** H. C. HEATH (Bot. Gaz., 1930, 90, 121—150).—Applications of potash shale to soils in pot cultures increased the potash intake of a number of crops. Roasting the shale considerably increased its water-soluble potash content and the response of plants to its application. Roasting with calcium carbonate or gypsum was still more effective. Plants having nearly neutral sap benefited more from shale applications than those with more acid sap. Plant injury from excessive applications of lime to a peat and a sandy soil could not be ascribed to depressed assimilability of potash, but was due to reduced photosynthesis resulting possibly from the immobility of iron in the overlimed soil.

A. G. POLLARD.

**Grassland manuring.** G. T. GARRATT (J. Min. Agric., 1930, 37, 778—781).—Notes on the difficulties of rotational grazing of intensively manured pasture on Cambridgeshire Boulder Clay in a season of abnormally low rainfall are given.

E. HOLMES.

**Nutrition of seedlings and its influence on the rooting of cereals.** KLEEMANN (Z. Pflanz. Düng., 1930, 9B, 433—456).—Growth of cereals is largely influenced by the activity of enzymes in the seed prior to actual germination. These activities are stimulated and root development is subsequently improved by moistening the seeds (to 35—40% moisture content) and storing for 3 days in a saturated atmosphere at 17—18°. Still better results are obtained if respiration losses are minimised by storage of the seeds in a vacuum. Convenient alternative conditions of storage and of drying are recorded. Respiration losses during drying are reduced by working at low temperatures or in a vacuum. The improved primary root production and increased growth of treated seed result from the production of a supply of soluble food reserve, notably carbohydrate, by enzymic action.

A. G. POLLARD.

**Nitrate fertilisers for oats in Iowa.** R. A. PENDLETON (J. Amer. Soc. Agron., 1930, 32, 663—668).—Early additions of sodium nitrate produced a larger increase in yield than late applications, but the latter accentuated the increase of protein in the grain. Phosphate did not increase the protein content. Superphosphate without nitrate had little effect.

CHEMICAL ABSTRACTS.

**Fertiliser trials with magnesium salts for oats to determine the mode of action of magnesium.** W. DIX and S. BISCHOF (Z. Pflanz. Düng., 1930, 18A, 158—179).—Magnesium sulphate increased, and the oxide and carbonate decreased, the acidity of soil. In field trials, magnesium additions decreased the crop yields in the order sulphate > carbonate = oxide > lime. Analyses of crops from treated soils are recorded and the intake of calcium and magnesium and their translocation in various organs of the plant discussed in the light of their rôle in plant nutrition.

A. G. POLLARD.



**Seed treatment for controlling covered smut of barley.** R. W. LEUKEL (U.S. Dept. Agric., Tech. Bull. 207, 1930).—Various types of fungicides are compared. Dusting powders were effective where seeds were sown in soils of average moisture content (less than 25% saturation decreased the efficiency of the materials), and were not influenced by soil reaction or by temperature variations within normal ranges. Seed injury by dusts was less than that by liquid treatments.

A. G. POLLARD.

**Effect of superphosphates on the germination of maize.** C. O. ROST (J. Amer. Soc. Agron., 1930, 22, 498—507).—Large amounts of 46% superphosphate appeared to be more toxic than equivalent amounts of 16% superphosphate. Heavy applications retard germination less when they have been in intimate contact with the soil for several weeks before planting.

CHEMICAL ABSTRACTS.

**Concentration of certain constituents of the soil solution under orchard conditions.** E. L. PROBSTING (Hilgardia, 1930, 5, 35—59).—Seasonal variations in the sulphate, nitrate, calcium, and magnesium contents of the soil solution were observed, the actual values being influenced by soil treatment and intercropping and by the nature of the orchard trees. The concentration of potassium remained practically the same throughout the season. An excess of total cations over total anions existed in the soil solution. This was greater under peaches than under pears, and varied with the soil treatment.

A. G. POLLARD.

**Citrus rust mite and its control.** W. W. YOTHERS and A. C. MASON (U.S. Dept. Agric., Tech. Bull. 176, 1930).—Sulphur, which was the most effective agent examined, kills the rust mite by fumes arising from its oxidation. Actual contact with the insect is unnecessary.

A. G. POLLARD.

**Growth of certain conifers as influenced by different fertiliser treatments.** A. C. MCINTYRE and J. W. WHITE (J. Amer. Soc. Agron., 1930, 22, 558—567).—The favourable effect of fertilisation is described.

CHEMICAL ABSTRACTS.

**Indications that available nitrogen may be a limiting factor in hard winter wheat production.** P. L. GAINES and M. C. SEWELL (J. Amer. Soc. Agron., 1930, 22, 639—641).

**Phosphate rock grinding.** GABELER. **Superphosphate.** SHOJI and others. **Rhenania phosphate.** SCHARER. **Polysulphide sprays.** TUCKER. **Nitrogen-fixation plant.** WESTBY.—See VII. **Sunflower seed.** ANON.—See XII.

PATENT.

**Ammonium sulphate** (U.S.P. 1,758,449).—See VII.

## XVII.—SUGARS; STARCHES; GUMS.

**Conductometric analysis in sugar factories.** K. ŠANDERA (Z. Zuckerind. Czechoslov., 1930, 55, 90—93).—A special conductometer is described and its use in the analysis of molasses etc. explained.

A. G. POLLARD.

**Carbonised molasses waste.** KILP.—See II. **Firing of boiler furnaces with bagasse fuel.** FREISE.

—See VIII. **Starch fermentation.** SYPNIEWSKI and others.—See XVIII. **Detection and determination of p-hydroxybenzoic acid.** WEISS.—See XIX.

PATENTS.

**Promotion of fermentation** (B.P. 335,972 and 336,207). **Lactic and acetic acids** (B.P. 335,596).—See XVIII.

## XVIII.—FERMENTATION INDUSTRIES.

**Fermentation phenomena of the *Nymphæa cocci* and their behaviour with regard to yeast.** J. GRÜSS (Woch. Brau., 1930, 47, 473—475).—The production of carbon dioxide by *Coccus zymophyllosepticus* from dextrose is greatly, and the decomposition of cellulose by a mixture of *C. phyllosepticus* and *C. zymophyllosepticus* is slightly, increased by the presence of calcium carbonate. The former action, in absence of calcium carbonate, is increased by aeration. In mixed culture yeast suppresses the activity of *C. zymophyllosepticus*, which does not increase the production of carbon dioxide by the yeast. In fermentations of dextrose in presence of sulphur the production of carbon dioxide by the coccus exceeds the production by yeast, which is much below that in normal fermentations; under these conditions the coccus produces more hydrogen sulphide than does yeast. The cocci become weaker in their power of hydrolysing cellulose when cultivated on artificial media. The production of carbon dioxide from dextrose varies inversely with that of hydrogen and methane.

