

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MARCH 2, 1928.

I.—GENERAL; PLANT; MACHINERY.

Small high-pressure autoclave. F. N. PETERS, JUN., and O. C. STANGER (Ind. Eng. Chem., 1928, 20, 74—76).—The autoclave is designed for the agitation of a liquid with hydrogen at 2000 lb./sq. in. pressure and temperature up to 200°. The shell consists of steel pipe with a T-piece and cap on one end, these joints being welded. The other end is machined and grooved and closed by a steel disc with lead gaskets. The hydrogen supply is connected to the T-piece by copper tube. Within the shell is a reaction tube with central opening, and agitation is effected by rocking longitudinally—a method which is very efficient. The shell is surrounded by asbestos cement insulation and provided with heating elements. C. IRWIN.

Apparatus for storing and circulating gases [in the laboratory]. A. H. WHITE and F. W. HIGHTOWER (Ind. Eng. Chem., 1928, 20, 95—97).—Two rubber balloons are each contained in an inverted bottle filled with water. These bottles are connected to each other and also to a higher water reservoir which rests upon one beam of a balance. Gas can be pumped from one balloon to the other. Any change in gas volume is immediately indicated and measured by the alteration of the water content of the reservoir. The pump used was of the mercury piston type, the mercury being operated by air pressure from a cycle pump geared to a motor. The apparatus provides a means of studying gas reactions without the use of any large quantity of mercury. The principal error arises from diffusion of gases through rubber, which is not serious during a few hours' work. C. IRWIN.

Gas mask. HEINRICH and PETZOLD.—See XXIII.

PATENTS.

Firing of furnaces with pulverised fuel. F. H. ROSENCRANTS (B.P. 281,850, 12.11.26).—For furnaces having fuel pulverisers directly connected to them, a slow-speed mill provides fuel sufficient for minimum load, and a high-speed mill that for the variable increase. A small variation in the speed of the first mill may be obtained by altering the speed of its motor. B. M. VENABLES.

Rotary hearths for furnaces. C. NELSON, and KING, TAUDEVIN, & GREGSON, LTD. (B.P. 282,135, 9.9.26).—The rotary hearth is supported on a ball bearing and driven by a worm, the whole mechanism being submerged in a shallow oil bath. The upper ball race is fixed to the hearth, but the lower is free to accommodate itself horizontally on the bottom of the bath. The hearth is centred horizontally by an axial pivot which is free vertically. B. M. VENABLES.

Heat-transmitting tubes. W. J. STILL (B.P. 282,244, 16.9.26).—A description is given of machines for winding wire on the outside of tubes to increase the heat-transmitting surface. B. M. VENABLES.

Pulverisers. H. WARING, and ASSOCIATED LEAD MANUFRS., LTD. (B.P. 282,145, 13.9.26).—A high-speed disintegrator for soft and fibrous material. B. M. VENABLES.

[Grinding body for] tube and drum mills. E. C. R. MARKS. From G. POLYSIUS (B.P. 281,528, 13.4.27).—The ends of a helical spring are bent across the axis, and in the space so enclosed are placed one or more loose cylinders or balls. A number of these elements form the ball charge of the mill. B. M. VENABLES.

Apparatus for stirring and mixing. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 281,852, 15.11.26).—In a vessel the contents of which are agitated by a propellor with inclined or helical blades, other fixed blades are used of opposite inclination to the moving ones, and so arranged that the circulating liquid passes through the fixed guides first. B. M. VENABLES.

Mixing and stirring apparatus. F. A. EASTERBROOK, and BROWN & SON (ALEMBIC WORKS), LTD. (B.P. 281,904, 25.2.27).—A form of mixing propellor suitable for the reconstitution of dried milk and similar purposes. B. M. VENABLES.

Apparatus for drying. A. F. WRIGHT (B.P. 281,785, 14.9.26).—A drying atmosphere is circulated by a fan through drying chambers, which alternate with re-heating chambers, to a conditioning chamber where a proportion of fresh air is admitted, back to the fan. The exhaust air, fresh air, and recirculated air are controlled by separate valves, and the re-heaters are controlled by a thermostat in the return air duct. B. M. VENABLES.

Centrifugal dryers and separators. A. HABERMANN (B.P. 265,561, 5.1.27).—The apparatus comprises a perforated drum—preferably conical—and a separately driven internal worm, both of which revolve at high speed, but have a slow relative motion by which, in conjunction with longitudinal ridges on the interior of the drum, the dried material is conveyed to the outlet end of the drum. A cylindrical extension of the drum carries scrapers to remove any discharged material adhering to the outer casing. B. M. VENABLES.

Centrifugal separator and dryer. P. FESCA (C. A. FESCA & SOHN) (B.P. 278,327, 15.8.27. Ger., 1.10.26).—In a centrifuge in which the basket comprises two cones base to base, held together when separating liquid from solids, and separated when discharging the collected

solid, the two portions are held together by a hydraulic cylinder and piston within the basket.

B. M. VENABLES.

Centrifugal machine. H. J. GREAVES (B.P. 281,483, 16.12.26).—The basket of a centrifugal separator is formed of a number of channel-shaped, vertical slats through which the liquid is filtered off continuously. Within and without the basket are shells rotating together at a very slightly different speed from that of the basket. The inner shell is provided with ports for supplying feed to the basket and the outer shell with an equal number of ports for discharge of solid matter. The same gearing which causes the slow differential speed also overturns the slats of the basket in turn when they are opposite the discharge ports. The inlet ports are always just behind the discharge ports.

B. M. VENABLES.

Apparatus for separating solid materials by the float and sink method. C. N. KEMP and J. L. THOMSON (B.P. 281,479, 13.12.26).—The material to be separated is placed in a cylindrical basket with perforated bottom, which is then placed in a cylindrical tank. Liquid of high sp. gr. is allowed to enter the tank from the bottom. The float is skimmed off by a perforated scoop which normally lies in a recess at the side of the tank. After the scoop has been placed under the float the liquid is drained off, and not till then is the scoop lifted. Thus all splashing and waste of liquid are avoided. The process may be repeated with heavier liquids in succession. (Cf. B., 1927, 641.)

B. M. VENABLES.

Separation or extraction of liquids from materials. ILLINGWORTH CARBONIZATION CO., LTD., and S. R. ILLINGWORTH (B.P. 281,390, 1.9.26).—The liquid is drained on a moving porous surface, and the liquid is removed from the other side of the porous surface by other moving means such as porous belts or brushes.

B. M. VENABLES.

Salt or evaporating pans. VEREIN. SCHWEIZERISCHE RHEINSALINEN (B.P. 277,639, 25.8.27. Switz., 15.9.26).—A mechanically-driven scraper for discharging salt comprises a carriage travelling longitudinally to and fro and supporting a hollow shaft across the pan, on which scraper arms can rock freely. The hollow shaft also carries, clamped to it, shorter lifting arms. Within the hollow shaft is a solid shaft which effects the reciprocating motion of the carriage.

B. M. VENABLES.

Containers for treating corrosive liquids etc. BEACON OIL CO., and H. H. HEWETSON (B.P. 281,928, 20.4.27).—A vessel suitable for treating oils at a high pressure and at temperatures between 230° and 480° (or other liquids of which the vapours are corrosive) is constructed with an easily renewable upward extension provided with pipes for oil and vapour and with a safety valve, and the apparatus is so worked that the liquid level is always kept in the comparatively small extension, which, as a further economy, may be lined inside with corrosion-resisting material.

B. M. VENABLES.

Production of filter-sieves. A. PAHL (B.P. 281,879, 29.12.26).—A filter sieve for liquids or gases is produced by dipping a woven wire screen in molten glass or enamel, and applying small fragments of porous lime, charcoal, or chalk before the glass or enamel has time to

set. The screens when in use may be saturated with solutions of caustic alkali or chlorine.

B. M. VENABLES.

Screening of materials. J. BURR (B.P. 282,176, 22.9.26).—The screen consists of a number of longitudinal spaced bars or slots, into which fingers carried by a continuous band or chain project. The motion of the fingers is upwards as well as longitudinal so that the material is rolled over the screen, not merely pushed along.

B. M. VENABLES.

Screening apparatus. R. A. LEAHY (U.S.P. 1,650,896, 29.11.27. Appl., 8.12.23).—The screens are agitated by means of a pulsating column of liquid, which is subjected to the action of a reciprocating piston.

T. S. WHEELER.

Apparatus for effecting intimate contact of gases and liquids. S. WRIGHT (B.P. 281,958, 9.6.27).—An apparatus similar to a rectifying tower is provided with centrifugal sprayers adapted to lift liquid from each tray. The space above each tray of liquid is subdivided horizontally and the gas is caused to pass round each sub-compartment before passing to the next.

B. M. VENABLES.

Apparatus for determining the strength of solutions [by colorimetric assay]. C. KOCOUR (B.P. 270,338, 30.4.27. U.S., 1.5.26).—In addition to the vials containing the assay and standard, others are used containing colours which will neutralise the colour of the assay; e.g., sight may be taken through a green assay superposed on a red vial. Several red vials may be used containing fractions of the constituent being determined, as well as the green standards for the units.

B. M. VENABLES.

Gas detector. W. JOENS (B.P. 273,296, 21.6.27. Ger., 24.6.26).—A gas detector for indicating the presence of a constituent which differs in density from the bulk of the atmosphere to be tested comprises a porous body, an indicating device in the form of a thin metal plate gilded all over, which, when distorted by the diffusion pressure, forms an electrical contact, and a battery, the whole being contained in a stick-like container. The contact in the diaphragm chamber has a screw adjustment, and the screw may be provided with a pointer indicating the percentage of the foreign constituent in the gas.

B. M. VENABLES.

Grinding mill. J. B. C. SCHERBAUM (U.S.P. 1,656,503, 17.1.28. Appl., 11.2.27. Ger., 15.2.26).—See B.P. 266,009; B., 1927, 432.

Mixing and agitating [means for freezing] machines. C. H. CROWLEY, and WATERMOTORS, LTD. (B.P. 281,833, 30.10.26).

Pneumatic suction apparatus for the removal of dust and like finely-divided material. PNEUMATIC CONVEYANCE & EXTRACTION, LTD., and W. A. SMITH (B.P. 281,811, 5.10.26).

Dust extractors. WOODALL-DUCKHAM (1920), LTD., S. N. WELLINGTON, and A. SCOTT (B.P. 282,258, 5.4.27).

Dust separator [for gases]. A. M. GOODLOE, Assr. to MIDWEST STEEL & SUPPLY CO., INC. (U.S.P. 1,649,220, 15.11.27. Appl., 6.12.24).

Apparatus for removing dust and other solid matters from air and other gases. D. HALL, J. H. KAY, and HALL & KAY, LTD. (B.P. 282,233, 9.2.27).

Apparatus for the removal of dust from gases. ATELIERS J. HANREZ SOC. ANON., and A. MODAVE (B.P. 277,281, 15.12.26. Belg., 13.9.26).

[Stop device for] apparatus for expressing liquid constituents from materials of various kinds. F. KRUPP GRUSONWERK A.-G. (B.P. 273,294, 20.6.27. Ger., 23.6.26. Addn. to B.P. 252,005; B., 1927, 241).

[Means for removing non-condensable gas from] refrigerating systems. W. H. CARRIER, and CARRIER ENGINEERING CORP. (B.P. 281,356, 20.7.26).

II.—FUEL; GAS; TAR; MINERAL OILS.

Lancashire coalfield. Wigan Four-feet seam. ANON. (Dept. Sci. Ind. Res., Fuel Res., 1927, Physical and Chemical Survey of the National Coal Resources, No. 10, 1927, 60 pp.).—Samples of coal taken from the Wigan Four-feet seam in ten different districts have been examined in detail. The seam is of good quality, consists of fairly hard and moderately bright coal containing a certain amount of durain, and is seldom split by thick shale or dirt bands. The thickness varies from about 2 ft. on the east to a maximum of 6 ft. 3 in. at Garswood, thence thinning slightly towards St. Helens. The seam yields a good coking coal, which is used chiefly as a house or as a gas coal; it produces a coke which is usually only slightly swollen. The moisture content is normal, about 2–3%, except for the Garswood sample (5.6%). The ash content varies from 2.6 to 9.9%, the average, however, lying between 3 and 4%. The average volatile matter content is high. The sulphur content tends to be high in the Tyldesley and Atherton districts, but elsewhere is comparatively low. The results of laboratory carbonisation assays at 600° indicate the suitability of this coal for gas manufacture. Washing tests made on three of the samples show that the coal lends itself to purification by both float-and-sink and froth-flotation methods. A. B. MANNING.

Cannel and pseudocannel coals. R. KATTWINKEL (Brennstoff-Chem., 1927, 8, 375–376).—A cannel and a pseudocannel coal from the Ruhr gave, respectively, on analysis: ash, 4.80% and 2.86%; volatile matter, 39.55% and 25.23%; carbon, 82.15% and 86.98%; hydrogen, 5.82% and 4.81%; oxygen, 5.08% and 2.80%; d 1.278 and 1.343. The results for the pseudocannel coal are characteristic of a "bright" coal, and it gives a strongly coherent coke button. A low silica content (17.1%) and high ferric oxide content (38.2%) are characteristic of the ash of the pseudocannel coal, the cannel coal ash containing 35.2% SiO_2 and 7.3% Fe_2O_3 . The latter also has a high alkali content. The difference between the two coals is maintained on low-temperature carbonisation, but they behave more similarly on high-temperature carbonisation. For the above reasons the pseudocannel coal is to be classified as intermediate between "bright" and "dull" coals, a conclusion agreeing with that based by Winter (Glückauf, 1913, 49, 1410) on a microscopical examination. W. T. K. BRAUNHOLTZ.

Spontaneous ignition of coal. D. J. W. KREULEN (Brennstoff-Chem., 1927, 6, 340–343).—Powdered coal (50-mesh), previously dried for $\frac{1}{2}$ hr. at 105°, was heated for 2 hrs. at 175° (actual temperature of the coal) in an air oven made of sheet asbestos and so designed that a thermometer dipped into the coal in the crucible and that air was constantly circulating over the sample. The partly oxidised coal was extracted with sodium hydroxide, and the quantity of humic acids produced was determined colorimetrically. This quantity increases with increasing content of volatile matter in the coal, and, in general, a definite relation exists between the two (both reckoned on the dry and ash-free coal). At the same time cases are found of coals with the same content of volatile matter having quite different susceptibilities to oxidation. For example, the Plessey and Yard seams (Newbiggin) contain 37.2% and 37.3% of volatile matter, respectively, but yield, under the above conditions, 159 mg. and 57 mg. of humic acids per g. of coal substance. By placing these two coals in adjacent crucibles in the asbestos oven, the temperature of which is gradually raised, and noting the temperature of each coal sample and of the surrounding air, the temperature of the Plessey and Yard coals is found to rise above that of the air at 115° and 162°, respectively, indicating the greater susceptibility of the former to oxidation.