F. E. DAY.

**Malt dust as a harmful factor in fermentation.** K. SCHUSTER (Woch. Brau., 1930, 47, 481—483).—Comparative laboratory fermentations of normal worts and of worts containing a proportion of extract from malt dust indicate that the latter are unsatisfactory, both as regards the course of fermentation and the quality of the yeast produced. The beers in bad cases are not quite bright and not easily cleared by filtration. The importance of "polishing" malt is stressed, and it is suggested that too fine grinding may give rise to similar effects by increased dissolution of husk constituents.

F. E. DAY.

**Continuous process of distillery-starch malt fermentation.** W. SYPNIEWSKI, A. JOSZT, and M. KUNIŃSKI (Przemysł Chem., 1930, 14, 392—405).—A laboratory apparatus is described for the continuous fermentation of rye- and potato-starch malts. A constant supply of sterile malt flows into the apparatus, which at the same time delivers fermented malt; yeast is added only at the beginning of the process. Yields of 52—54 litres of alcohol from 100 kg. of starch are readily obtained.

R. TRUSZKOWSKI.

**Simple universal thermostat for fermentation laboratories with arrangement for fermentation experiments, refractometry, and pycnometry.** H. FINK (Woch. Brau., 1930, 47, 461—464).—A rectangular water-bath of 100 litres capacity is heated by a flat, electrical heating element. This is controlled through a relay by an Ostwald-Luther or other thermoregulator, and the temperature, equalised by a



motor-driven stirrer, can be maintained constant to  $0.2^{\circ}$ . The same motor actuates a rack for holding fermentation vessels, the speed and motion of which can be varied. A stand attached to the length of the bath carries a number of gas burettes connected by flexible capillaries to the fermentation vessels. The shaking rack is readily removable to allow special stands for the immersion refractometer or a number of pyknometers to be fitted into the bath. F. E. DAY.

**Influence of the distilling apparatus and of other factors on the quality of spirit.** T. CHRZASZCZ and J. RESZETNIAK (Przemysł Chem., 1930, 14, 415—429, 437—448).—The quality and potability of potato spirit obtained in 132 distilleries have been studied. A satisfactory product is obtainable only when sound potatoes are used and infection of the malt is avoided. The spirit obtained from abnormal fermentation, characterised by production of hydrogen sulphide, foam, etc., is invariably of poor quality. The greater the concentration of alcohol obtained from a given rectifier, the purer is the product. Only continuous-process apparatus should be used; periodic and two-column stills give inferior products. The material used for the construction of stills is of minor importance. The potability of spirits depends chiefly on the absence of furfuraldehyde and other aldehydes and acids. The decolorisation of permanganate by rectified spirits is due chiefly to furfuraldehyde. R. TRUSZKOWSKI.

**Determination of carbon dioxide in beer.** J. BLOM and B. KRAUSE (Woch. Brau., 1930, 47, 471—473).—To well-cooled beer 50% caustic soda solution is introduced, if possible by a special piercing pipette and compressed air. The weight of the beer is determined before and after the introduction of the soda solution. Then 20 g. of the alkaline beer are weighed into a 50-c.c. wide-mouthed flask provided with an inlet tube through which acid and air free from carbon dioxide can be drawn to the bottom of the flask. An outlet tube, provided with a small cotton-wool filter, connects with two 200-c.c. flasks in series, containing, respectively, 50 c.c. and 10 c.c. of 0.1N-baryta. These have splash-traps, and are shaken continuously during the absorption. 20 C.c. of 25% sulphuric acid are introduced into the 50-c.c. flask and air is drawn through the apparatus, slowly at first. After about 10 min. the air current is increased, and 15 min. later the apparatus is disconnected and the excess of baryta titrated with 0.1N-hydrochloric acid. Alternatively, the carbon dioxide may be absorbed in caustic potash solution and weighed.

F. E. DAY.

**Sulphurous and lactic acids in the production of fruit wines.** RÜDIGER and K. SICHERT (Wein u. Rebe, 1930, 11, 560—568; Chem. Zentr., 1930, i, 3495).—The addition of lactic acid (2—6 g. per litre) to perry prepared from pears low in acid is recommended in place of that of sulphurous acid. A. A. ELDRIDGE.

**Colorimetric method for the determination of the amount of potassium ferrocyanide required for the clarification [of wine].** A. HANAK (Z. Unters. Lebensm., 1930, 59, 506—511).—A method is described for the rapid determination of iron in wine. Into each

of two similar tubes (*A* the test sample, *B* the standard) are measured 25 c.c. of wine, and to one sample, *A*, are added a few drops of ferric chloride solution (0.05%). A measured volume of potassium ferrocyanide solution (0.5%) is now added to each tube, followed by 1—2 drops of hydrogen peroxide (30%) or dilute nitric acid. The average amount of ferrocyanide solution required is 0.6 c.c., but if, after the addition of peroxide, the colour in *B* be not more intense than that in *A*, insufficient has been added, and the process must be commenced afresh, using 0.8 c.c. Some wines may require 1 c.c. or more. The sample *B* is now diluted with water or a dilute solution of yellow dye to colour equality with *A*. The amount of ferrocyanide required to clarify *A* may now be derived from the amount of solution used, and the ratio of the resultant volumes when colour equality is reached. The addition of gum arabic to the wine helps to prevent flocculation of the Prussian-blue. H. J. DOWDEN.

**Use of the quartz lamp in wine analysis.** A. HEIDUSCHKA and E. MÖHLAU (Pharm. Zentr., 1930, 71, 689—691).—Provided the wine is decolorised by animal charcoal, the adulteration of normal wine by raisin or currant wine is detected by the blue to violet luminescence which the latter shows under the quartz lamp, and which is not given by natural wine. The glycerin-water mixtures which were formerly used as standards for the determination of the intensity of luminescence have been replaced by ethyl-alcoholic solutions of sodium saccharin. The latter standards can be prepared very accurately by weighing, are not hygroscopic, and show no change in luminescence after 4 months.

C. RANKEN.

**Amylase and catalase reactions of milk.** WEINSTEIN. **Diastatic activity of honey.** LAMPITT and others.—See XIX.

## PATENTS.

**Promotion of fermentation.** DARCO SALES CORP. (B.P. 335,972 and 336,207, 3.7.29. U.S., [A] 294,29, [B] 24,29).—(A) An improved yield of fermentation products is obtained by fermenting a molasses solution of medium sp. gr., which is treated as in (B), and raising later the sp. gr. of the partly fermented solution above that of the initial solution by the addition of a molasses solution of high sp. gr. (B) The time of fermentation is shortened and a purer and greater yield of alcohol is obtained by the addition to the fermenting solution of an organic nitrogenous substance and an actively adsorbent material such as vegetable char. A molasses solution of greater sp. gr. than is ordinarily possible can thus be fermented.

C. RANKEN.

**Production of lactic and acetic acids [by fermentation].** W. W. TRIGGS. From WISCONSIN ALUMNI RES. FOUNDATION (B.P. 335,596, 20.6.29).—Pentoses and/or hexoses, e.g., hydrolysed wood, corn-cobs, straw, molasses, etc., are fermented by means of a new ferment (morphology etc. given) isolated by known methods from fermenting silage or sauerkraut. The enzyme grows best at  $28-30^{\circ}$  and at  $pH$  6.5, and is anaerobic. It converts pentoses into acetic and lactic acids, hexoses into lactic acid, without production of gas or ethyl alcohol. C. HOLLINS.



**[Microbiological] preparation of 2:3[ $\beta$ ]-butylene glycol from carbohydrates.** T. H. VERHAVE, SEN. (B.P. 335,280, 26.6.29. Addn. to B.P. 315,263; B., 1930, 1088).—After a first fermentation as described in the prior patent, the alcohol is removed by distillation, and the residue added to a second batch or used as solvent, in place of water, for the next carbohydrate mash. Preferably air is blown through the fermenting mash and alcohol recovered from the exit gases. The presence of  $\beta$ -butylene glycol does not retard its formation in the succeeding batches, and relatively high glycol concentrations are reached. C. HOLLINS.

## XIX.—FOODS.

**Qualities of combined wheats as affected by type of bin, moisture, and temperature conditions.** I. F. C. FENTON and C. O. SWANSON (Cereal Chem., 1930, 7, 428—448).—Details are given of the variation in moisture content of wheat during harvesting, also the effect of the amount of moisture on the keeping quality of the wheat, 15% of moisture being considered the maximum safe amount. Wheats of higher moisture content are improved in keeping quality by moving by means of compressed air. E. B. HUGHES.

**Variations in properties of acetone extracts of common and durum wheat flours.** A. W. WALDE and C. E. MANGELS (Cereal Chem., 1930, 7, 480—486).—Comparison of acetone extracts of Marquis and durum flours showed the former to contain a sterol, probably an ester of sitosterol, which was not present in the durum flour. Acetone extract of flour contains little phosphate, but a relatively large amount of unsaponifiable matter. E. B. HUGHES.

**Proteolysis in bread doughs.** W. E. BROWNLEE and C. H. BAILEY (Cereal Chem., 1930, 7, 487—517).—In sampling the doughs, quick freezing is followed by grinding and acetone dehydration. Several methods of detecting change in the composition of the proteins have been used, including peptisation by salt solutions, precipitation of the primary proteins by heavy-metal salts, measurement of viscosity of the water extract, and fractional analysis of a water extract of the protein. No alteration in the structure of the gluten was detected during the process of fermentation. E. B. HUGHES.

**Test for ropiness in bread.** H. H. BUNZELL and M. FORBES (Cereal Chem., 1930, 7, 465—472).—The method depends on the measurement of the catalase activity in the bread. A totally enclosed apparatus is described in which a solution of hydrogen peroxide is added to a water suspension of the bread, and the evolved oxygen measured. In applying the method to flour, a biscuit is made from flour, water, and baking-powder and the product is baked.

E. B. HUGHES.

**Gluten and non-gluten proteins.** M. J. BLISH (Cereal Chem., 1930, 7, 421—427).—Methods used for the characterisation of the flour proteins involve processes which probably alter the structure. Gliadin is the only protein of which the chemical individuality has been definitely established. The ratio of gliadin to total

protein is fairly constant, but the ratio of gluten to non-gluten proteins varies with different flours.

E. B. HUGHES.

**Normal and modified foaming properties of whey-protein and egg-albumin solutions.** P. N. PETER and R. W. BELL (Ind. Eng. Chem., 1930, 22, 1124—1128).—Whey-proteins have good foaming and emulsifying properties. The best conditions for the utilisation of these properties are given, and as whey-protein is cheaper and has an excellent food value its use in place of egg products is suggested. It can replace egg-white or -yolk in those uses which are dependent on whipping or emulsifying properties, such as in salad dressings, cake icing, ice cream, etc., but it cannot replace eggs in uses which are dependent on heat-coagulative properties such as baked or cooked products.

B. W. TOWN.

**Wheat-protein test digestion studies.** C. F. DAVIS (Cereal Chem., 1930, 7, 518—525).—When using a "high heat" electric heater, capable of evaporating 210 c.c. of water from 300 c.c. in 20 min., the Kjeldahl method is quicker than the Gunning modification.

E. B. HUGHES.

**Gasometric determination of carbon dioxide in baking powder.** J. R. CHITTICK, F. L. DUNLAP, and G. D. RICHARDS (Cereal Chem., 1930, 7, 473—480; cf. Hertwig and Hicks, B. 1929, 187).—The A.O.A.C. gasometric method (cf. J. Assoc. Off. Agric. Chem., 1927, 10, 36) gives concordant and accurate results. Although no correction is applied for the vapour tension of the reaction mixture, this is balanced (at ordinary temperatures) by the carbon dioxide dissolved in the liquid.

E. B. HUGHES.

**Biuret reaction in cereal chemistry.** H. KÜHL (Z. ges. Getreidewesen, 1930, 17, 28—32, 67—70; Chem. Zentr., 1930, i, 3496).—The extract (50:1000, 94 c.c.) is treated with 0.1*N*-sodium hydroxide (6 c.c.), heated at 50°, and then treated with 10 drops of 2% copper sulphate solution, stirring meanwhile. The colour is observed before and after the solution is boiled. The test can be used to determine the age of flour. The reaction is not appreciably affected by maltose or diastase.