W. T. K. BRAUNHOLTZ.

Reactions between oxygen and coal. W. FRANCIS and R. V. WHEELER (J.C.S., 1927, 2958–2967).—Oxidation of newly-won coal at 60° with oxygen proceeds through an adsorbed layer, which reacts with the external groupings of the ulmin molecule. These oxygenated groupings decompose giving carbon oxides and water with the formation of new, unstable oxygenated groupings. On raising the temperature in a vacuum, the latter decompose into the same products, which contain an amount of oxygen equal to that "fixed" in the oxidation. On repetition of this revivification process the decomposition products alter in amount owing to alteration in the character of the external groupings. The groupings of the soluble ulmin prepared by atmospheric oxidation of this coal at 150° are converted probably into carboxylic groups by similar oxidation in oxygen. Revivification cannot be effected to any great extent since the decomposition leaves a comparatively stable substance. G. A. C. GOUGH.

Differentiation between lignite and coal. W. FUCHS (Brennstoff-Chem., 1927, 8, 337–340).—Lignites are composed mainly of humic acids, whilst coals are almost or entirely free from them. A differentiation is thus afforded by determining the total humic acid (free and combined) present in the given sample. The free humic acid is determined by shaking the sample (10 g.) with *N*-calcium acetate (100 c.c.), filtering, and titrating the acetic acid liberated with 0.1*N*-alkali, the equivalent of humic acid being taken as 350. The humic acid combined with a metal (other than calcium) is found by making use of the permutit nature of the humates (cf. B., 1927, 833). The calcium in the original filtrate is precipitated as oxalate, and the latter titrated with permanganate in acid solution. Finally, the calcium humate is found by shaking the sample with *N*-sodium acetate and determining the calcium as above,

the reaction being the reverse of the previous one. The value found for free humic acid is probably, that for calcium humate certainly, too low, whereas the value for the humic acid combined with metals other than calcium is probably too high. The total humic acid present, however, affords a clear differentiation between typical lignites and coals, being not less than 60% for lignites (in the dry sample) and not more than 5% for coals.

W. T. K. BRAUNHOLTZ.

Alleged discovery of "caramelic acid." W. FUCHS (*Z. angew. Chem.*, 1928, **41**, 85—88).—The so-called caramelic acid which Marcusson claims to have isolated (cf. B., 1928, 76) both from humic acid and from dextrose by oxidation does not in fact exist. Material prepared from humic acid, following exactly the directions given by Marcusson, is not a definite compound, does not give a lead salt of constant lead content, and proves in fact to be partly changed starting material. The product obtained from dextrose by following Marcusson's directions is entirely different from that obtained from humic acid.

S. I. LEVY.

Reactivity of coke. I. Standardised method for the determination of comparative values. J. H. JONES, J. G. KING, and F. S. SINNATT (*Dept. Sci. Ind. Res., Fuel Res.*, 1927, *Tech. Paper No. 18*, 32 pp.).—The reactivity of coke has been determined by measuring the volume of carbon monoxide produced when 100 c.c. of carbon dioxide are passed through a column of coke maintained at 950°. An apparatus suitable for such determinations is described in detail. The coke is ground to pass 10-mesh and remain on 20-mesh; the coke column, contained in a silica tube, is 7.5 cm. in length and 1.4 cm. in diameter; the rate of passage of carbon dioxide is 5 c.c./min. These arbitrarily chosen standard conditions are such that the reactivities, which are expressed as c.c. of carbon monoxide produced, will lie between 30 and 140 for most normal coals. In general, the reactivity falls with continued passage of nitrogen or carbon dioxide over the heated coke. For each coke investigated three reactivities have been determined, (a) the initial value, (b) the constant value reached after 5 hrs. passage of nitrogen at 950°, and (c) the approximately constant value reached after continued passage of carbon dioxide at 950°. The last is not strictly constant, being further slowly reduced by prolonged passage of the gas. The same value of c is reached either directly from a or via b. Typical results for the three reactivities of good metallurgical coals such as South Wales coke, Durham coke, and Yorkshire beehive are: (a) 69, 72, 70; (b) 69, 63, 67.5; and (c) 40, 48, 67.5, respectively. These may be contrasted with the corresponding values for horizontal-retort gas coke, by-product coke, and an active charcoal, which are: (a) 145, 138, 192; (b) 131, 112, —; and (c) 78, 95, —, respectively. The form of the curve obtained when reactivity is plotted against the volume of carbon dioxide passed over the coke appears to afford a good indication of its value for industrial purposes. There is also some evidence of a relationship between the reactivity and the hardness of a coke, the resistance to the "shatter" test increasing with decreasing reactivity. The fall in reactivity from a to b is a result of the removal

of volatile matter, but the cause of the further fall on the continued passage of carbon dioxide has not yet been fully elucidated. Some phenomena connected therewith, and in particular a transitory reactivation which occurs with certain coals, are briefly discussed.

A. B. MANNING.

Production of carbon by the decomposition of carbon monoxide. VON WANGENHEIM (*Brennstoff-Chem.*, 1927, **8**, 385—388).—When carbon monoxide is passed over pure iron oxide at 650°, deposition of carbon occurs until the iron content of the catalyst is reduced to about 1%. The presence of hydrogen in the gas increases the carbon deposition. The iron in the carbon cannot be completely removed, either by the action of acids or by heating in a current of chlorine and volatilising the ferric chloride formed. The final carbon, containing traces of iron, was not magnetic, but was a conductor of electricity.

W. T. K. BRAUNHOLTZ.

Production of pure carbon at low temperatures. F. FISCHER and P. DILTHEY (*Brennstoff-Chem.*, 1927, **8**, 388—391; 1928, **9**, 24—30; cf. preceding abstract).—Carbon is deposited when carbon monoxide or water-gas is passed over a catalyst containing ferric oxide at about 500°. The gas must be free from sulphur compounds, but even with sulphur-free gas an initial increase in the activity of the catalyst, followed by a decrease in activity, is observed. Hydrogen sulphide, produced by the action of water-gas on sulphur compounds in the iron on which the catalyst is supported, reduces the activity of the latter, but this is restored by dipping the iron in 0.1N-sodium carbonate. A soft iron, particularly free from sulphur, was found the most suitable carrier for the catalyst, which was painted on in the form of a mixture of ferric oxide and sodium silicate. The apparatus, in its final form, consisted of a vertical, externally-heated iron tube, in which Ω -shaped strips of iron, carrying the catalyst, were suspended. The gas passed over these strips and the deposit of carbon was knocked off them from the top of the tube and collected at the bottom, which was constricted and closed by a stopper. The composition of the gases leaving the apparatus was determined with an Orsat apparatus or, preferably, with an interferometer. Various proportions of carbon monoxide and hydrogen in the initial gas were used, a gas richer in carbon monoxide giving a decreased formation of methane (as by-product) and an increased carbon yield. The carbon was pyrophoric, and contained small quantities of iron, probably in the form of a carbide which can be dissolved with 2.5N-hydrochloric acid. Chemical and X-ray tests appear to indicate the presence of graphite in the carbon. The reactivity of the carbon towards carbon dioxide is intermediate between that of wood charcoal and metallurgical coke, removal of iron decreasing the reactivity. The sulphur-free carbon might find application in the iron industry, and carbon monoxide can be continuously regenerated from the carbon dioxide in the final gases by passing the latter over red-hot coke. As an example of the yield obtained in continuous working, the passage of 26.3 cub. m. during 173 hrs. gave 1.27 kg. of carbon without any diminution in the activity of the catalyst. The iron content of the carbon may be up to 7%, and is greater the more active the catalyst.

W. T. K. BRAUNHOLTZ.

Synthesis of methane from water-gas. F. W. HIGHTOWER and A. H. WHITE (Ind. Eng. Chem., 1928, 20, 10—15).—Further data on the catalytic reduction of carbon monoxide or dioxide to methane are provided by analysis of the gases produced when water-gas is circulated over a nickel catalyst; this was prepared on pumice and heated in an electric furnace at temperatures between 280 and 377°. The conversion into methane reaches a maximum at about 350°, but the observations are complicated by deposition of carbon, and are not proved to represent equilibrium conditions. Recirculation of the gas, after cooling to remove some of the water produced, shows that the reduction of carbon monoxide is the main reaction, but that dissociation of the monoxide to dioxide and carbon also occurs; when no water vapour is removed the reactions are modified in accordance with theoretical predictions. The reduction of carbon dioxide proceeds without any side reactions. Values of the equilibrium constants ($\log_e K_p$) for the composite reaction $\text{CO} + \text{CO}_2 + 7\text{H}_2 = 2\text{CH}_4 + 3\text{H}_2\text{O}$ have been determined and are compared with those given by calculation from data published by other workers; the new experimental results are generally lower, and suggest that equilibrium conditions have not been reached.

R. H. GRIFFITH.

Equilibrium conditions in the formation of hydrocarbons and alcohols from water-gas. H. TROPSCH (Brennstoff-Chem., 1927, 8, 376).—Smith has deduced (B., 1927, 593), from calculations of the equilibrium constants, that increasing temperature should favour the formation of higher rather than lower paraffins from water-gas, whereas in reality it favours the formation of methane. The further conclusion that increased pressure should tend to produce the higher paraffins agrees with the results of experiments carried out under conditions in which these compounds remain in the catalyst. At higher temperatures, however, at which these higher paraffins are expelled from the catalyst, oxygenated substances are obtained, possibly as secondary products.

W. T. K. BRAUNHOLTZ.

Estimation of firedamp: flame caps. C. S. W. GRICE and D. W. WOODHEAD (Safety in Mines Res. Bd., Paper No. 37, 1927, 8 pp.).—The "caps" visible on the flame of a safety lamp when the latter is introduced into atmospheres containing varying amounts of firedamp have been photographed. A vignetting method was used to avoid the halation which otherwise is caused by the testing flame when sufficient exposure is given to obtain the detail of the cap. The prints were dyed to reproduce the blue colour of the flame caps, and the yellow of the testing flame was then added by hand. Coloured reproductions of these are given.

A. B. MANNING.

Blue oil of lignite producer tar and its relationship to the azulene and the sesquiterpenes of oil of camomile. S. RUHEMANN and K. LEWY (Ber., 1927, 60, 2459—2469; cf. B., 1926, 42).—Azulene is isolated from the neutral fractions of high b.p. of the tar by treatment with hydroferrocyanic acid followed by action of alkali hydroxide on the precipitate thus produced; separation of azulene from the mixture of

liberated hydrocarbons is effected by dissolution in concentrated sulphuric acid and subsequent precipitation with water. The azulene is converted into its *picrate*, $\text{C}_{15}\text{H}_{18}\text{C}_6\text{H}_2(\text{NO}_2)_3\cdot\text{OH}$, m.p. 116°. Camomile oil on treatment with hydroferrocyanic acid gives the *compound*, $\text{C}_{15}\text{H}_{18}\text{H}_4\text{FeC}_6\text{N}_6$; azulene, b.p. 163°/14 mm., derived from this source is shown to be identical with that from lignite tar by comparison of the picrates. The validity of the optical method used by Herzenberg and Ruhemann (*loc. cit.*) in the examination of azulene from lignite tar and gurjunene is thus established (cf. Ruzicka and Rudolph, A., 1926, 299). Catalytic hydrogenation of azulene in the presence of colloidal palladium gives colourless *octahydroazulene*, $\text{C}_{15}\text{H}_{26}$, b.p. 123—124.5°/10 mm., d^{15}_4 0.8967, n^{15}_D 1.4921, the optical constants of which suggest the presence of a difficultly hydrogenated double linking (cf. Ruzicka and Rudolph, *loc. cit.*) or a tricyclic system, and are not in harmony with the benzofulvene structure assigned to azulene by Kremers (A., 1923, i, 454). Further, the power to react with hydroferrocyanic acid is not exhibited by dimethyl- or diphenyl-benzofulvene or by indene or hydrindene. A fraction of camomile oil, b.p. 160—170°/14 mm., ester number 41, after being freed from azulene was hydrolysed, but the alcohol could not be isolated in substance, as hydrogen phthalate, phenylurethane, or triphenylmethyl ether; a non-crystalline benzoate was obtained. Treatment of the crude alcohol with potassium hydrogen sulphate at 180° gives a non-homogeneous sesquiterpene *A*, $\text{C}_{15}\text{H}_{24}$, b.p. 137—139°/13 mm., d^{17}_4 0.8975, n^{17}_D 1.5046. Repeated distillation of the fractions of camomile oil, of b.p. 130—140°/10 mm. and 120—130°/10 mm. yields a *sesquiterpene C*, b.p. 129—131°/10 mm., d^{20}_4 0.8998, n^{20}_D 1.5029, $[\alpha]^{20}_D + 16.13^\circ$ in chloroform, in small amount and a *sesquiterpene B*, b.p. 124—125°/10 mm., d^{20}_4 0.8689, n^{20}_D 1.4905, $[\alpha]^{20}_D - 4.35^\circ$ (*hydrochloride*, $\text{C}_{15}\text{H}_{27}\text{Cl}_3$, m.p. 45°). The compound *B* appears to be monocyclic. It is converted by hydrogen in the presence of a nickel-aluminium oxide catalyst into the *hexahydride*, $\text{C}_{15}\text{H}_{30}$, b.p. 114—115°/10 mm., d^{15}_4 0.8200, n^{15}_D 1.4531, which is not dehydrogenated to a derivative of naphthalene. Azulene does not appear to exist preformed in camomile oil, but is apparently derived by fermentative action from sesquiterpenes.

H. WREN.

Tar from Russian oil shale. G. STADNIKOV and A. WEIZMANN (Brennstoff-Chem., 1927, 8, 343—345).—Kaschper oil shale, containing 13.4% of moisture, 46.2% of ash, and 3.8% S, when distilled in a Fischer retort, gave 11.1% of tar, the yield being raised to 15% by distilling in a current of carbon dioxide. The light oil from the tar was treated with formaldehyde and naphthasulphonic acid ("Petrov's contact") to remove acidic sulphur compounds and a portion of the unsaturated compounds, and the treated oil was distilled with steam and the distillate washed with dilute sulphuric acid and dilute sodium hydroxide. The remaining oil was fractionally distilled, and the distillate boiled with and finally redistilled over metallic sodium. The fractions of the distillate (having b.p. ranging from 105° to 220°) had high sulphur contents (11.6—7.6%), and were acetylated with acetyl chloride in the presence

of stannic chloride. Analyses of the purified and re-distilled acetyl products proved the presence in the original oil of a thioxen or ethylthiophen, and of propylthiophen or one of its isomerides.

W. T. K. BRAUNHOLTZ.

Action of strong sulphuric acid on olefines and alcohols. W. R. ORMANDY and E. C. CRAVEN (J. Inst. Petrol. Tech., 1927, 13, 844—854).—The reactions involved in determining the unsaturated content of liquid hydrocarbon mixtures by absorption in sulphuric acid are investigated, attention being concentrated on oily reaction products obtained with olefines and alcohols, and sulphuric acid. Ethylene showed no hydrocarbon formation, but propylene, amylene, and mixed olefines obtained from cracked spirit all gave good yields of open-chain paraffins and highly unsaturated substances which remain dissolved in the acid. If olefines and aromatic hydrocarbons are treated simultaneously, the latter also are involved in the reaction. *iso*- and *n*-Propyl and amyl alcohols, cyclohexanol, and 1- and 4-methylcyclohexanols similarly gave two types of reaction product, one of which remained in the acid; the oily products from open-chain alcohols were open-chain paraffins, but those from cyclohexanols were cyclic hydrocarbons.

R. H. GRIFFITH.

Purification of benzol for motor fuel. F. T. HATSWELL (Gas World, 1928, 88, Coking Sect., 11—14).—Crude benzol is washed with dilute acid, and, after separation of the latter, is treated with the purifying medium at 50—60° for about 1 hr. It is then filtered through flannel, neutralised, and finally distilled. The medium, which is a finely-divided mixture of an absorbent material and ferric sulphate in an active form, polymerises or interacts with the impurities and then adsorbs the products of these reactions. Large-scale tests have shown that the losses are much smaller than by the usual acid washing, and that a motor benzol is produced which, although not able to pass the acid tests, is stable, free from gumming materials and sulphur, and gives a better performance in the engine than a normal high-grade benzol. The cost of the process compares favourably with that of the usual acid washing.

A. B. MANNING.

Causes of varying sp. gr. of gas-works benzol. A. SCHNEIDER and W. KONRAD (Gas- u. Wasserfach, 1927, 70, 1188—1189).—Gas-works benzol, having d_{4}^{20} 0.840, was found to contain only 1.7% of paraffins. Its refractive index (1.481—1.490 for fractions of b.p. 60—83°) suggested the presence of considerable quantities of cycloparaffins, which would also account for the low sp. gr. These were not derived from the wash oil, but were produced in the process of gas manufacture, particularly when the carbonising temperature fell at times below 1000°.

W. T. K. BRAUNHOLTZ.

Phenols from natural petroleum. Y. TANAKA and R. KOBAYASHI (J. Fac. Eng., Tokyo Imp. Univ., 1927, 17, 127—133, and Proc. Imp. Acad., Tokyo, 1927, 3, 595—596).—The greater part of acid substances found in petroleum consists of naphthenic acids, but phenols are also present; those occurring in the kerosene fraction of Japanese petroleum have been isolated and examined. The oil is treated with strong aqueous caustic soda,

and the crude mixture obtained on acidifying this solution is redissolved in a limited amount of alkali. The difference in the degree of hydrolytic dissociation of salts of naphthenic acids and phenols makes possible the selective extraction of the latter by means of ether. The purified mixture of phenols obtained by several repetitions of this treatment was fractionated and the physical and chemical properties of the fractions were determined; *o*- and *p*-cresols and *o*- and *m*-xylenols were determined by interaction with chloroacetic acid, and *m*-cresol by formation of its trinitro-derivative. The phenols thus identified were 50% of *p*-cresol, with less *o*- and *m*-cresols, and small amounts of *m*- and *o*-xylenols, diethylphenol, and triethylphenol.

R. H. GRIFFITH.

Cholesterol as parent of petroleum. W. STEINKOPF (Ber., 1927, 60, 2609—2610).—Zelinski's view that cholesterol is the parent of petroleum (B., 1927, 865) cannot be upheld since it has not been available in sufficient quantity. It is possible that decomposition products of cholesterol may be mixed with petroleum. By use of kieselguhr in place of aluminium chloride the author has obtained from cholesterol products very similar to those described by Zelinski (cf. A., 1921, i, 24).

H. WREN.

Corrosion by oil. H. J. YOUNG (J. Inst. Petrol. Tech., 1927, 13, 760—772).—Two cases of crank-shaft corrosion in motor-ships were traced to lubricating oils which, according to standard tests, had no properties usually associated with corrosive action. A direct test has accordingly been devised in which warm oil flows continuously over slabs of steel kept at a uniform temperature; part of the surface is white-metalled with bearing metal in order to produce conditions similar to those in the engine, and the specimens are polished before treatment. Examination of numerous oils, including some contaminated with acid or other inorganic impurities, showed that determination of inorganic acid was valueless, but determination of the "sulphate value" would indicate roughly the behaviour of a lubricant; the new test is, however, more sensitive and less laborious.

R. H. GRIFFITH.

Resins in lubricating oils and their action on steel. M. W. BORODULIN (Petroleum, 1927, 23, 1515—1518).—The Akzys method is the most satisfactory for determining the content of resin-forming compounds in lubricating oils. The oil, diluted with an equal volume (or more) of standard benzine, is shaken with 95% sulphuric acid and the increase in volume of the acid layer is taken as representing the volume of the resin-forming compounds present in the oil. The modification of Armani and Rodano's method (B., 1921, 138 A) does not give satisfactory results, and may even give a negative value for the resin content. The rusting of steel plates coated with oils of varying resin content bears no relation to the latter, but depends mainly on the viscosity of the oil. Indeed, the protective action of oils on steel is enhanced by increasing their viscosity by addition of resin, and oils from which the resins have not been completely removed afford better protection than those that have been more thoroughly refined.

W. T. K. BRAUNHOLTZ.

Extension of the formolite analysis of lubricating

oils. A. M. NASTUKOV (Petroleum, 1927, 23, 1451—1452).—The Akzys test does not give the same value for lubricating oils as the fourth formolite value, although it does for crude oils. The content of saturated and unsaturated hydrocarbons, in both lubricating and crude oils, is, however, given by the formolite analysis. Further, if E is the Engler viscosity at a given temperature, F_3 the third formolite value, b the resin content, and C a constant, then $(E + b)/(F_3 + 2b - 3.5) = C$. The values of C at 100° for cylinder oils and "viscosines," and at 50° for most machine oils, were found to be 0.20, 0.22, and 0.29, respectively. The equation enables the viscosity of an oil to be calculated from its formolite value. W. T. K. BRAUNHOLTZ.

Motor spirit, with special reference to its use in internal-combustion engines. H. Y. V. JACKSON (J. Inst. Petrol. Tech., 1927, 13, 855—874).—A summary of contemporary information concerning chemical and physical properties of motor fuels, and the mechanism of combustion in the engine. R. H. GRIFFITH.

Carbon monoxide in two large garages. S. H. KATZ and H. W. FREVERT (Ind. Eng. Chem., 1928, 20, 31—36).—Continuous records of the carbon monoxide content of the air in two garages have been obtained over a period of four months, with an instrument previously described (cf. U.S.P. 1,578,666; B., 1926, 430). Each day is divided into four periods, for which maximum and average values for carbon monoxide in parts per ten thousand of air are shown. No injurious concentrations were found except for short times, or near engines running without load on a rich fuel-air mixture, but conditions might have been worse in cold weather when less efficient ventilation was possible. The behaviour of the recording instrument during long use is described. R. H. GRIFFITH.

Burkheiser ammonium sulphite-bisulphite process. TERRES and HEINSEN.—See VII.

PATENTS.

Manufacture of briquettes. L. M. JOHNSTON and J. L. FARRELL (U.S.P. 1,655,728, 10.1.28. Appl., 24.2.26).—Acid tars are used mixed with pulverised carbonaceous material, and compressed into briquettes which are then roasted to drive off the acid.

H. ROYAL-DAWSON.

Gas producer. C. W. LUMMIS, Assr. to MORGAN CONSTRUCTION Co. (U.S.P. 1,655,320, 3.1.28. Appl., 10.7.19).—A gas producer having an annular base for supporting the fuel bed is provided with a flange at the inner periphery of its base, whereby a depth of water is maintained thereon. By a mechanical device the ashes at the bottom are crowded inwardly and over the flange.

C. O. HARVEY.

Manufacture of gas from petroleum oil. E. M. CLARK, N. E. LOOMIS, and F. A. HOWARD, Assrs. to STANDARD DEVELOPMENT Co. (U.S.P. 1,651,115, 29.11.27. Appl., 27.5.20).—A heavy oil is cracked and the pitch formed is treated with steam to yield water-gas, which is carburetted with the light oil formed in the cracking and mixed with the gas similarly produced.

T. S. WHEELER.

Oxidation of hydrogen sulphide contained in industrial [illuminating] gases to sulphur. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 282,508, 27.9.26).—The gases containing hydrogen sulphide are brought into intimate contact with water or steam to give them a humidity of about 80%, and are then passed through active carbon or other highly adsorptive material which may be cooled and/or moistened to increase its humidity.

W. G. CAREY.

Operation of internal-combustion engines and apparatus therefor. G. B. ELLIS. From MOORE INVENTIONS CORP. (B.P. 281,739, 14.6.26).—In an internal-combustion engine a portion of exhaust gases and/or high-pressure gases from the working stroke are injected into the inlet pipe beyond the throttle, in quantities that vary with the throttle opening, so that the fuel mixture is converted into perfectly dry gas.

B. M. VENABLES.

Retort for the distillation of oil shales. L. T. FAIRHALL (U.S.P. 1,656,107, 10.1.28. Appl., 23.4.26).—A rotatable, horizontal, cylindrical retort has two centrally and axially disposed conduits, the one extending for practically the whole length of the retort and being closed at one end, the other end extending beyond the end of the retort. The portion of this conduit situated inside the retort is perforated. The other conduit supports the closed end of the first conduit and serves for the admission of steam, which enters the retort through a number of radially disposed pipes.

C. O. HARVEY.

Oil-cracking apparatus. F. C. VAN DE WATER and F. R. SUNDERMAN, Assrs. to PETROLEUM LABORATORIES INC. (U.S.P. 1,655,030, 3.1.28. Appl., 29.8.22).—An oil still is provided with a central flue for combustion gases and with a baffle which projects inwardly from the still body and upwardly in a position substantially midway between the walls of the flue and the still body.

C. O. HARVEY.

Distillation of petroleum oil. R. W. HANNA, Assr. to STANDARD OIL Co. OF CALIFORNIA (U.S.P. 1,655,603, 10.1.28. Appl., 31.3.23).—Lubricating oils are obtained from petroleum oil fractions which would ordinarily undergo decomposition below their b.p. (under partial vacuum) by vacuum distillation under reduced pressure, while adding lighter lubricating oil fractions. The distillation is carried out at a substantially constant temperature, below that at which decomposition of the heavy oil would occur, and the vapours are withdrawn at such a rate as to cause vaporisation of both the heavy and the light (added) lubricating oil fractions.

C. O. HARVEY.

Treatment of petroleum oil. A. D. DAVID, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,655,596, 10.1.28. Appl., 14.2.23).—A furnace-heated tubular still is connected with two vaporising chambers either of which may be isolated from the system. Means are provided for introducing a cooling medium into the transfer lines communicating with the isolated vaporising chamber.

C. O. HARVEY.

Treatment of [mineral] oils. A. F. L. BELL, Assr. to ASSOCIATED OIL Co. (U.S.P. 1,655,890, 10.1.28. Appl., 11.8.24).—Oil and a treating agent are delivered

to a settling tank provided with means for the prevention of agitation. The oil is withdrawn from the top of the tank, and the treating agent may be recirculated or passed to an evaporating vessel whence the evolved vapours and unevaporated residue are withdrawn.

C. O. HARVEY.

Refining of petroleum oils. P. McMICHAEL, ASSR. to HYDROCARBON REFINING PROCESS CO., INC. (U.S.P. 1,655,068—9, 3.1.28. Appl., [A] 23.10.23, [B] 14.2.24).—(A) The oil is heated and digested with an aqueous solution of a fixed alkali hydroxide (*e.g.*, with 10—33 pts. of an aqueous solution containing about 1 pt. of calcium hydroxide and 0.1 pt. of ammonium chloride per 100 pts. of oil for 1—4 hrs. at about 100°) and is subsequently treated with 70—83% sulphuric acid followed by treatment with more concentrated acid. (B) The oil is treated with a fixed alkali and ferrous hydrate in the presence of air, and subsequently with 1.5—15% of 70—83% sulphuric acid. Finally the oil is steam-distilled in the presence of a fixed alkali. C. O. HARVEY.

Refining of petroleum and oil-field emulsions. L. E. WINKLER and F. C. KOCH (U.S.P. 1,650,813, 29.11.27. Appl., 20.9.26).—A spray head is fitted below the liquid contained in a battery of tanks elevated progressively in series and connected together for the purpose of maintaining a predetermined level above the head. A gas supply pipe communicates with the spray head of the first tank, and each tank is connected, above the liquid level therein, with the spray head of the next succeeding tank. H. ROYAL-DAWSON.

Desulphurising and purifying petroleum oil. R. CROSS (U.S.P. 1,654,581, 3.1.28. Appl., 21.11.23).—The oil is purified and freed from sulphur by treatment with a sodium plumbite solution at temperatures above 93°; the solution and oil are made to flow in counter-current and the operation is carried out under sufficient pressure to prevent vaporisation of the liquid materials. C. O. HARVEY.

Refining of elaterite. R. E. HAUGHEY (U.S.P. 1,653,766, 27.12.27. Appl., 15.4.25).—The elaterite, contained in an open retort resting upon and connected (by means of a restricted opening) with an air-tight chamber, is ignited, the melted material flowing downwards through the opening into the air-tight chamber. C. O. HARVEY.

Deodorising and filtering of oils. V. C. BENJAMIN (U.S.P. 1,655,175, 3.1.28. Appl., 29.9.25).—Oil which has been heated at a high temperature with an adsorbent material is freed from the latter by passing it (along with entrained steam) through a foraminous medium contained in a closed filtering system. C. O. HARVEY.

Decolorisation of oils under pressure. P. W. PRUTZMAN, ASSR. to CONTACT FILTRATION CO. (U.S.P. 1,653,735, 27.12.27. Appl., 6.12.26).—The oil, admixed with an adsorbent, is heated to a temperature above its vaporisation point under a pressure greater than that which may result from frictional resistance to movement of the oil. C. O. HARVEY.

Purification and drying of oil separated from aqueous emulsions. G. B. ELLIS. FROM AMER. SHEET & TIN PLATE CO. (B.P. 282,321, 13.9.26).—Oil is recovered from oil-water emulsions, *e.g.*, the emulsion

discharged from the washing machine in a typical tin-plating process described, by breaking the emulsion in a suitably designed vessel by the addition of acid and introduction of live steam, separating the oil layer as far as possible, passing it through a screen to remove large particles of foreign matter, and thence through a heated coil or some other heating arrangement wherein the water is converted into vapour. The mixture of oil and water vapour is now discharged, *e.g.*, into a stack, the water vapour being carried upward in the air draught and the oil collected and centrifuged while still hot. S. S. WOOLF.

Agent for recovery of volatile solvents [petrol]. W. RUNGE, ASSR. to BREGREAT CORP. OF AMERICA (U.S.P. 1,651,155, 29.11.27. Appl., 11.2.24).—Low-temperature tar distillates free from acids and phenols are employed. T. S. WHEELER.

Coke oven. J. STEPHENSON (U.S.P. 1,656,841, 17.1.28. Appl., 4.12.25. Can., 20.12.24).—See B.P. 244,772; B., 1926, 940.

Production of hard, homogeneous fuel or similar objects from peat, peat moss, lignite, etc. B. JIROTKA, ASSR. to DR. O. SPRENGER PATENTWERWERTUNG JIROTKA M.B.H. (U.S.P. 1,656,859, 17.1.28. Appl., 29.5.26. Ger., 13.8.21).—See B.P. 276,471; B., 1927, 867.

Pulverising apparatus for solid fuels. G. S. LOY (U.S.P. 1,656,862, 17.1.28. Appl., 22.11.22. Fr., 6.12.21).—See B.P. 190,132; B., 1923, 917 A.