A. A. ELDRIDGE.

**Influence of the fat content on the keeping quality of milk.** H. BARKWORTH (J. Min. Agric., 1930, 37, 803—806).—An examination of the results of tests of 4000 samples of milk, extending over 6 years, indicates that if rich milks are considered in terms of butter fat content there is no commercial difference in keeping quality between poor and rich milks. E. HOLMES.

**Catalase test [of milk].** G. ROEDER (Milch. Forsch., 1930, 9, 516—590; Chem. Zentr., 1930, i, 3371).—In Lobeck's test shaking causes no variation in the apparent catalase value, but at high hydrogen-ion concentration the action of the enzyme is considerably retarded. The catalase is more active when present in high concentration and less active when that of the hydrogen peroxide is high.

A. A. ELDRIDGE.

**Catalase reaction of milk.** P. WEINSTEIN (Z. Unters. Lebensm., 1930, 59, 514—515).—Milk which had



been treated by the holding process was accidentally contaminated with raw milk and a considerable rise in the catalase value was caused thereby (cf. B., 1929, 374).  
H. J. DOWDEN.

**Amylase detection in milk.** P. WEINSTEIN (Z. Unters. Lebensm., 1930, 59, 513–514).—The earlier work on the determination of the degree of heating to which milk has been subjected (B., 1929, 374) has been extended to cover the flash treatment at 85°. It is imperative to use pure unswollen starch grains, preferably potato starch. The test works satisfactorily with sour milk and the temperature limits are 35–40°.

H. J. DOWDEN.

**[Detection of] artificial whole-milk powder.** P. WEINSTEIN (Z. Unters. Lebensm., 1930, 59, 515–516).—The powder was observed to contain a relatively high proportion of large fat globules, and the characteristics of the isolated fat suggested adulteration. Extraction with light petroleum gave a yield of 18% of fat (refractometer reading at 25° 62·8°, at 40° 53·0°), whereas normal whole-milk powder gave only 1% (reading at 40° 43·3°). Examination of the fat proved that the powder had been prepared from skim-milk powder with the addition of 17–18% of olive oil.

H. J. DOWDEN.

**Watering of buttermilk.** G. KEMPINSKI (Milch. Forsch., 1930, 9, 506–510; Chem. Zentr., 1930, i, 3371).—Owing to the progressive decomposition of lactose with increase in acidity the density of the serum diminishes and cannot therefore be employed to determine the degree of watering; the ash content is, however, constant.

A. A. ELDRIDGE.

**Influence of pasteurisation on the conservation of butter.** L. PIK (Chem. Listy, 1930, 24, 396–398).—Pasteurisation of buttermilk at 85–95° leads to coagulation of lactalbumin, as a result of which the rate of development of bacteria in the pasteurised product is retarded.

R. TRUSZKOWSKI.

**Colloid chemistry of butter.** "Histology of technical substances." N. KING (Kolloid-Z., 1930, 52, 319–332).—A considerable amount of previously published literature on butter is reviewed from the colloid-chemical point of view, particularly in regard to the relation between structure and physical properties, and chemical and bacteriological changes. Butter is discussed as an emulsion of the fat-in-water type and also as the water-in-fat type, and a new view of its structure is advanced, according to which butter is a disperse system of water droplets and fat globules in liquid fat, the fat thus appearing in two forms. The fatty globules are complex in structure, being composed of an adsorbed layer of protein and an inner layer of oriented crystallites of the fat. The structure, composition, and reactions of the aqueous phase are discussed and also the distribution of bacteria. An account is given of the rôles played by the water-fat and air-fat boundaries in the chemical and bacteriological changes and of their effects on the flavour and odour of the butter.

E. S. HEDGES.

**Masking of the colour reaction for margarine.** B. HEPNER and S. ZALC (Przemysl Chem., 1930, 14, 412–415).—Baudouin's furfuraldehyde reaction is not given

by sesamé oil previously treated by acids or subjected to temperatures above 180°; this applies also to margarine heated above 180° after addition of crude sesamé oil. The development of rancidity in sesamé oil leads to the production of a green coloration with hydrochloric acid alone; this may mask the red coloration due to Baudouin's reaction. When phenolic dyes, such as Sudan Yellow, have been added to the margarine, these should be removed by extraction with potassium hydroxide solution before applying the test. In rancid margarine the Baudouin reaction may fail to give positive results even though the product originally answered to the test.  
R. TRUSZKOWSKI.

**Oil absorption of shell eggs.** T. L. SWENSON and H. H. MOTTEN (Science, 1930, 72, 98).—Eggs dipped in oil at the ordinary and under reduced pressure show a diminution in the loss of weight during storage. Penetration is marked around the air cells.

L. S. THEOBALD.

**Composition of meal from pressed and dried potatoes.** B. LAMPE (Z. Spiritusind., 1930, 53, 298).—Pulped potatoes from which the juice has been expressed are dried below 65° till the moisture content of 60% is reduced to 20%. By drying for approx. 6 hrs. at not above 100°, a final moisture content of 10–11% is obtained. The dried potatoes rapidly crumble in water, show an intact starch grain, and contain, if the potatoes are new, 1·49 and 3·65% of ash and protein, respectively, calc. on dry matter. For old potatoes the corresponding values are 2·04 and 4·38, and approximate to 40–50% of those obtained from flakes made from unpressed potatoes.

C. RANKEN.

**Composition of European and Californian almonds.** C. V. HART (Ind. Eng. Chem., 1930, 22, 1128–1129).—Analyses are recorded. No significant difference in composition was detectable. B. W. TOWN.

**Further comparison of Californian and imported almonds.** G. PITMAN (Ind. Eng. Chem., 1930, 22, 1129–1131).—Californian almonds show a somewhat greater shearing stress than imported varieties, which could not be accounted for by differences in hemi-cellulose content. Almonds from different districts of California had practically the same composition.

B. W. TOWN.

**Do fu: an oriental food.** J. S. HEPBURN and K. S. SOHN (Amer. J. Pharm., 1930, 102, 570).—This preparation of the soya bean contains total solids 13·4, moisture 86·6, ether extract 4·65, crude protein 4·2, ash 0·2, and nitrogen-free extractives 3·91%.