Liquid purification of fuel gases. F. W. SPERR, JUN., and D. L. JACOBSON, ASSRS. to KOPPERS CO. (U.S.P. 1,656,881, 17.1.28. Appl., 7.8.24).—See B.P. 238,172; B., 1925, 872.

Refining and cracking of hydrocarbons. R. K. COLLINS, ASSR. to COLLINS PROCESS INC. (U.S.P. 1,654,577—1,654,580, 3.1.28. Appl., [A] 18.5.22, [B] 23.5.22, [C] 24.3.26, [D] 28.7.26).—See B.P. 280,034 and 280,039; B., 1928, 80.

Coke conveying and quenching apparatus. SOUTH METROPOLITAN GAS CO., and C. H. SMITH (B.P. 282,861, 24.9.26).

Apparatus for separating solids (B.P. 281,479). Separation of liquids from materials (B.P. 281,390). Containers for corrosive liquids (B.P. 281,928).—See I. Lead tetraethyl (U.S.P. 1,652,812). Vanadium from petroleum soot (U.S.P. 1,651,967).—See VII.

III.—ORGANIC INTERMEDIATES.

Preparation of acetone. E. DONATH (Chem.-Ztg., 1927, 51, 924).—The author refers to his previous work (A., 1889, 230) in which he showed that acetone is formed by passing alcohol vapour over manganese dioxide (containing 90.7% MnO₂ and 2.7% BaO) heated at 540°. The bulk of the acetone is probably formed by direct oxidation, but part of the alcohol may be oxidised to acetic acid, the resulting acetates being then decomposed to acetone.

W. T. K. BRAUNHOLTZ.

Microchemical identification of lactic acid. J. GRÜSS (Woch. Brau., 1928, 45, 16—18).—A few drops

of the liquid to be tested are placed on a microscope slide and a small crystal of cobalt acetate is added. On drying, the slide is examined for characteristic reddish aggregates of cobalt lactate. If none is seen, the dry residue is quickly rinsed with a drop of distilled water which is evaporated on a clean slide, and the residue from this examined for cobalt lactate. For a further test, 3 c.c. of the liquid are distilled to half volume with 1 c.c. of strong sulphuric acid, and the distillate is collected. To a portion of the distillate a speck of sodium nitroprusside and a drop of piperidine are added, when a blue colour indicates acetaldehyde. Since this may arise from other substances than lactic acid, the remainder of the distillate is tested for the formic acid also produced, by neutralising with magnesia, boiling, and filtering. The filtrate after evaporation is tested on a slide with a speck of cerium nitrate. The characteristic rhombic dodecahedra of cerium formate may be recognised even if imperfectly formed, by the dark cross exhibited when examined between crossed Nicols. The preparation may be purified by quickly rinsing on to a fresh slide. The microscopic appearance of the crystals is figured, and details are given of results obtained in examining yeasts. F. E. DAY.

Analysis of mixtures of similar organic compounds. F. H. RHODES, F. T. GARDNER, and A. W. LEWIS (Ind. Eng. Chem., 1928, 20, 85—86).—The amount of a substance present in admixture with other isomeric, homologous, or unknown substances can be determined from the apparent average mol. wt. of the mixture in two different solvents, one of which is not a component of the mixture whilst the other is identical with the substance being determined. In the first case the true average mol. wt. is obtained, whereas in the second the amount of the particular substance present in the sample increases the amount of the solvent and a corresponding difference in the depression of the f.p. is obtained, from which the concentration of the particular component can be calculated. The method is limited to substances which have f.p. within the range of temperature covered by a Beckmann thermometer, and cannot be used when the compounds are ionised or polymerised or if they form compounds with the other components. Examples of the analysis of various synthetic mixtures of naphthalene, its nitro-derivatives, diphenyl, and a heavy coal-tar oil are described and satisfactory results are obtained.

E. H. SHARPLES.

Hydrocarbons and alcohols from water-gas. TROPSCH. **Action of sulphuric acid on olefines and alcohols.** ORMANDY and CRAVEN. **Phenols from petroleum.** TANAKA and KOBAYASHI.—See II.

PATENTS.

Manufacture of esters. H. F. BUC, Assr. to STANDARD DEVELOPMENT Co. (U.S.P. 1,651,666, 6.12.27. Appl., 22.12.22).—The reaction between the acid, e.g., acetic acid, and the alcohol, e.g., isobutyl alcohol, is performed in presence of a selective solvent for the ester, e.g., a hydrocarbon oil. T. S. WHEELER.

Manufacture of butyric aldehyde. CONSORTIUM F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 271,103, 13.5.27. Ger., 15.5.26).—Crotonaldehyde is hydrogenated in the

liquid phase in the presence of a nickel catalyst under a pressure of 10—30 atm., at temperatures below 100°, with thorough agitation. In the liquid phase the velocity of reduction of crotonaldehyde to butaldehyde is greater than that of the latter to butyl alcohol, and the reaction is discontinued before the appearance of an appreciable quantity of alcohol. E.g., 0.03 pt. of a nickel-kieselguhr catalyst (containing 15% Ni and prepared by reducing with hydrogen nickel carbonate or oxide precipitated on kieselguhr) and 1 pt. of crotonaldehyde are stirred at 85—90°, hydrogen being passed through at a pressure of 10—20 atm. After 2½ hrs. the liquid contains 74% of butaldehyde. B. FULLMAN.

Recovery of dehydrogenation products. I. G. FARBENIND. A.-G. (B.P. 262,086, 15.11.26. Ger., 27.11.25).—The hydrogen obtained in the dehydrogenation of the higher aliphatic alcohols (e.g., isobutyl alcohol, by treatment with zinc oxide) may be stripped of the volatile dehydrogenation products which it contains (even after separation of the latter as usual), by washing it with the alcohol to be dehydrogenated. The dehydrogenation products may be recovered by, e.g., heating the alcohol containing them. B. FULLMAN.

Extraction of guaiacol. O. MOSER, Assr. to VER. F. CHEM. IND. A.-G. (U.S.P. 1,651,617, 6.12.27. Appl., 28.12.26. Ger., 17.7.25).—Wood-tar oil is extracted with a limited amount of sodium hydroxide solution to remove substances of the guaiacol type only.

T. S. WHEELER.

Manufacture of aromatic oxamic acid halides. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 282,891, 30.9.26).—Aryloxamyl halides, e.g., $\text{NHAr}\cdot\text{CO}\cdot\text{COCl}$, are obtained by action of oxalyl halides on salts of aromatic amines (the free bases are useless for this purpose). Phenylloxamyl chloride, m.p. 82.5°, *o*-tolylloxamyl chloride, m.p. 89—90°, α -naphthylloxamyl chloride, m.p. 86°, and β -naphthylloxamyl chloride, m.p. 114—115°, are described.

C. HOLLINS.

Manufacture of anthracene derivatives and of benzanthrene. I. G. FARBENIND. A.-G., Asses. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (B.P. 260,000, 18.10.26. Ger., 17.10.25).—Anthraquinone derivatives and benzanthrene, preferably mixed with copper or zinc dust, are sublimed and the vapours are carried in a stream of hydrogen over a hydrogenating catalyst (e.g., a mixture of reduced copper and zinc) at 325—475° according to the catalyst and anthraquinone derivative used. 2-Aminoanthraquinone, sublimed with hydrogen over copper and zinc at 460—475°, gives 2-aminoanthracene, m.p. 235°. 1-Aminoanthracene, m.p. 120—130°, 2-methylaminoanthracene, m.p. 220—221°, 1-diethylaminoanthracene, m.p. 200° (from 1-diethylaminoanthraquinone, m.p. 118—120°), 2:6-dimethoxyanthracene, m.p. 255—256°, 2-methylanthracene, m.p. 199°, and benzanthrene, m.p. 82—84°, are similarly obtained.

C. HOLLINS.

Manufacture of isatin derivatives and of indigoid dyes therefrom. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 282,863, 25.9.26).—Isatin α -chlorides [2-chloro-3-ketoindolenines] react at 90° with aqueous sodium sulphite to give soluble products, which

may also be obtained (together with 50% of the corresponding indigo) by treating dehydroindigo bisulphite compounds (cf. Kalb, A., 1909, i, 967; B.P. 16,377 of 1909) with hot sodium carbonate or sulphite solution. 5-Chloroisatin α -chloride gives with hot sodium sulphite a yellow solution from which the new product may be salted out; it is stable to dilute acid, but hot acids convert it into dichloroindigo, and on reduction with hyposulphite it gives leuco-dichloroindigo. It gives indigoid dyes by condensation with α -naphthol and other reactive methylene-ketones. The new products contain sulphur and nitrogen in the atomic ratio 2:1.

C. HOLLINS.

Manufacture of oxidation products of acenaphthene. H. F. LEWIS, Assr. to NAT. ANILINE & CHEMICAL Co., INC. (U.S.P. 1,649,833, 22.11.27. Appl., 24.1.21).—Acenaphthene vapour mixed with air (9–12 vols.) is passed over manganese dioxide or an oxide of molybdenum or vanadium at 200–600° to yield mixtures containing according to conditions more or less of acenaphthylene, acenaphthenequinone, and naphthalic anhydride, which are fractionally condensed.

T. S. WHEELER.

Manufacture of *o*-anisidine and *o*-aminophenol ethers. J. TCHERNIAC (B.P. 282,907, 2.10.26).—*o*-Alkoxybenzamides are heated with a hypochlorite and caustic alkali solution. *o*-Methoxybenzamide gives an 80% yield of *o*-anisidine.

C. HOLLINS.

Preparation of mercaptobenzthiazoles. L. B. SEBRELL and J. TEPPEMA (B.P. 282,947, 9.11.26).—*o*-Chloronitrobenzene is treated with sodium sulphide, hydrogen sulphide, and carbon disulphide, to give 1-mercaptobenzthiazole. Alternatively, *oo'*-dinitrodiphenyl disulphide, obtained from *o*-chloronitrobenzene and sodium disulphide, is treated with hydrogen sulphide and carbon disulphide. The reaction is extended to the preparation of the 3-methyl and 3-phenyl derivatives of 1-mercaptobenzthiazole.

C. HOLLINS.

Compositions of matter suitable as emulsifying agents. I. G. FARBENIND. A.-G. (B.P. 258,551, 2.7.26. Ger., 18.9.25).—Emulsions of oils, fats, and other insoluble compounds in water are obtained, without employing an auxiliary solvent, by the help of organic sulphonic acids (*e.g.*, Turkey red oil, isopropyl-naphthalene-sulphonic acid, ligninsulphonic acid, etc.) or their salts in conjunction with a gelatinisable substance (glue, gelatin, gum arabic, or any other substance which swells in water and gels when the warm aqueous solution is cooled).

C. HOLLINS.

Preparation of emulsifying agents. I. G. FARBENIND. A.-G. (B.P. 268,387, 28.3.27. Ger., 27.3.26. Addn. to B.P. 258,551; preceding).—An alkylated cellulose is used in place of the gelatinisable substance.

C. HOLLINS.

Manufacture of ketones. L. LEFRANC (U.S.P. 1,656,488, 17.1.28. Appl., 16.5.24. Fr., 17.5.23).—See B.P. 216,120; B., 1925, 337.

Manufacture of useful products by means of Friedel and Crafts reaction [*o*-benzoylbenzoic acid]. H. G. STONE and B. H. JACOBSON, Assrs. to E. C. KLIPSTEIN & SONS Co. (U.S.P. 1,656,575, 17.1.28. Appl., 28.12.23).—See Can. P. 254,834; B., 1926, 869.

Splitting-off sulpho-groups from anthraquinone-sulphonic acid derivatives. R. E. SCHMIDT, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,650,158, 22.11.27. Appl., 3.8.26. Austr., 8.4.26).—See B.P. 250,968; B., 1927, 743.

Manufacture of a composition of matter [halogen-substituted oxindole-3-carboxylic acids]. W. SCHOELLER and K. SCHMIDT, Assrs. to CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (U.S.P. 1,656,239, 17.1.28. Appl., 4.3.26. Ger., 10.3.25).—See G.P. 443,099; B., 1927, 286.

Cellulose nitrate solutions (B.P. 282,172).—See V.

IV.—DYESTUFFS.

PATENTS.

Manufacture and use of new vat dyes. BRIT. DYESTUFFS CORP., LTD., and [A] S. THORNLEY, [B] A. SHEPHERDSON and S. THORNLEY (B.P. 282,852, 2.9.26. and 282,913, 8.10.26 [B]).—(A) Flavanthrone and (B) pyranthrone (and its derivatives) are treated with hydroxylamine in presence of sulphuric acid and ferrous sulphate to give (A) green and (B) brown vat dyes, which are improved by acylation. The acylated dyes become fast to chlorine after treatment with hypochlorite.

C. HOLLINS.

Manufacture of vat dyes. BRIT. DYESTUFFS CORP., LTD., W. D. ROGERS, H. EVANS, and W. F. A. ERMEN (B.P. 282,481, 13.9.26).—Pyranthrone is brominated in chlorosulphonic acid in presence of iodine. C. HOLLINS.

Manufacture of new azo dyes. I. G. FARBENIND. A.-G., Asses. of FARBENFABR. VORM. F. BAYER & Co. (B.P. 258,894, 24.9.26. Ger., 25.9.25).—Wool dyes fast to fulling, suitable also for printing on wool or silk, are obtained by coupling tetrazotised 2:2'-dichlorobenzidine or *m*-tolidine [2:2'-dimethylbenzidine] with 1 mol. of *m*-phenylenediamine-4:6-disulphonic acid and in alkaline solution with 1 mol. of 2-amino-8-naphthol-6-sulphonic acid ("γ-acid") or its *N*-substituted derivatives. With γ-acid a reddish-brown dye is produced, with *N*-aryl-γ-acids yellower shades. C. HOLLINS.

New azo dyes and their application. BRIT. DYESTUFFS CORP., LTD., and H. W. MOSS (B.P. 282,548, 7.12.26).—Direct black shades on cotton are obtained by means of developable trisazo dyes of the type Ar → N ← D → α-naphthylamine or its 6(7)-sulphonic acid, where D is a diamine of the benzidine series, N is a 1:8-aminonaphtholsulphonic acid, and Ar is a phenylenediamine. *E.g.*, 1:8-aminonaphthol-3:6-disulphonic acid is coupled acid with *p*-nitro-diazobenzene, then alkaline with tetrazotised benzidine or dianisidine, and the resulting diazodisazo compound is coupled acid with α-naphthylamine or its 6(or 7)-sulphonic acid; the nitro-group is reduced with sodium sulphide at any stage after the first coupling. The same result is obtained by using *p*-aminoacetanilide for the first coupling, with subsequent hydrolysis in place of reduction. The depth and fastness of the dyeings are improved by diazotisation and development on the fibre with β-naphthol, 2:4-tolylenediamine, etc. C. HOLLINS.

Manufacture of azo dyes. I. G. FARBENIND. A.-G. (F.P. 621,209, 9.9.26).—Azo dyes containing as end-

component a dihydroxynaphthalene or a sulphonic acid thereof are treated with an arylsulphonyl halide in presence of an acid binding agent; e.g., the *p*-toluenesulphonic ester of the dye *o*-anisidine \rightarrow 1 : 8-dihydroxynaphthalene-3 : 6-disulphonic acid gives on wool bluish-red shades fast to milling and light. C. HOLLINS.