H. E. F. NOTTON.

**Nutritive values of some typical Indian hays.** F. J. WARTH (Mem. Dept. Agric. India, 1930, 11, 73–84).—The composition and digestibility of 30 typical Indian hays are recorded. The protein content varied between 18 and 2%, and these figures were found to be correlated with the percentages of soluble ash, and to decrease in similar samples at increased maturity. The amount of digestible protein in the samples decreased more rapidly than the total amount of protein; samples with less than 2·5% of total protein contributed practically no digestible protein to the diet. E. HOLMES.



**Nutritive value of gram husk.** F. J. WARTH and L. C. SIKKA (Mem. Dept. Agric. India, 1930, 11, 85—99).—Feeding tests on calves indicated that gram husk is comparable with wheat bran as a feeding-stuff. It has a distinctly lower starch-equivalent value than wheat bran, and provides no digestible protein; on the contrary, it has a depressing effect on the digestibility of the protein in the rest of the ration. Despite this, gram husk must be considered a concentrated food of merit. Observations are appended regarding European and American figures for starch-equivalent values.

E. HOLMES.

**Vitamin-D and the antirachitic activation of foods by irradiation with ultra-violet light.** F. L. GUNDERSON (Cereal Chem., 1930, 7, 449—455).—An outline of the known relationship which exists between occurrence of rickets, vitamin-D, and irradiation of foodstuffs, with special reference to cereals. Antirachitic potency of irradiated foods is stable during normal storage and household cooking, but may be partly destroyed at the higher temperatures of commercial baking.

E. B. HUGHES.

**Microscopy and histo-chemistry of certain fruits [citrus fruits, pears, plums, bananas, and tomatoes].** A. NIETHAMMER (Z. Unters. Lebensm., 1930, 59, 501—506).—The endocarp of oranges contains crystals of calcium oxalate, which can be isolated and identified by treatment of the fruit with phosphoric acid followed by sublimation under reduced pressure. After storage for several weeks, slow resorption of the crystals occurs with the concomitant formation of acetaldehyde. Lemons and mandarins exhibit identical phenomena. Pears contain characteristic inclusions, and in the ripe condition acetaldehyde can always be detected, the amount increasing considerably in the mellow and over-ripe states. Ripe pears on sublimation give malic and citric acids, whilst over-ripe samples give oxalic acid in addition. In plums the acetaldehyde test is always positive, and calcium oxalate can be detected. From bananas in the ripening stage, malic acid can be sublimed, but after prolonged storage traces only of oxalic and tartaric acids can be detected. Tomatoes in the unripe state contain appreciable amounts of malic acid with traces of oxalic and tartaric acids, whilst ripe samples contain malic, citric, and tartaric acids. Acetaldehyde is present in ripe tomatoes only.

H. J. DOWDEN.

**[Colorimetric] determination of copper in green vegetables.** A. HANAK (Z. Unters. Lebensm., 1930, 59, 511—512).—The sample (10 g.) of finely-ground material is treated with 20—30 drops of sulphuric acid and ignited, and the residue is then boiled with dilute (1:5) nitric acid and filtered. The filter and its contents are ashed and the ash is dissolved in the warmed filtrate. The solution is treated with ammonia in excess followed by ammonium carbonate and is then filtered. The residue is washed, redissolved, treated further with ammonia, and filtered. The united filtrates, if highly coloured, are compared against a solution of ammoniacal copper sulphate of known concentration. If only small amounts of copper are present, a dilute solution of gum arabic containing acetic acid and a few drops of potassium

ferrocyanide solution is used. The results obtained with peas, beans, and spinach of known copper content were very satisfactory.

H. J. DOWDEN.

**Diastatic activity of honey.** L. H. LAMPITT, E. B. HUGHES, and H. S. ROOKE (Analyst, 1930, 55, 666—672).—The dextrinogen-amylase activity of honey at various  $p_H$  values does not differ from that of most enzymes, whilst the optimum acidity was found to be at  $p_H$  5.0—5.3 whatever the temperature. A reaction period of 16 hrs. is adopted as a standard for the determination of the saccharogen-amylase activity, since the increase of reducing sugars is very slow after that period and the optimum  $p_H$  is about 5.3. At a  $p_H$  value below 4.0 and above 9.0 both the enzymes are destroyed, but at  $p_H$  4—6 the dextrinogenic activity seems to be stable. Measurements of diastatic activity are not reliable for detecting adulteration or heating of honey.

D. G. HEWER.

**Detection of carrots in marmalade.** A. HANAK (Z. Unters. Lebensm., 1930, 59, 513).—A portion of marmalade is stirred with ether, and if carrots be present the liquid acquires the characteristic colour of carotene. If the supernatant liquid is decanted and allowed to evaporate spontaneously, concentric rings are formed in which the presence of carotene may be verified. Conclusive proof must be found by microscopical examination of the original materials.

H. J. DOWDEN.

**Detection and determination of *p*-hydroxybenzoic acid and its esters in foodstuffs.** F. WEISS (Z. Unters. Lebensm., 1930, 59, 472—480).—A study has been made of the methyl, ethyl, and propyl esters and their sodium derivatives, which have recently been employed as preservatives for foods. Isolation of the preservative is effected by methods previously described (B., 1928, 386). The methyl ester may be identified by Denigès' method, and the ethyl and propyl esters by alkaline hydrolysis and distillation, followed by Griebel's micro-beaker method (cf. B., 1924, 922; 1925, 470). The alkaline residue is acidified, extracted with ether, and, after removal of the solvent, *p*-hydroxybenzoic acid may be identified in the residue by its m.p. (215°), by testing with Millon's reagent, and by the formation of the crystalline blue copper derivative. Salicylic and *o*- and *p*-chlorobenzoic acids do not interfere, but vanillin must be removed by warming with semioxamide. In the presence of esters etc. the free acid is best removed as the copper salt, or, alternatively, the other materials may first be removed by carbon tetrachloride, in which *p*-hydroxybenzoic acid is sparingly soluble, or by steam distillation. The acid may be determined by Zeisel's method, vanillin, if present, being removed by semioxamide. Examples are given of the application of the methods to sugar solution, raspberry juice, margarine, and mayonnaise, 60—100% of the *p*-hydroxybenzoic acid being recovered.

H. J. DOWDEN.

**Determination of fat in chocolate.** C. E. WISEMAN (Analyst, 1930, 55, 684—685).—The Gottlieb method for determining fat in condensed milk may be adapted to chocolate or cocoa by immersing the aqueous ammonia suspension of the material in boiling water



for 5 min., and subsequently adding rather more than the usual proportion of alcohol. D. G. HEWER.

**Determination of crude fibre [cellulose] in cacao.** K. KÜRSCHNER and A. HANAK (Z. Unters. Lebensm., 1930, 59, 484—494).—The method devised previously for the determination of cellulose in wood has been modified by the substitution of acetic acid for alcohol in the mixture used for decomposing the non-cellulosic constituents (cf. Kürschner and Hoffer, B., 1930, 608). The sample of defatted cacao (0.3 g.) is intimately mixed with 15 c.c. of 80% acetic acid and 1.5 c.c. of nitric acid ( $d$  1.4) in a flask fitted with a ground-in air-condenser. The mixture is gently boiled for 15—25 min. and then filtered through a large-pored porcelain or glass filter previously moistened with acetic acid. The residue is washed successively with 7—10 c.c. of the hot mixture of nitric and acetic acids, hot water, a few drops of alcohol, 5—10 c.c. of ether, 1—2 c.c. of the acid mixture, and finally hot water, until all traces of acetic acid have been removed, the flask and walls of the filter being washed at each stage. The residue is dried at 105—108° and weighed. By this method, which gives most consistent results, white undegraded cellulose was extracted from cacao and the determination required less than 1 hr. to make.

H. J. DOWDEN.

**Analysis of chocolate. II. Adulterated chocolate.** R. LECOQ (J. Pharm. Chim., 1930, [viii], 12, 149—159).—If the sum of the total sugar and thrice the insoluble matter other than fat exceeds 100% adulteration with cacao husks and germs, gluten flour, starch, etc. should be suspected. Addition of coffee or of nut paste must be detected microscopically or in the latter case by determining the iodine value of the fat. If  $x$  is the content of cacao butter and  $y$  that of nut oil, then  $x + y$  = fat content of the sample of chocolate. Also the iodine value of cacao butter being 36, and that of the nut oil being denoted by  $I$ , then  $36x + Iy$  = iodine value of the fat of the sample  $\times$  fat content of the sample. From these equations the value of  $y$  and, the fat content of the nuts being known, the proportion of nuts in the sample may be calculated. The fat content and iodine value of various nuts and nut oils are given. With mixtures of different nuts the problem is practically insoluble. In the case of milk chocolate of known components, the milk content may be found approximately by examination of the fat or by determination of the casein, but if nuts, honey, glucose, or other admixture is present it is impossible to form reliable conclusions as to the composition.

W. J. BOYD.

**Determination of crude fibre.** FEIST and KUNTZ. —See V. "Dry-ice." KILLEFFER. —See VII. Firing of boiler furnace with coffee-pod fuel. FREISE. —See VIII. Determination of butter fat. SCHWEIZER and GROSSFELD. Tung oil seeds. Sunflower seed. Dhupa kernels. *Moringa aptera* seed. ANON. Cherry-kernel oil. JAMIESON and GERTLER. —See XII.

PATENTS.

**Preparing green-coloured preserved vegetables.** P. DE BACKER (B.P. 337,354, 27.7.29).—Vegetables which would otherwise turn a yellowish colour during

processing may be kept a bright green by the addition of a harmless blue dye. Methylene-blue, water-blue 6B, indigo-carmin, Lyon-blue, and indanthrene-blue are suitable. E. B. HUGHES.

**Dehydration of vegetable substances and products of organic character.** B. J. OWEN, Assr. to SUGAR BEET & CROP DRIERS, LTD. (U.S.P. 1,781,473, 11.11.30. Appl., 27.11.26, U.K., 7.12.25).—See B.P. 267,203; B., 1927, 377.

**Sterilisation of liquids** (B.P. 337,027).—See I. **Alimentary salt** (B.P. 319,203 and 336,279).—See VII. **Extraction of fatty oil** (B.P. 336,273—4). **Edible oils** (U.S.P. 1,749,976).—See XII.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Analysis of Liquor aluminii acetico-tartarici and of Liquor aluminii acetici, D.A.B. VI.** R. HOLDERMANN (Pharm. Ztg., 1930, 75, 1331—1332).—The amount of aluminium acetotartrate in a sample of the former determined by evaporation ( $a$ ) at 100° (Official method); ( $b$ ) at 45° in a vacuum, or by precipitation of the aluminium; ( $c$ ) as hydroxide; ( $d$ ) as its 8-hydroxyquinoline derivative, was ( $a$ ) 44%, ( $b$ ) 48.1%, ( $c$ ) 53.3%, and ( $d$ ) 54.7%. The last result is in agreement with the method of preparation and the low values obtained in ( $a$ ) and ( $b$ ) are due to the formation of varying quantities of the basic salt,  $Al_2O(OAc)_2 \cdot C_4H_4O_6$ , during evaporation. This may also be present in the original solution. Liquor aluminii acetici is also conveniently standardised by means of 8-hydroxyquinoline.

H. E. F. NOTTON.

**Determination of potassium guaiacolsulphonate in syrups.** W. SCHÖBEL (Pharm. Ztg., 1930, 75, 1232—1233).—Previous methods (cf. D.A.B. VI; Rupp, B., 1928, 586; Rojahn and Struffmann, B., 1927, 617) are inconvenient and inaccurate. The proportions of potassium guaiacol-4-sulphonate (anhydrous), -5-sulphonate ( $+2H_2O$ ), and of the corresponding dipotassium salts ( $+2H_2O$ ) may be calculated from the alkalinity and water content of commercial samples, and official upper limits should be imposed for these values. The sulphur content, determined as sulphate after oxidation with permanganate, agrees with that obtained by the above method and remains practically constant ( $37.8 \pm 1\%$   $SO_4$ ) although the proportions of the three constituents vary widely. Syrups containing potassium guaiacolsulphonate are defecated with lead acetate and sodium phosphate, which, contrary to the statement of Rupp, do not precipitate the sulphonate, and the sulphur is determined in the above manner. The amount of drug is calculated from the mean sulphur content.