Manufacture of mordant dyes. I. G. FARBENIND. A.-G. (B.P. 270,308, 27.4.27. Ger., 27.4.26).—A diazotised *o*-aminophenol or *o*-aminonaphthol is coupled with 2 : 6-dihydroxynaphthalene-3-carboxylic acid to give chrome olive-green to green-black wool dyes fast to washing, fulling, and potting, and suitable for single-bath chrome dyeing. Examples of suitable first components are 2-aminophenol-4-sulphonic acid, 4-chloro-2-aminophenol-6-sulphonic acid, and 4-nitro-2-aminophenol. C. HOLLINS.

Manufacture of anthraquinone derivatives. O. Y. IMRAY. From SOC. CHEM. IND. IN BASLE (B.P. 282,853, 21.9.26).—Aminoanthraquinones are alkylated or aralkylated by heating at 100° with an aldehyde and formic acid. From 4-nitro-1-aminoanthraquinone, formic acid, and formaldehyde (or trioxymethylene), 4-nitro-1-methylaminoanthraquinone is obtained; benzaldehyde gives a benzyl derivative. The nitro-group may be reduced or may be exchanged for other groups, such as arylamino-groups. Other examples are benzylation of 1 : 4-diaminoanthraquinone, ethylation of 1-amino-4-anilinoanthraquinone, methylation of 1 : 5(8)-diaminoanthraquinone, 1-amino-4-hydroxyanthraquinone, 1- and 2-aminoanthraquinones, 1-amino-4-methoxyanthraquinone, and diamino-1 : 5(8)-dihydroxyanthraquinone, and the butylation (?) of 1-amino-4-hydroxyanthraquinone with crotonaldehyde and formic acid. Alternatively, either the aldehyde or the formic acid may be condensed first with the aminoanthraquinone. The products dye acetate silk. C. HOLLINS.

Manufacture of dyes containing chromium. F. STRAUB, G. DE MONTMOLLIN, J. SPIELER, and C. VON PLANTA, Asss. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,656,844, 17.1.28. Appl., 1.6.25. Switz., 17.6.24).—See B.P. 235,862; B., 1926, 702.

Manufacture of hydroxyaryl-*p*-diamoanthrarufin compounds [8-diamino-2-*p*-hydroxyphenylanthrarufin-6-sulphonic acids]. R. E. SCHMIDT, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,652,584, 13.12.27. Appl., 13.4.26. Ger., 17.4.25).—See B.P. 274,211; B., 1927, 743.

Manufacture of coeruleinsulphonic acids. W. HERZBERG and G. HOPPE, Asss. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,656,483, 17.1.28. Appl., 20.4.26. Ger., 7.5.25).—See B.P. 251,968; B., 1927, 325.

Indigoid dyes (B.P. 282,863).—See III. **Carrying out exothermic reactions** (B.P. 282,559).—See X. **Diazo compounds** (B.P. 282,894).—See XXI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Measurement of the resistance of flax yarns to wear. G. F. NEW (J. Text. Inst., 1927, 18, 595—605 T).—The test samples, suspended vertically and carrying tension pieces, are submitted to rubbing through the oscillatory motion in a vertical plane of hardened steel

bars of cylindrical cross-section. The angle of contact between the yarn and the rubbing surface, and the tension in the yarn largely affect the number of oscillations withstood, and must be suitably chosen according to the material tested. Increase in fibre quality (as estimated in the trade), yarn twist, or amount of pre-spinning treatment given (within ordinary limits) produces a yarn with higher resistance to wear; boiling decreases this resistance whilst sizing greatly increases it. The results, in general, are concordant with the actual weaving behaviour of a series of yarns or sizes. B. P. RIDGE.

Determination of oil in textiles. H. R. HIRST (J. Text. Inst., 1927, 18, 606—607 T).—A sample of the material is treated with a known volume of dry acetone for 24 hrs. at the ordinary temperature, part of the liquid withdrawn, and the total oil determined by evaporation of the solvent and weighing the dry residue. Results obtained for a number of commercial oils on wool by this method agree closely with the theoretical values, whilst oil can also be determined in the presence of soda soaps. B. P. RIDGE.

Oxidation of cellulose in solution. I. L. KALB and F. VON FALKENHAUSEN (Ber., 1927, 60, 2514—2520).—The oxidation of cellulose by potassium permanganate in ammoniacal copper solution has been studied. With amounts of oxygen less than about 0.5 atom per $C_6H_{10}O_5$ group, the products show a continuous transition from the original precipitated cellulose to oxycellulose completely soluble in 10% sodium hydroxide. Complete solubility commences with 0.03 and 0.2 atom of oxygen in the cases of filter paper and cotton wool, respectively. Formation of products soluble in water is observed when more than 0.5 atom of oxygen is used for each $C_6H_{10}O_5$ group. The relationship between reducing power and acidity of the products derived by the use of relatively very small quantities of oxygen (whereby materials soluble in water are not formed) indicates that primary alcoholic groups of the cellulose are first converted into aldehydic and subsequently into carboxylic groups. The product obtained by the action of 2 atoms of oxygen per $C_6H_{10}O_5$ when subjected to dialysis yields 58% of residue, copper number 41.5, acid value about 240, and dissolves to a clear solution in water. From this material glycuronic acid is isolated as the cinchonine salt, m.p. 204°. The presence of this acid in the residues from the dialysis shows that it is probably contained therein in some form of chemical or adsorptive union from which it is liberated during the formation of the cinchonine salt. H. WREN.

Fluorescence of pine bark, pine wood, sulphite pulp and liquor. O. GERNGROSS (Z. angew. Chem., 1928, 41, 50—51; cf. Gerngross, Bán, and Sándor, B., 1926, 23, 839; Gerngross, *ibid.*, 1927, 137).—A reply to the criticisms of Hägglund and Johnson (B., 1927, 871). Pine wood on heating for a short time with dilute hydrochloric acid yields a solution with a weak reddish fluorescence in ultra-violet light, whilst cotton wool dipped in this solution acquires a strong violet fluorescence similar to that shown by the aqueous extract of pine wood obtained by boiling under pressure. These observations are not in accordance with the view of Hägglund and Johnson that the fluorescence of

sulphite pulp is due to the presence of lignosulphonic acid.

W. J. POWELL.

[Fluorescence of sulphite pulp]. E. HÄGGLUND and T. JOHNSON (Z. angew. Chem., 1928, 41, 51).—A brief reply to Gerngross (cf. preceding abstract).

W. J. POWELL.

PATENTS.

Protection of wool from damage by bacteria.

I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (B.P. [A] 256,273 and Addn. B.P. [B] 261,342, 3. and 27.8.26. Ger., 3.8.25 and 10.11.25).—Alkylated naphthalenesulphonic acids are used to protect wool from attack by bacteria, but to be effective they should be applied (A) before or after (but not during) alkaline treatment of the wool, or (B) before carbonisation of the wool by acids.

D. J. NORMAN.

Washing of clothes. H. VONTOBEL (B.P. 282,588, 19.5.27).—Clothes are steeped in a cold solution of washing soda, soap, borax, and sodium perborate, to which have been added enzymes (oxidases, peroxidases, oxygenases, catalase, tyrosinase, etc.) and catalysts such as salts of the heavy metals (*e.g.*, ferrous or copper sulphate etc.), or substances containing enzyme and catalyst. The enzymes cause the perborate to give up its oxygen. After 10–12 hrs. the clothes are rubbed with soap and warm water, and rinsed in hot and then in cold water.

B. FULLMAN.

Treatment of fabrics. S. M. CADWELL and O. H. SMITH, Assrs. to MORGAN & WRIGHT (U.S.P. 1,651,751, 6.12.27. Appl., 13.3.25).—Cotton fabric employed to wrap rubber during vulcanisation is rendered more resistant to the acid developed by impregnation with mono- and di-sodium hydrogen phosphates. T. S. WHEELER.

Wetting of fibrous materials. I. G. FARBENIND. A.-G., Assees. of A.-G. F. ANILIN-FABR. (B.P. 245,098, 15.12.25. Ger., 23.12.24).—1:4-Dioxan is a satisfactory wetting-out agent for use in scouring and dyeing textile materials (cf. B.P. 275,653; B., 1927, 905).

A. J. HALL.

Production of cellulose from cellulose-containing materials. A. CLASSEN (B.P. 279,147, 7.6.26 and 21.8.26).—Cellulose in a finely-divided form is obtained by treating cellulosic material, *e.g.*, wood meal, with concentrated hydrochloric acid, optionally in admixture with other strong mineral acids and preferably in conjunction with gaseous hydrogen chloride, at temperature below, at, or not substantially above 0°. Thus, 1 pt. of wood meal is moistened with an acid mixture containing 3.5 pts. of commercial concentrated hydrochloric acid and 1 pt. of concentrated sulphuric acid, and is then treated with gaseous hydrogen chloride in a rotary drum at 0° to –15°. When dissolution of the cellulose is complete, the ligneous matter is removed by filtration and the cellulose precipitated by the addition of suitable salts, either dry or in strong aqueous solution, *e.g.*, concentrated potassium chloride solution. The cellulose is collected, washed, and freed from the last traces of acid in a current of air or inert gas, whilst the residual acid liquor is treated with gaseous hydrogen chloride to precipitate the potassium chloride and regenerate

hydrochloric acid of sufficiently high concentration to be suitable for the treatment of a further quantity of raw material.

D. J. NORMAN.

Opening-up of materials containing cellulose.

I. G. FARBENIND. A.-G. (B.P. [A] 274,892 and Addn. B.P. [B] 276,025, 21.7. and 15.8.27. Ger., 21.7. and 13.8.26).—(A) Cellulose of high purity is obtained in good yield by exposing comminuted cellulosic material, *e.g.*, deal shavings, to the action of a current of air that has been heated at 30° and passed through nitric acid of 60% strength. (B) Alternatively, the material, *e.g.*, steamed firwood, is impregnated with warm or cold dilute (10%) nitric acid, freed from excess of acid by draining, and exposed to the action of a current of air at about 50° until a test sample is completely disintegrated when boiled with dilute sodium carbonate solution. In both cases the pulp is finally boiled with an alkaline solution.

D. J. NORMAN.

Manufacture of artificial silk and apparatus therefor. COURTAULDS, LTD., F. T. WOOD, and E. G. TURNEY (B.P. 281,058, 11.10.26).—In the production of artificial filaments from cellulose esters or ethers by the dry-spinning process, uniformity of the product is ensured by providing the spinning cells with heating elements of substantially identical construction communicating in parallel with supply and discharge mains of such capacity that the flow of water is sufficiently plentiful and rapid to limit the temperature drop through the heating elements to, *e.g.*, 0.1–0.2°. Further, the spinning cells are all in communication with a common aspirating system (sufficiently large to maintain a constant rate of flow of air through the cells) through interchangeable, but not individually adjustable, outlets of the same size.

D. J. NORMAN.

Manufacture of artificial silk, bands, ribbons, etc.

W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 280,628, 16.8.26).—Cuprammonium hydroxide solutions of wood pulp, straw, or grasses give, in contradistinction to cuprammonium hydroxide solutions of cotton cellulose, products of good lustre and strength when the temperature of the spinning bath is below 30° or even below 20°, but preferably between 20° and 30°.

D. J. NORMAN.

Treatment [weighting] of artificial silks.

R. CLAVEL (B.P. 277,602, 17.11.26. Ger., 17.9.26).—The process of B.P. 266,640 (B., 1927, 295) is applied to artificial silks of all types. *Example*: Cellulose acetate fabric is passed through a bath containing 5 litres of a 10% aqueous solution of albumin and 50 g. of ammonium carbonate, and, after squeezing, is transferred to a second bath consisting of a 4% solution of tin chloride containing 10% of phosphoric acid (the latter to prevent precipitation of the tin). The dried fabric shows a greatly increased affinity for sulphonated direct dyes.

D. J. NORMAN.

Manufacture of preparations of alkylcellulose and of artificial material and articles therefrom.

I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (B.P. 252,176, 12.5.26. Ger., 15.5.25).—Water-soluble cellulose ethers can be satisfactorily used in the preparation of cellulose ether compositions which neither swell nor dissolve to any

substantial extent in water if latex or a suitable quantity, *e.g.*, 2%, of a water-insoluble cellulose ether is colloidal dispersed in their aqueous solutions.

D. J. NORMAN.

Manufacture of nitrocellulose (or cellulose acetate) solutions and plastics. I. G. FARBENIND. A.-G. (B.P. [A] 251,303, and Addn. B.P. [B] 278,735 and [C] 279,771, 26.4.26. Ger., [A, B] 24.4.25).—Esters of organic acids with monoalkyl ethers of ethylene, propylene, and butylene glycols are good solvents for (A) nitrocellulose and (B) cellulose acetate and for many other compounds, including many natural and artificial resins, drying and non-drying oils, colouring agents, etc., which may with advantage be incorporated with cellulose ester lacquers and plastics. (C) The formate, acetate, and phthalate of ethylene glycol monomethyl ether are particularly suitable solvents or plasticisers (according to their b.p.) for cellulose acetate.

D. J. NORMAN.

Manufacture of cellulose nitrate solutions and plastics. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 282,172, 10.6.26).—As solvents for cellulose nitrate (alone or together with resins, colouring matters, oils, etc. of use in cellulose nitrate lacquers and plastics), there are used the oily products obtained by the catalytic hydrogenation of carbon oxides (*cf.* B.P. 227,147, 229,714, 237,030, and 238,390; B., 1925, 189, 338, 784), or fractions from these products, or the products (superior for this purpose) of their acylation, hydrogenation, or condensation. These may be mixed with one or more other solvents, *e.g.*, aliphatic alcohols or their esters, aliphatic or aromatic hydrocarbons, hydroaromatic substances, etc.

B. FULLMAN.

Viscose product. S. A. NEIDICH (U.S.P. 1,651,404, 6.12.27. Appl., 1.5.26).—Coagulated viscose is dehydrated by treatment with warm alcohol vapour to give a product with increased capacity for elongation.

T. S. WHEELER.

Recovery of sodium compounds from waste sulphite[*-*cellulose] liquors. C. H. MILLIGAN (U.S.P. 1,652,725, 13.12.27. Appl., 30.10.26).—The process of U.S.P. 1,545,522 (B., 1925, 707) is modified in that an organic acid, *e.g.*, stearic acid, is used in place of carbon dioxide to decompose sodium sulphide.

T. S. WHEELER.

Production of wood pulp. N. V. HANDELMAATSCHAPPIJ "FIBRA" (B.P. 267,107, 21.2.27. Ger., 5.3.26).—The preparation of mechanical wood pulp, approximating in quality to hot-ground pulp, from logs or wood waste is described. The raw material with its natural content of moisture is suitably comminuted, freed from wood flour, sand, and other waste material, and is then dry-refined, after treatment, if necessary, with sufficient water to replace any moisture lost during the chipping operation. Provision is made for the frictional generation of heat in the dry refiner in order to promote separation of the fibre bundles by the formation of vapour therein. After dry-refining, the pulp is mixed with a suitable quantity of water and passed to a wet refiner, this operation also being conducted under conditions which cause the generation of heat by friction. The pulp is finally screened and formed into sheets. Suitable apparatus is described.