H. E. F. NOTTON.

**Determination of arsenic in pharmaceutical mixtures.** G. WEISSMANN and S. BABITSCH (Pharm. Zentr., 1930, 71, 721—724).—Several methods are reviewed (e.g., Rupp and Lehmann, A., 1912, ii, 866; Fridli, A., 1926, 591; and others) and the following, based on that of Andrews (A., 1909, ii, 637; 1914, ii, 291; 1915; ii, 279), is recommended. Liquid mixtures are evaporated to dryness, an amount containing 0.005—0.05 g. As being taken; the dry residue (or pulverised material in the case of pills etc.) is triturated with 20%



sulphuric acid, then treated dropwise with sulphuric acid and "perhydrol" until colourless. After dilution, removal of excess of hydrogen peroxide by permanganate, and decolorisation by oxalic acid, the colourless liquid is treated with 15% aqueous caustic soda and reduced with Thiele's hypophosphite solution (D.A.B. VI., 771) for 15 min., and set aside for 1 hr. The precipitated arsenic is collected, washed, and transferred to a stoppered flask; after addition of sodium carbonate, 0.1N-iodine is run in and the mixture shaken until the arsenic has completely dissolved, when excess of iodine is back-titrated with thiosulphate. Preparations containing iron are treated as above, except that stannous chloride is added to the mixture immediately prior to the addition of Thiele's reagent (cf. Wallrabe, B., 1928, 172). The precipitated arsenic may alternatively be oxidised by hydrogen peroxide (as above), dissolved in 25% hydrochloric acid and potassium iodide, and the free iodine in the diluted solution titrated with thiosulphate. Good agreement is shown by the two methods for such mixtures as Fowler's solution, Bland's pills, etc.

R. CHILD.

**Tinctures of cinchona. I. Preliminary report.** A. LICHTIN (Amer. J. Pharm., 1930, 102, 583—590).—The sedimentation observed in old samples of the U.S.P. X tincture also occurs to a smaller extent in the improved tinctures of Scoville (B., 1928, 139). It is not prevented by the addition of small quantities of acid, alkali, or oxidising or reducing agents, nor by refrigeration nor the use of special bottles. After a preliminary extraction of the bark with acetone, which removes tannins but practically none of the alkaloidal matter, a tincture prepared by the official method shows only a slight sediment after 3 months, whilst one obtained by percolation with 78% alcohol with subsequent addition of 7.5% of glycerin gives no sediment. H. E. F. NOTTON.

**Use of the centrifuge in determining morphine in opium.** C. STICH (Pharm. Ztg., 1930, 75, 1233—1234).—The precipitate of morphine obtained by the method of the Swiss pharmacopoeia is separated more rapidly and completely from the solution and wash waters by centrifugation than by filtration, and its greater purity renders the subsequent titration more precise.

H. E. F. NOTTON.

**Evaluation of drugs containing caffeine. II. Guarana and kola.** G. VON MIKÓ (Magyar Gyó Társ. Ert., 1930, 6, 30—42; Chem. Zentr., 1930, i, 2286).—A rapid macro- and a refractometric micro-method of evaluation, similar to those employed for tea (B., 1930, 585), are described. The micro-Kjeldahl method is applicable also.

A. A. ELDRIDGE.

**Evaluation of alkanet root: a colorimetric test.** W. A. N. MARKWELL and L. J. WALKER (Pharm. J., 1930, 125, 429).—The sample (5 g.) in No. 20 Powder is exhausted by continuous extraction in a Bolton-Revis apparatus with a mixture of 90% methylated spirit (50 c.c.) and benzene (50 c.c.). The extract is transferred to a 200-c.c. measuring flask, washings are added, and the volume is made up by addition of the mixed solvent. 2 C.c. of the resulting solution are diluted with solvent to 50 c.c. in a Nessler flask, when the colour should be equal in depth to that shown by a solution

prepared by mixing 40 c.c. of 0.01% (w./v.) aqueous potassium permanganate and 10 c.c. of 0.1% (w./v.) aqueous potassium dichromate (both of the latter being freshly made up).

R. CHILD.

**Steam-air method of treating tobacco.** P. K. DOROXOV, I. I. LOSEV, A. O. REPPIC, and A. A. SCHMUCK (U.S.S.R. State Inst. Tobacco Inv., 1930, Bull. 68, 36 pp.).—By the method described, which consists in treating the leaves with steam and air, and effects uniform moistening and sorting, the time occupied is reduced to a few minutes, all the conveying of the tobacco being by mechanical devices. At the same time the nicotine (recovered as sulphate), ammonia, and methyl alcohol are considerably diminished in amount and the flavour is improved. The process has been tested on a semi-manufacturing scale with satisfactory results.

T. H. POPE.

**Chemical composition of the [Russian] tobacco crops of 1927 and 1928.** V. BALABUCHA-POPCOVA and V. ZAPOLSKI (U.S.S.R. State Inst. Tobacco Inv., 1930, Bull. 66, 52 pp.).—A large number of data for tobaccos grown in different parts of Russia are given.

T. H. POPE.

**Orange oils from South Africa.** ANON. (Bull. Imp. Inst., 1930, 28, 282—284).—Three samples of orange oil, prepared in Pretoria, had the following respective constants:  $d_{15}^{25}$  0.848, 0.849, 0.849;  $\alpha_D^{25}$  +99.25°, +96.12°, +97.17°;  $n_D^{20}$  1.4735, 1.4735, 1.4730; non-volatile residue (at 100°) 2.3%, 3.6%, 2.9%.

E. H. SHARPLES.

**Petitgrain oil from Tanganyika.** ANON. (Bull. Imp. Inst., 1930, 28, 281—282).—A sample of the oil distilled in Tanganyika had  $d_{15}^{25}$  0.8957,  $\alpha_D^{25}$  —5.25°,  $n_D^{20}$  1.460, acid value 0.7, ester value 159.4, esters (as linalyl acetate) 55.8%, and gave a turbid solution even with 12 vols. of 80% alcohol at 15°. It compares favourably with Paraguayan oil.

E. H. SHARPLES.

**Ultra-violet window glazing.** BECKETT.—See VIII. **Cherry-kernel oil.** JAMIESON and GERTLER. **Moringa aptera seed.** ANON.—See XII. **Antirachitic activation of foods.** GUNDERSON.—See XIX.