D. J. NORMAN.

Manufacture of pulp for paper production. D. R. NANJI (B.P. 280,629, 17.8.26).—Good yields of easy-bleaching pulp are obtained with a cooking time of 0.5–1 hr. at the working temperature by using a cooking liquor containing up to 4% of caustic soda at pressures approximating to 10 atm. or at temperatures corresponding to these pressures. When considerable quantities of pectins and sugars are present, the material may conveniently be pretreated with depectinising agents such as a dilute (0.5%) solution of ammonium sulphate or other neutral alkali or ammonium salt. Bulky materials such as grasses may be subjected to a preliminary acid hydrolysis with, *e.g.*, 0.5% sulphuric acid for 0.5–1 hr. at 4–6 atm., to remove non-cellulosic material. The application of this process to the treatment of bagasse from sugar cane is described.

D. J. NORMAN.

Manufacture of pulp and paper. L. BRADLEY and E. P. MCKEEFFE (U.S.P. 1,651,665, 6.12.27. Appl., 8.2.22).—A cooking liquor for the digestion of wood under pressure contains caustic soda and sodium sulphite in such proportions that the sulphite content does not exceed the caustic soda content.

D. J. NORMAN.

Preparation of stuff [size precipitant] for paper making. E. MAHLER and H. A. ROTHCHILD, Assrs. to KIMBERLY-CLARK Co. (U.S.P. 1,650,022, 22.11.27. Appl., 21.9.23).—China clay is heated with 50% sulphuric acid at 170° for 1½ hrs. to form basic aluminium sulphate solution containing suspended silica, which is used as formed to precipitate a size on the stock.

T. S. WHEELER.

Degumming of flax straw. L. N. GILLIS (B.P. 279,302, 15.2.27).—Flax straw, previously dried at a temperature not exceeding 49°, is crushed, freed from shive as far as possible, and treated, either in hank form or supported on, *e.g.*, reticulated copper trays (to preserve parallelism of the fibres), for 10–20 min. with a boiling degumming liquor made by dissolving 1.75 lb. of commercial caustic soda (90%) and 3.5 oz. of potassium dichromate in 12 gals. of water. The material is then successively washed with hot (preferably running) water, immersed in soap solution, again washed with water, and finally treated for about ½ min. in a neutralising bath containing a small quantity of sulphuric acid.

D. J. NORMAN.

Bleaching of paper [sulphate] pulp. O. KRESS, Assr. to AMER. LAKES PAPER Co. (U.S.P. 1,651,530, 6.12.27. Appl., 24.12.25).—The pulp is treated in succession with bleaching powder, sodium hydrogen sulphite, and again with bleaching powder.

T. S. WHEELER.

Manufacture of cellulose acetate. L. A. LEVY, Assr. to APEX (BRITISH) ARTIFICIAL SILK, LTD. (U.S.P. 1,652,024, 6.12.27. Appl. 2.10.26. U.K., 8.10.25).—See B.P. 240,624; B., 1926, 10.

Preparation of coconut fibres for spinning. H. WESTPHALEN (B.P. 271,900, 27.5.27. Ger., 29.5.26).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Bleaching by means of oxygen [peroxides]. G. ADOLPH and A. PIETZSCH (B.P. 268,325, 14.3.27).

Ger., 29.3.26).—The natural colouring substances present in hair and feathers are bleached by immersion in cold solutions containing hydrogen peroxide and persulphates or benzoyl peroxide, alcohol being added for the purpose of controlling the rate of bleaching; the combination of bleaching agents is more effective than hydrogen peroxide alone. [Stat. Ref. to B.P. 187,575, 180,325, and 9247 of 1913.] A. J. HALL.

Production of combined shades from sulphur dyes and ice-colours on vegetable fibre. I. G. FARBE-ENIND. A.-G. (B.P. 266,387, 22.2.27. Ger., 22.2.26).—Cotton is padded with a hydroxynaphthoic arylamide or other coupling component for ice-colours in a bath containing also a sulphide dye dissolved as usual in sodium sulphide solution. After dyeing at 25–50° the ice-colour is developed with a diazo compound, and the dyeing is after-treated in the usual manner. A dye preparation consisting of a coupling component, a sulphide dye, and suitable additions for dissolving these may be made up. The process is useful for shading sulphide dyes towards the red. C. HOLLINS.

Dyeing, printing, or stencilling of materials made with or containing cellulose acetate. BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLPIN, and W. B. MILLER (B.P. 283,081, 30.9.26).—Acetate silk is dyed, printed, etc. by any of the usual methods with nitro-derivatives of carbazoles. *E.g.*, nitrocarbazole, m.p. 164°, applied with the aid of sodium sulphuric-oleate, gives a greenish-yellow shade. C. HOLLINS.

Vat dyes (B.P. 282,852 and 282,913). **Azo dyes** (B.P. 282,548).—See IV.

Printing of leather (B.P. 256,195).—See XV.

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

Technical production of sodium fluoride. A. E. J. MÜLLER (Chem.-Ztg., 1928, 52, 5–6).—Sodium carbonate is mixed with sodium fluoride mother-liquor from a previous operation and hydrofluoric acid is added with constant stirring until effervescence ceases, the temperature rising to 60–70°. The product has an acid reaction owing to the precipitation of the difficultly soluble acid sodium fluoride and also of sodium silicofluoride derived from the silica present in the fluorspar used for the preparation of the hydrofluoric acid. In order to decompose these compounds more sodium carbonate is added, until a portion of the mixture on removal gives a strong red colour with phenolphthalein, and the mixture is stirred and heated at 80–90° by passing in steam. The mother-liquor is removed from the sodium fluoride sludge as far as possible by a filter press, and the press cake of sodium fluoride containing about 50% of water is dried slowly. The final product is microcrystalline and contains 95–98% NaF.

F. R. ENNOS.

Determination of sodium borate. M. FRANÇOIS and (Mlle.) L. SEGUIN (J. Pharm. Chim., 1927, [viii], 6, 244–248).—1.91 g. of the finely-powdered borax are heated with 25 c.c. of water and 50 c.c. of glycerin until just dissolved. After cooling, 2 drops of phenolphthalein solution are added and the liquid is titrated with *N*-sodium hydroxide solution. The end-point is

sharp and, if the borax is pure, exactly 10 c.c. of *N*-sodium hydroxide solution are required. It is shown that the action of the glycerin is to transform the tetraborate into 2 mols. of sodium metaborate and 2 mols. of metaboric acid, the latter requiring 2 mols. of sodium hydroxide for neutralisation. E. H. SHARPLES.

Burkheiser ammonium sulphite-bisulphite process. II. E. TERRES and A. HEINSEN (Gas- u. Wasser-fach, 1927, 70, 1157–1161, 1193–1197, 1217–1220; cf. Terres and Hahn, B., 1927, 675).—Solubility curves for the salt pairs ammonium sulphate and sulphite and ammonium sulphate and bisulphite are plotted for temperatures between 0° and 60°. Similar curves are also drawn showing the conditions prevailing when (1) saturated solutions of ammonium sulphite in the absence of the solid phase and of a mixture of sulphate and sulphite in the presence of the solid phase are oxidised, (2) sulphur dioxide is passed into saturated solutions of a mixture of ammonium sulphate and sulphite, (3) solutions containing ammonium sulphate and bisulphite are neutralised with ammonia. In the oxidation of saturated solutions of ammonium sulphite in the absence of the solid phase, unsaturated solutions of sulphate and sulphite are formed. In the presence of an excess of solid sulphite a solid phase consisting of a mixture of sulphite and sulphate is first formed, and eventually the solid phase comprises pure sulphate. When solutions containing ammonium sulphate and sulphite are acidified, a separation of sulphate occurs at certain concentrations; in all other cases the solutions are unsaturated both as to sulphate and sulphite. Neutralisation of solutions containing sulphate and bisulphite leads first to separation of sulphite, which is later accompanied by sulphate. In practice the gases are never free from oxygen, and a mixture of ammonium sulphate and sulphite ("Burkheiser salt") in varying proportions is always obtained in the Burkheiser process.

W. T. K. BRAUNHOLTZ.

Occurrence of indium in commercial iron sulphide and its extraction. J. G. F. DRUCE (Z. angew. Chem., 1928, 41, 79).—A residue containing indium was found after continued treatment of commercial iron sulphide with sulphuric acid; indium oxide was eventually separated. The original proportion was about 6 pts. in 100,000. S. I. LEVY.

Elimination of antimony in the refining of arsenic trioxide. C. L. READ (Ind. Eng. Chem., 1928, 20, 97–100).—The commercial preparation of white arsenic (99% As₂O₃) from arsenical flue dusts in two sublimations is not possible if much antimony is present. An investigation was carried out on the separation of the two metallic oxides from the vapours produced by washing "black dust" containing 68% As and 6% Sb. It was found that if the dust was sublimed in a tube furnace with temperature control and in a current of air a vitreous deposit containing 25–30% Sb was first formed, and later a crystalline deposit of arsenious oxide containing only 0.7% Sb. The temperature at which the vitreous deposit was best formed was 350°, and a large surface for its deposition was necessary. The current of air should be as slow as possible. The furnace temperature is immaterial. C. IRWIN.

Extraction of radium and mesothorium from radioactive chlorides in the cold. I. BASCHLOFF (Z. angew. Chem., 1928, 41, 57—59).—A description of a technical method, already patented, for the separation of radium or mesothorium chloride from barium chloride without evaporation. The mixed chlorides are dissolved in water, and a quantity of strong calcium chloride solution is added sufficient to precipitate at most one third of the barium chloride present, which then contains about twice as much active chloride as the original mixture. By repeated fractional precipitation a product rich in radium or mesothorium is obtained, together with a solution of barium and calcium chlorides, which are recovered by evaporation and crystallisation. The process is suitable for working up chloride mixtures containing as little as 0.1 pt. per million of radioactive chloride. Aluminium and ferric chlorides may also be used as precipitating agents, but as they are susceptible to hydrolysis calcium chloride is preferred.

W. J. POWELL.

Working with compressed chlorine gas in practice. R. FREUND (Chem.-Ztg., 1928, 52, 33—34).—A review of present-day practice. F. R. ENNOS.

Alteration of gas samples when kept and precautions against it. O. HACKL (Chem.-Ztg., 1929, 57, 993—994).—Samples of hydrogen and carbon dioxide, containing less than 1% of impurities, were kept in glass bottles carefully closed with rubber stoppers. After 7 weeks they were found to contain 15—20% O, together with a considerable quantity of nitrogen. In a second test, in which the rubber stoppers were dipped in paraffin after insertion, the composition of the gas was unchanged after 1 month's keeping.

W. T. K. BRAUNHOLTZ.

Production of carbon. WANGENHEIM, also FISCHER and DILTHEY.—See II. Milori blue. MÜLLER-MAGDEBURG.—See XIII. Barium compounds from starch. STERN.—See XVII. Sodium salts as germicides. LEVINE and others.—See XXIII.

PATENTS.

Apparatus for concentration of acids. O. MANTUS (U.S.P. 1,655,019, 3.1.28. Appl., 15.8.25).—The concentrator has a closed circuitous passage through which acid flows, and is maintained at a predetermined level so that heating elements spaced apart longitudinally to the passage are totally submerged in the acid, which is heated to progressively higher temperatures in the direction of flow, vapours evolved from the acid being drawn off through a vapour outlet. W. G. CAREY.

Apparatus for the purification of impure solutions of caustic soda or the like on osmotic principles. L. CERINI (B.P. 272,211, 31.5.27. Italy, 1.6.26. Addn. to B.P. 265,126; B., 1927, 329).—The dialysing membranes are operated without forced circulation either as independent units or as units connected in parallel, so that instead of forcing counter-currents in the horizontal direction they will be established automatically in the solutions as a function of the various densities produced by the osmotic exchange.

W. G. CAREY.

Formation of sodium tungstate. W. B. STODDARD and I. HOCHSTADTER (U.S.P. 1,652,646, 13.12.27. Appl.,

30.3.22).—A mixture of a tungsten ore, charcoal, and sodium nitrate reacts autogenously on ignition to give a product containing sodium tungstate.

T. S. WHEELER.

Hypochlorite composition. M. C. TAYLOR, Assr. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,650,054, 22.11.27. Appl., 14.8.26).—The composition described in U.S.P. 1,481,003 (B., 1924, 334) is modified in that sodium fluoride is used in place of sodium carbonate.

T. S. WHEELER.

Preparation of [pure] calcium cyanide. R. W. POINDEXTER, JUN., Assr. to CALIFORNIA CYANIDE CO., INC. (U.S.P. 1,652,874 and 1,652,909, 13.12.27. Appl., [A] 5.2.27, [B] 13.4.26).—The compound of calcium cyanide and ammonia described in U.S.P. 1,596,120 (B., 1926, 946) is heated under reduced pressure at 325° until free from ammonia.

T. S. WHEELER.

Purification of the reagents used in preparing per-salts and other per-compounds. HENKEL & CIE., G.M.B.H. (B.P. 282,302, 12.7.27. Ger., 24.12.26).—A solution of the chemicals is stirred in the cold, or is boiled, with powdered silica gel, the clear decanted solution then being allowed to crystallise. The silica gel after absorbing impurities deleterious to the manufacture is regenerated by washing, treatment with acid, and heating.

W. G. CAREY.

Manufacture of caesium compounds of pure organic colouring matters. R. DELAPLACE (F.P. 621,420, 3.7.26).—A solution of caesium hydroxide is added to a solution of an acid dye, giving, especially in the case of eosin and erythrosin, solutions which absorb light of determined wave-lengths and are therefore useful in histology.

C. HOLLINS.

Recovery of lead or valuable lead compounds from lead-sulphur compounds. A. L. MOND. From NORDDEUTS. CHEM. FABR. IN HARBURG (B.P. 282,306, 8.8.27).—The lead compound is converted into lead sulphate and is then treated with concentrated sodium chloride solution at 4—5 atm. pressure at 140—150°. After settling, the liquid is passed through a filter-press and pure lead chloride crystallises therefrom.

W. G. CAREY.

Manufacture of lead tetraethyl. W. S. CALCOTT and F. L. ENGLISH, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,652,812, 13.12.27. Appl., 4.11.25).—Sodium-lead alloy is agitated at 60° in an autoclave which is connected by a vapour line with a reservoir of liquid ethyl chloride maintained at 40°, so that the pressure of ethyl chloride in the autoclave is constant.

T. S. WHEELER.

Dissolution of titaniferous materials in acids. TITAN Co. A./S. (B.P. 275,578, 2.7.27. Norw., 3.8.26).—During the dissolution of titaniferous materials, e.g., ilmenite, hydrolytic dissociation and consequent precipitation of titania is inhibited by gradually diluting the reaction mass with an aqueous solution of a salt or acid, and at the same time reducing the temperature, e.g., from 170° to 130°. By this method solutions containing 100—150 g. of TiO₂ per litre may be obtained.

C. A. KING.

Manufacture of a vanadium compound [from products of combustion of petroleum]. A. C.

REED, Assr. to T. COTTER and E. L. LASIER (U.S.P. 1,651,967, 6.12.27. Appl., 20.2.24).—Petroleum soot containing vanadium is briquetted with sodium silicate solution and fused with sodium hydrogen sulphate to yield soluble vanadium compounds, which are extracted with water.
T. S. WHEELER.