## PATENTS.

**Manufacture of [pharmaceutical] complex iron compounds.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 335,965, 5.6.29).—A polyhydroxylated mono- or di-carboxylic acid is treated with iron or an iron compound, the mixture being neutralised simultaneously or subsequently with an alkali or organic base. Complex salts are prepared from sodium gluconate and ferric acetate or chloride; from potassium hydrogen saccharate and ferric chloride or ferric ammonium sulphate, with or without quinine; from pure iron, gluconic acid, and sodium hydroxide; from diethylamine mucate, ferric chloride, and sodium hydroxide.

C. HOLLINS.

**Manufacture of non-irritant organic arsenic compound capable of being injected.** I. G. FARBENIND. A.-G. (B.P. 318,835, 9.9.29. Ger., 7.9.28).—The bis-formaldehyde-bisulphite compound of arseno-(4-amino-1-phenyl-2 : 3-dimethyl-5-pyrazolone) is



treated with an alkaline-reacting solution, *e.g.*, aqueous sodium sulphite, so that the product precipitated by addition of alcohol and ether has in 1% aqueous solution  $p_H$  above 4 (*e.g.*, 4.5). C. HOLLINS.

**Purification, and manufacture, of hormone preparations.** SCHERING-KAHLBAUM A.-G. (B.P. 336,470—1, [A] 13.11.29, [B] 14.11.29. Ger., [A] 6.12.28, [B] 1.12.28).—(A) Hormone preparations from organs, body liquids, or vegetable materials are hydrolysed with water in presence of catalysts such as Twitchell reagents, Dartring saponifier, or lipase of castor seed, thus avoiding the necessity for the removal of the large quantities of salts produced during hydrolysis with alkalis. (B) Impurities are removed from solutions of hormones by treatment with a definite amount of a water-miscible organic solvent and, after filtration, the hormone is separated by the addition of an organic liquid which is immiscible with water.

E. H. SHARPLES.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

PATENT.

Films (B.P. 315,840 and 318,250).—See V.

## XXII.—EXPLOSIVES; MATCHES.

PATENTS.

[Preparation of] nitrocellulose [from wood pulp]. N. PICTON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 336,235, 27.5.29).—For the preparation of nitrocellulose of high density, wood-pulp sheets are cut into pieces about  $\frac{1}{8}$  in. square, so that the conglomeration of the fibres remains undisturbed and the product has a bulk density of 240–340 g. per litre. The mixed acid used for nitration must contain not less than 40% of nitric acid, a suitable composition to give nitrocellulose containing 11.7% N consisting of nitric acid 70%, sulphuric acid 20.5%, water etc. 9.5%.

W. J. WRIGHT.

**Continuous separation and after-separation of nitroglycerin and residuary acid.** A. SCHMID and J. MEISSNER (B.P. 336,253, 5.7.29).—An inclined separator, with cooling jacket, is employed, so that the emulsion of nitroglycerin and residuary acid, which is continuously introduced, being heavier than the nitroglycerin at the top and lighter than the residuary acid at the bottom, forms a horizontal layer at the zone of separation. As the acid flows downwards, drops of nitroglycerin separate at the top wall of the separator and move upwards, and owing to this separation and the cooling of the acid the latter gradually becomes heavier, so that circulation is ensured. Outlets are provided at the top and bottom for the nitroglycerin and residuary acid, respectively. The cooling jacket may be dispensed with if desired, or it may be replaced by an internal cooling coil and external insulation. A series of plates may be inserted in the separator in the direction of its longitudinal axis to guide the acid in its descent, and if an empty space is maintained at the upper end of these plates, a preliminary separation of the nitro-

glycerin takes place, a window at this part of the separator enabling the process to be watched.

W. J. WRIGHT.

## XXIII.—SANITATION; WATER PURIFICATION.

**Special equipment for protection against toxics in the chemical industry.** G. L. TURNER (Ind. Eng. Chem., 1930, 22, 1096–1099).—Industrial gas masks afford protection against every known gas, including carbon monoxide, for which a special catalyst is employed. Sectional drawings of the canister of the Burrell All-Service gas mask are shown. In confined spaces, where there is deficiency of oxygen, hose masks are used. Self-contained oxygen apparatus demands expert use, and is worn only in extreme emergencies. A description is given of the F.-M. indicator for detecting the percentage of inflammable gas, including the lower explosive concentration, in the atmosphere, and of a detector, designed by G. W. Jones, for rapid determinations. For determining carbon monoxide, the Hoolamite detector, involving a colorimetric test, gives rapid results; the Bell Telephone Laboratories employ palladium chloride in their device, the colour changing from canary-yellow to grey in presence of varying amounts of the gas. The M.-S.-A. continuous carbon monoxide recorder is briefly described. Rescue appliances include an inhalator, by means of which carbogen, a mixture of 93% of oxygen and 7% of carbon dioxide, is administered in cases of gassing. W. J. WRIGHT.

**Fatalities due to vitiated air produced by the oxidation of vegetable refuse.** A. R. TANKARD and D. J. T. BAGNALL (Analyst, 1930, 55, 673–676).—A case of poisoning due to carbon dioxide is reported.

D. G. HEWER.

**Soundness of concrete aggregates.** KRIEGER.—See IX.

PATENTS.

**Accelerating the action of breathing cartridges filled with peroxide.** DEUTS. GASLÜHLICHT-AUERGES.M.B.H., and HANSEATISCHE APPARATEBAU GES. (B.P. 337,170, 9.10.29. Ger., 7.11.28).—The initial rate of discharge of oxygen is accelerated by the discharge into the breathing bag of a small amount of a gasiform acid, *e.g.*, carbon dioxide, liberated from a small cylinder containing the gas under pressure. C. JEPSON.

**Insecticide.** H. V. McCLELLAN, Assr. to E. S. McCLELLAN (U.S.P. 1,758,734, 13.5.30. Appl., 24.2.27).—An insecticide containing finely-powdered ferrous carbonate (10–100 wt.-%) is claimed. It may be mixed or coated with starch and/or sugar. E. H. SHARPLES.

**Composition for repelling insects.** D. H. GRANT, Assr. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,755,178, 22.4.30. Appl., 29.12.28).—Solutions containing terpinyl esters, such as the acetate, propionate, etc., either alone or mixed with vehicles such as petrolatum, waxes, pyrethrum extract, kerosene or other mineral oils, and an emulsifying agent, are claimed.

E. H. SHARPLES.

**Cooling of air** (U.S.P. 1,749,763). **Dust filters** (B.P. 337,304).—See I. **Zeolites** (B.P. 319,746). **Glauconite** (U.S.P. 1,757,374).—See VII.