Recovery of sulphur from ammonium polysulphide. P. KOPPE, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,656,563, 17.1.28. Appl., 7.4.27. Ger., 16.4.26).—See B.P. 269,546; B., 1928, 91.

Separation of chlorides of aluminium and potassium present in mixed solutions obtained in the treatment of leucite. G. A. BLANC (U.S.P. 1,656,769, 17.1.28. Appl., 19.10.21. Italy, 7.3.21).—See B.P. 176,770; B., 1922, 812 A.

Evaporating pans (B.P. 277,639).—See I. Oxidation of hydrogen sulphide (B.P. 282,508). **Cyanides (U.S.P. 1,651,114).**—See II. Sulphite-cellulose liquors (U.S.P. 1,652,725).—See V.

VIII.—GLASS; CERAMICS.

PATENTS.

Heat-resistant compounds. BRIT. THOMSON-HOUSTON Co., LTD., Asses. of W. R. STEELE (B.P. 271,503, 20.5.27. U.S., 20.5.26).—Material to resist intermittent arcing etc. is made by mixing 20–40% (30%) of asbestos, 10–75% (65%) of zirconia, and 5–60% (5%) of lime; the mass is moistened, pressed into shape, and hardened by treatment with steam at 120–150 lb. pressure for 6–12 hrs. W. G. CAREY.

Manufacture of [refractory coatings for] silicon carbide articles. R. H. MARTIN, Assr. to NORTON Co. (U.S.P. 1,653,918, 27.12.27. Appl., 18.4.25).—A protective coating impervious to kiln gases under normal conditions is formed on a refractory article containing silicon carbide by coating it with a composition of magnesia and ferric oxide and firing at a suitable temperature.
W. G. CAREY.

Annealing glass sheets and plates produced by an intermittent rolling operation. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST. GOBAIN, CHAUNY, & CIREY (B.P. 269,569, 13.4.27. Fr., 15.4.26).

IX.—BUILDING MATERIALS.

Aluminium and [its use in] cement. J. MEYER (Chem.-Ztg., 1928, 52, 4–5).—Contrary to the conclusions of Platzmann (B., 1927, 908), the author's experiments on the use of aluminium for producing porosity in the manufacture of porous concrete indicate that it is quite unsuitable. Thus, it is too expensive, and the degree of porosity obtained with any one set of materials is not constant, but is influenced by such factors as the completeness of mixing of the aluminium with the cement, and the effect of small amounts of colloidal materials in protecting the aluminium from the action of the lime solution. The crushing strength also of such porous cements containing the necessary amount of sand is much too low for them to be of technical use.
F. R. ENNOS.

Diffusion of water-soluble substances in impregnated wood. R. NOWOTNY (Z. angew. Chem., 1928,

41, 46–49; cf. B., 1926, 408).—On impregnating wood with sodium fluoride and sodium dinitrophenoxide solution, diffusion is more rapid along the outer portion of the trunk than in a direction perpendicular to the side (radial diffusion). The diffusion is followed by cutting a portion of the tree 10 months after impregnation into thin layers and examining each layer colorimetrically for the presence of either of the two substances. Fir is more suitable than Scotch pine for such experiments, since the latter is too permeable, and the zones of diffusion from the individual centres interfere with one another. Radial diffusion is slower in the layers furthest from the surface, since there is less solution available and the wood is dryer. The diffusion of both substances is very slow around the trunk; sodium fluoride, for instance, travels 2.2 cm. round the circumference in the time required for it to travel over 16 cm. along the trunk, whilst the rate of diffusion around the inner year-rings is slower than around the exterior rings. Impregnations carried out on a pole partially buried in the earth showed that rates of diffusion from the centres above and below the ground level differed considerably. Below ground, the rate in an upward direction was twice that in a downward direction; above ground the downward rate was three times the upward rate, whilst diffusion was more rapid below than above ground, owing to the higher moisture content of the wood. For preserving wood by impregnation, a network of centres of diffusion are created (by boring holes and introducing the solution), and the centres are so arranged that overlapping of the diffusion zones will take place within a short period of time.
W. J. POWELL.

Blood-albumin and its use as an adhesive for veneer and plywood. H. STADLINGER (Chem.-Ztg., 1928, 52, 8–9, 35–36).—Two commercial varieties of albumin are obtained from blood-serum albumin which is prepared from blood serum after straining off the solid material, and black albumin obtained by extracting the solid material with water. In order that the material may retain its solubility it must be carefully dried below 60° either by a hot current of air or *in vacuo*. Black albumin, which is strongly coloured by hæmoglobin, is the variety used in the woodwork industry since it is cheaper and its colour is not objectionable for this purpose. Blood-albumin is soluble in cold or lukewarm water (85–95% in commercial varieties), its solution sets by coagulation when heated at 70° or over, and the setting power is increased by the addition of electrolytes such as milk of lime or ammonia. Joints made with blood-albumin are waterproof, their strength being diminished by only 25% after soaking for several days in cold water or for several hours in boiling water. In practice, 6 pts. of blood-serum are dissolved in 11 pts. of water at 27°, 0.25 pt. of ammonia solution (*d* 0.90) and 0.13 pt. of slaked lime are added, and the solution is applied to the article, which is then placed in a press and maintained at 80–90° for a sufficient period.
F. R. ENNOS.

PATENTS.

Treating, impregnating, seasoning, and stabilising wood. G. E. RICE, Assr. to CONSERVATION CORP. OF AMERICA (U.S.P. 1,650,738, 29.11.27. Appl., 30.8.24).

—An impregnating solution containing sucrose, maltose, and a toxic dye, *e.g.*, Crystal Violet, is employed.

T. S. WHEELER.

Impregnation of wood. D. B. BRADNER, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,652,811, 13.12.27. Appl., 19.11.25).—Wood is impregnated with a solution of arsenic pentasulphide in molten sulphur.

T. S. WHEELER.

Preservation of wood. E. B. FULKS, Assr. to AMER. CREOSOTING Co. (U.S.P. 1,652,109, 6.12.27. Appl., 9.1.26).—The wood is saturated with zinc chloride solution prior to impregnation with creosote.

T. S. WHEELER.

Fibrous paint [for coating walls]. G. E. HEYL (U.S.P. 1,656,198, 17.1.28. Appl., 29.6.26. U.K., 17.3.26).—See B.P. 259,826; B., 1926, 1016.

Wood-preserving composition. K. H. WOLMAN (U.S.P. 1,656,804, 17.1.28. Appl., 9.8.24. Austr., 23.5.24).—See B.P. 229,179; B., 1925, 284.

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

Energy losses in arc furnaces for steel. S. KRIZ (Arch. Eisenhüttenw., 1927, 1, 413—419; Stahl u. Eisen, 1928, 48, 71—72).—The losses of electrical energy during the melting of the charge in an arc furnace making steel are distributed as follows: transformer 3%, leads 6%, cooling water in electrode holders 4%, radiation and conduction from the walls of the furnace 15%, radiation from openings in the furnace 7%, and loss of heat in escaping gases 4%; thus 61% of the energy input is utilised in melting the charge. The corresponding loss figures for the refining of the molten charge are 6%, 3%, 7%, 29%, 11%, 7%; in this operation, therefore, only 37% of the energy input is usefully employed.

A. R. POWELL.

Rapid determination of sulphur in pig iron and steel. J. CIOCHINA (Z. anal. Chem., 1927, 72, 301—303).—A sample of the iron (1 g.) or steel (2 g.) is heated in a current of dry hydrogen at a temperature of 1240—1320° in an electric furnace. The sulphur is quantitatively converted into hydrogen sulphide, which is absorbed in a solution containing sodium, cadmium, and zinc acetates, and determined by titration with iodine and thiosulphate. The hydrogen should be washed by sodium carbonate and lead acetate solutions, and carbon dioxide should be led through the furnace at the beginning and at the end of each determination. The results obtained are usually higher than those given by dissolving the metal in dilute acid and determining the hydrogen sulphide formed, whilst the time taken for each determination is considerably less.

F. S. HAWKINS.

Alloys resistant to hydrochloric acid. B. WAESER (Chem. Fabr., 1928, 17—18).—A review of recent work, together with a table showing the composition and resistance to attack by hydrochloric acid solution of various strengths of 39 American acid-resisting alloys, 3 German special steels, and 2 ferrosilicon alloys.

A. R. POWELL.

Utilisation and behaviour of zinciferous [iron] ores, especially Meggen burnt pyrites, in the blast furnace. M. PASCHKE (Arch. Eisenhüttenw., 1927, 1,

387—402; Stahl u. Eisen, 1928, 48, 102).—Burnt pyrites containing zinc and sulphur may be smelted directly in iron blast furnaces provided that it is converted into porous briquettes, *e.g.*, by sintering in rotating kilns or in Dwight-Lloyd furnaces followed by grinding and briquetting. The sinter from either furnace should not be charged directly to the blast furnace as it requires a high temperature for reduction and entails a high coke consumption as well as giving a low-grade zinc fume. The addition of sodium chloride to the briquettes results in a higher recovery of zinc (87%) and a better quality of iron. The sintering operation may be avoided by adding the burnt pyrites directly to liquid ferromanganese slag together with coke dust, and blowing the mixture with hot air, whereby both sulphur and zinc are volatilised and a synthetic iron ore suitable for reduction in the blast furnace is obtained.

A. R. POWELL.

Extraction of zinc and other metals from ores. H. E. COLEY (Bull. Inst. Min. Met., 1928, [280], 11 pp.).—The theoretical view taken is that carbon in a "volatile" or nascent condition is a more active reducing agent than carbon monoxide, which reacts only at comparatively high temperatures. The active form of carbon is formed at the point of contact with heated ore by dissociation of hydrocarbons which have been kept cool and undissociated up to this point. In treating zinc ores, the ore, ground to $\frac{1}{4}$ -mesh, is preheated to about 500°, fed through a seal to a rotary furnace maintained at this temperature, and, by means of baffles, given contact with dissociation products of hydrocarbons injected into the tube furnace. The time for reduction is about 3 hrs., and the residue is removed through a seal excluding air, the zinc vapours passing in contradiction to the ore. The temperature required is 300—350° lower than that of present practice, and a recovery of 97.3% has been obtained on commercial-scale working, which presented no difficulties of a chemical nature. The practical difficulties arising are of a mechanical nature associated with the thermal expansion of the furnace and the maintenance of air seals at high temperature.

C. A. KING.

Tin plate and the electrochemical series. E. F. KOHMAN and N. H. SANBORN (Ind. Eng. Chem., 1928, 20, 76—79).—Experience shows that many of the phenomena of the corrosion of tin cans containing fruit are not such as are capable of simple explanation by a tin-iron galvanic couple with tin as cathode. Corrosion tests were carried out with specimens of base plate and sheet tin immersed in fruit juice in sealed glass bottles. Corrosion was determined by weighing, and electrical potentials were also determined. It was found that the corrosion of iron was greater than that of tin when the specimens were not in contact. When they were in contact, that of iron was decreased and that of tin increased so that the relative positions of the metals in the electrochemical series were reversed. When the area of iron relative to tin was increased the protection of the former was decreased. Iron was usually anodic to tin at the beginning of the test, but then became cathodic. These results may be explained by polarisation or differences in hydrogen overvoltages.

C. IRWIN.

Thermal expansion of beryllium and aluminium-beryllium alloys. P. HIDNERT and W. T. SWEENEY (U.S. Bur. Standards, Sci. Paper No. 565, 1927, 22, 533—545).—The coefficient of expansion of beryllium (98.9%) increases rapidly with temperature, the average values per °C. for temperature intervals being 12.3 (20—100°), 13.3 (20—200°), 14.0 (20—300°), 14.8 (20—400°), and 15.5 (20—500°), all $\times 10^{-6}$. Beryllium expands considerably less than the other elements of its sub-group. Expansion of aluminium-beryllium alloys decreases with increase in the content of beryllium and increases with temperature, being also greater on a second heating of the alloy. An alloy containing 30% Be has coefficients of expansion approximately 20% less than those of aluminium. Values for a range of alloys containing 4—33% Be are given.

C. A. KING.

Alloys of beryllium with copper, nickel, cobalt, and iron. G. MASING (Z. Metallk., 1928, 20, 19—21).—Conductivity measurements show that the saturated solid solution of beryllium in copper contains 2.4% Be at 850°, and only 0.75% Be at 400°, hence alloys containing 0.75—2.4% Be undergo age-hardening after quenching from 850°. The hardness of the quenched alloys increases from 65 to 100 with increase of beryllium within the above range, and that of the corresponding alloys after age-hardening at 300—350° from 70 to 440. The elastic limit, yield point, tensile strength, elongation, and bending strength of the 2% Be alloy quenched from 800° are, respectively, 6.2, 11.0, 51.3 kg./mm.², 46%, and 45 kg./mm.²; the corresponding values after ageing at 350° are 25, 74, 87 kg./mm.², 4%, and 163.7 kg./mm.². The electrical conductivity of copper is considerably reduced by addition of beryllium, but the resistance to corrosion is increased. Alloys containing 5—10% Be quenched from above 600° consist entirely of the hard β -solid solution, and cannot be worked cold; below 590° the β -alloy decomposes into $\alpha + \gamma$, when the alloys may be extruded satisfactorily, but not cold-worked. Beryllium dissolves to a limited extent (about 2%) in nickel, cobalt, and iron, and as the solid solubility is greater at 1100° than at 600° the alloys may be heat-treated and aged in a similar manner to the copper alloys.

A. R. POWELL.

Age-hardening phenomena in beryllium-copper alloys. O. DAHL (Z. Metallk., 1928, 20, 22—24; cf. preceding abstract).—The maximum hardness (440) of the copper alloy containing 2.5% Be is obtained by ageing for 1—4 hrs. at 300—350°; at lower temperatures the hardness rises only slowly with the time of ageing, 16 hrs. being required at 250° to obtain a hardness of 320°. At 450° the finely-dispersed β -particles agglomerate rapidly, and a maximum hardness of 180 is obtained in 45 min., further heating resulting in a rapid fall in the hardness number. The optimum quenching temperature is about 840°; at lower temperatures the time required to obtain the maximum hardness is increased, and the value obtained is lower. During ageing at 150—250° the electrical conductivity first falls slightly, then rises at a rate which is greater the higher the ageing temperature; this behaviour is similar to that of duralumin under the same conditions, and appears to indicate that there are two reactions

proceeding during the ageing of beryllium-copper alloys. Microscopical and röntgenographic investigation has so far failed to establish the nature of these reactions.

A. R. POWELL.

Corrosion research. J. CZOCHRALSKI and E. SCHMID (Z. Metallk., 1928, 20, 1—7).—The rate of corrosion of a metal in wire or strip form may be followed by determining the tensile strength and ductility (elongation) after immersion for varying periods in the corroding medium, or by immersing the specimen under tension in the medium and observing the time that elapses before breaking occurs. Tests carried out in this manner with aluminium showed that hard-drawn wire dissolves more readily than annealed wire, whereas the converse is true in the case of copper. The dezincification of ($\alpha + \beta$)-brass in 17% hydrochloric acid at 50° is confined entirely to the β -constituent, which may be dissolved practically completely from a 3 mm. wire before any attack on the α -constituent is apparent; if the brass contains lead this also dissolves. Thus, a wire consisting of 57.9% Cu, 40.4% Zn, and 1.7% Pb was freed from the β -constituent after immersion for 208 hrs. in the acid, and then assayed 72% Cu, 27.9% Zn, and 0.05% Pb.

A. R. POWELL.

Gases in metals. III. Determination of nitrogen in metals by fusion in vacuum. L. JORDAN and J. R. ECKMAN (U.S. Bur. Stand., Sci. Paper No. 563, 1927, 22, 467—485).—For the determination of nitrogen in metals, the metal is fused *in vacuo* in a high-frequency furnace and the gases are absorbed by calcium vapour contained in an iron tube enclosed in a silica combustion tube heated at about 800°, the whole system being evacuated; this method should determine also uncombined nitrogen in a metal. The resultant calcium nitride is decomposed by hydrogen chloride and ammonia determined in the usual manner. Similar values for nitrogen were obtained by the acid-dissolution and vacuum-fusion methods in the analyses of aluminium, zirconium, and chromium nitrides, and appreciably higher values by the latter method for nitrides of silicon, titanium, and vanadium, and also for high-silicon ferrous alloys.

C. A. KING.

Cohesion at soldered surfaces. T. B. CROW (Trans. Faraday Soc., 1927, 24, 159—161).—A number of tensile tests on soldered joints are described from which an attempt is made to relate the tensile strength of the joint to that of the solder.

R. W. LUNT.

Corrosion by oil. YOUNG. Lubricating oils and steel. BORODULIN.—See II. Aluminium and cement. MEYER.—See IX. Precipitation of dust. DEUTSCH.—See XI.

PATENTS.

Cupola furnace. O. WEICHEL (B.P. 272,216, 1.7.27. Ger., 2.6.26).—In a cupola furnace provided with more than one row of nozzles, the doors of the nozzles in a vertical line are carried on one spindle, constructed with hollow and solid members so that each carries a separate door. The whole is enclosed by the air box.

C. A. KING.

Bright-annealing furnace. SIEMENS-SCHUCKERT-WERKE G.M.B.H., Assees. of T. STASSINET (B.P. 262,803, 9.12.26. Ger., 12.12.25).—Vapours produced in annealing

pots when bright annealing are removed with a slow current of inert gas by maintaining the entire length of the gas outlet above the b.p. of the vapour constituents, either by heat insulation or by electrical heating coils.

C. A. KING.

Manufacture of steel. J. K. SMITH, Assr. to GRANULAR IRON Co. (U.S.P. 1,651,638, 6.12.27. Appl., 10.1.25).—When iron produced directly in the solid form from the ore is used in the open-hearth furnace, the charge is covered with a layer of molten pig iron to inhibit oxidation.

T. S. WHEELER.

Treatment of ferrous metal [steel]. W. F. HODGES, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,651,891, 6.12.27. Appl., 26.4.24).—Decarbonisation of steel by hydrogen during brazing is inhibited by previously plating it with copper.

T. S. WHEELER.

Elimination of phosphorus from pig iron. R. SCHENCK, Assr. to VEREIN. STAHLWERKE A.-G. (U.S.P. 1,650,157, 22.11.27. Appl., 24.12.25. Ger., 11.9.24).—The metal is blown with carbon monoxide in presence of calcium oxide to convert phosphorus into calcium phosphate.

T. S. WHEELER.

Heat-resisting metallic articles. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of W. E. RUDER (B.P. 258,295, 14.9.26. U.S., 14.9.25).—Articles made from a malleable alloy of iron and chromium, containing at least 10–30% Cr, are subjected to heat-treatment in the presence of aluminium, whereby the latter is caused to alloy with the surface of the article so as to form a protective layer. The aluminium may be applied either by placing the article, surrounded with a mixture of aluminium, ammonium chloride, and zinc, into a rotatory oven, heating at about 450°, and then firing at 700–800°; or, in the case of wires, by passing them through a bath of molten aluminium and then firing; or the article may be sprayed with a mixture of powdered aluminium and a suitable liquid binder and then fired at 750–900°.

M. E. NOTTAGE.

Joining of metals. F. C. KELLEY, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,655,273, 3.1.28. Appl., 13.4.23).—The ferrous metal article is coated with a film of molten copper in the presence of a reducing agent, and a copper article may then be brazed on to the coated surface.

M. E. NOTTAGE.

Production of aluminium conducting material of high electric conductivity, strength, and chemical resistance. T. GOLDSCHMIDT A.-G. (B.P. 269,580, 14.4.27. Ger., 19.4.26).—The material, e.g., aluminium alloy, after glowing, quenching, and, if necessary, cold compression, is stored at room temperature for 3–5 days, and is subsequently heated at about 200° for 10–20 hrs. [Stat. ref. to B.P. 260,275.]

M. E. NOTTAGE.

Extraction of platinum from ores etc. J. W. MELLOR (B.P. 282,543, 18.11.26).—Finely-divided platinumiferous ore is agitated with, or blown by means of a current of producer gas into, a mass of molten lead or other metal which will alloy with the platinum, and the gangue is subsequently blown off the surface of the metal. The process is repeated until the molten metal is sufficiently rich in platinum for further treatment, e.g., cupellation in the case of lead. The addition

of a small quantity of alkali or alkaline-earth metal to the lead improves its power of removing platinum from the ore.

A. R. POWELL.

Metal alloys. G. C. DEETER (U.S.P. 1,656,695–6, 17.1.28. Appl., [A, B] 20.4.26).—Alloys for scavenging purposes consist of (A) 40–50% Ni, 3–10% Sn, 0.3–1% P, 1.5–5% Mn, and the balance copper, or (B) 5–10% Ni, 3–10% Sn, 0.75–5% Mn, 0.25–3% P, and 72–92% Cu.

F. G. CROSSE.

[Amalgams for] carrying out exothermic chemical reactions. E. C. R. MARKS. From SELDEN Co. (B.P. 282,559, 10.1.27).—The temperature of exothermic reactions (e.g., air oxidation of naphthalene to phthalic anhydride, or anthracene to anthraquinone in presence of catalysts) is controlled by a jacket containing an alloy or amalgam of metals one of which boils below and one above the desired reaction temperature. E.g., a cadmium amalgam containing 12% Cd (b.p. 778°) and 88% Hg (b.p. 357°) is semi-liquid at 20° and has b.p. 370°; the amalgam with 40% Cd is liquid at 151° and boils at 430°; with 25% Cd, m.p. 100°, b.p. 400°. An alloy of 10% Sn, 10% Pb, and 80% Hg has m.p. 20°, b.p. 370°; of 40% Sn, 40% Pb, and 20% Hg, m.p. 160°, b.p. 450°; of 30% Sn, 30% Pb, and 40% Hg, m.p. 120°, b.p. 405°. The amalgams have higher heat conductivity and lower density than mercury alone.

C. HOLLINS.

Production of protective and resistant coatings on metals. SIEMENS & HALSKE A.-G., Assees. of C. G. FINK and L. C. PAN (B.P. 263,183, 17.12.26. U.S., 18.12.25).—After plating a foundation metal with a protective metal, e.g., chromium, the plated metal is immersed in molten lead to eliminate "pin-hole" defects. Lead adhering to the plating is removed by abrasion or acids.

C. A. KING.

[Anti-corrosive] treatment of metals. G. H. HOWE, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,655,269, 3.1.28. Appl., 1.4.25).—Metallic articles are protected from oxidation by covering them at ordinary temperatures with a coating composed of aluminium powder and a liquid nitrocellulose binder which wets and firmly adheres to metals at ordinary temperatures and volatilises at higher temperatures. The coating is dried and then fired at a temperature at which the aluminium alloys with the metal and above that at which the binder volatilises.

M. E. NOTTAGE.

Coating articles of various kinds with metal. E. D. FELDMAN (B.P. 261,017, 5.11.26. U.S., 6.11.25).—Articles made from non-conducting materials such as wood, felt, tile, etc. are coated with a sticky substance, e.g., varnish or bitumen, on to which an alloy of lead and tin is sprayed. The article is then electroplated with the desired metal.

C. A. KING.

Thermostatic material. P. H. BRACE, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,650,979, 29.11.27. Appl., 12.3.25).—A thermostatic strip suitable for use at 800° comprises elements of molybdenum and of an iron alloy containing 22% Co and 22% Cr, both elements being chromium-plated.

T. S. WHEELER.

Pyrometer thermocouple [for immersion in molten metals]. O. BROPHY (U.S.P. 1,651,750, 6.12.27.

Appl., 27.3.23).—The elements comprise a central core and a sheath, the latter being plated with a resistant metal, *e.g.*, tungsten.
T. S. WHEELER.

Concentration of mineral ores in sluice boxes etc. F. H. NASH (B.P. 282,298, 21.6.27).—A sluice box for treating mineral ores is provided with an agitator substantially of the form of an agricultural harrow which is capable of vertical movement and also of a horizontal reciprocating motion so as to disturb the bed to any desired depth.
C. A. KING.

Concentration of ores and minerals by flotation. [A] C. C. SMITH and [B] W. A. DOUGLAS, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,652,060 and 1,652,099, 6.12.27. Appl., [A] 10.1.27, [B] 25.10.26).—(A) Mercaptobenzthiazole, mixed with sodium sulphite, produces selective flotation of lead sulphide in presence of zinc sulphide. The latter is recovered when further quantities of the thiazole and copper sulphate are added. (B) An alkyl xanthoformate is used. T. S. WHEELER.

Deoxidiser. C. J. RODMAN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,652,496, 13.12.27. Appl., 21.12.23).—A composition for treating the atmosphere of transformers comprises a finely-divided metal, lower in the electrochemical series than hydrogen, capable of combining with oxygen at ordinary temperatures but not capable of decomposing water, *e.g.*, copper, and a catalyst for the oxidation, *e.g.*, calcium chloride.
T. S. WHEELER.

Desulphurising of molten metal and briquettes used therefor. R. WALTER (U.S.P. 1,651,492, 6.12.27. Appl., 16.3.22. Ger., 8.4.21).—See B.P. 179,146; B., 1923, 783 A.

Alloy suitable for loading telephone and telegraph conductors. W. S. SMITH and H. J. GARNETT (U.S.P. 1,656,764, 17.1.28. Appl., 29.4.26. U.K., 10.7.25).—See B.P. 261,060; B., 1927, 80.

XI.—ELECTROTECHNICS.

Electrical precipitation of metalliferous dust from industrial gases. W. DEUTSCH (Z. Metallk., 1928, 20, 25—27).—A brief description with diagram of the Lurgi modification of the Cottrell electrical gas-purification plant and a review of its uses in the recovery of lead, zinc, silver, and alumina from metallurgical gases.
A. R. POWELL.

Inexpensive cell for the purification of colloids by electro dialysis. R. BRADFIELD (Ind. Eng. Chem., 1928, 20, 79—80).—The cell is constructed from a rubber storage battery case sawn into three. The edges of each section are squared so that water-tight joints can be made by merely tightening up the brass bracing rods which hold the whole together, and these joints hold the parchment or other membrane. The cathode is of nickel or copper gauze and the anode of platinum or carbon, the electrodes being similar in area to the membranes. With parchment the anion requires longer time for removal than the cation.
C. IRWIN.

Tin plate. KOHMAN and SANBORN. **Conductivity of aluminium.** BOHNER.—See X. **Quinhydrone and hydrogen electrodes.** SNYDER.—See XVI.

PATENTS.

[Electric] induction furnace. P. H. BRACE, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,655,983, 10.1.28. Appl., 2.4.27).—A crucible containing a metallic charge is arranged within an induction coil, and a partial core of magnetic material projects partially into the crucible so that one end of the core is surrounded by the metallic charge. J. S. G. THOMAS.

Insulation of finely-divided magnetic material [for dust cores]. H. H. LOWRY, Assr. to BELL TELEPHONE LABORATORIES, INC. (U.S.P. 1,651,957—8, 6.12.27. Appl., 3.1.27; cf. U.S.P. 1,647,737—8; B., 1928, 59).—The particles of the nickel-iron alloy used are coated with a mixture (A) of magnesium borate and silica or (B) of zinc and magnesium oxides.
T. S. WHEELER.

Insulated electrical conductor. F. L. ROMAN and H. T. WINSEMIUS, Assrs. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,649,924, 22.11.27. Appl., 12.12.25).—A fireproof coating for wire comprises cotton impregnated with a mixture of crude chloronaphthalenes.
T. S. WHEELER.

Manufacture of [gas-filled] incandescence electric lamps. D. S. GUSTIN, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,651,387, 6.12.27. Appl., 22.6.23).—The lamp is filled with a mixture of argon and hydrogen (10%) containing a small quantity of the vapour of a carbon compound free from oxygen and oxidising material, *e.g.*, *p*-dibromobenzene or aniline.
T. S. WHEELER.

Electric incandescence lamp. H. WOLFF, F. KOREF, and F. SKAUPY, Assrs. to GEN. ELECTRIC Co. (U.S.P. 1,655,488, 10.1.28. Appl., 3.11.25. Ger., 18.3.25).—An electric lamp contains a gaseous filling, *e.g.*, hydrogen, including a substantial amount of halogen acid gas, at a sufficiently high pressure to reduce evaporation of the material operated at incandescence.
J. S. G. THOMAS.

Incandescence electric lamp. G. HOLST, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,655,502, 10.1.28. Appl., 11.3.27. Holl., 10.4.26).—A filament is secured to leading-in wires extending into a sealed envelope, and an auxiliary electrode, provided with a material which in response to an electrical discharge evolves a gas forming with the vaporised material of the filament a compound which is more transparent than the latter, is connected so as to act as an anode to a portion of the filament.
J. S. G. THOMAS.

Production of highly emitting oxide cathodes for electron tubes. E. SCHRACK and R. ETENREICH (RADIOWERK E. SCHRACK) (B.P. 275,542, 18.11.26. Austr., 6.8.26).—Material containing an alkaline earth is applied to a carrier material by means of a spray producing a finely-divided mist or by sprinkling. *E.g.*, a tungsten wire is coated with a thin layer of carbon by annealing the wire in an atmosphere of hydrogen saturated with tetrahydronaphthalene or containing acetylene, and sprinkled with a suspension of strontium carbonate in dilute alcoholic resin solution. The strontium carbonate is reduced to oxide by heating at 800° *in vacuo*.
J. S. G. THOMAS.

Manufacture of cathodes for thermionic valves. GEN. ELECTRIC CO., LTD. From A. JUST (B.P. 282,539, 10.11.26).—A refractory metal, *e.g.*, molybdenum, is mixed or coated with about 3% of a mixture of an alkaline-earth oxide with a very small proportion of either another alkaline-earth oxide or with an oxide of the rare-earth metals, *e.g.*, thorium oxide or praseodymium oxide, the mixture being made up in the approximate ratio of 1000 : 1.
J. S. G. THOMAS.

Vacuum tube [thermionic valve]. L. WINKELMANN (U.S.P. 1,650,921, 29.11.27. Appl., 9.5.23).—The performance of thermionic valves is improved by coating the electrodes or the inner surface of the bulb with a mixture of phosphorus and a radioactive substance.
T. S. WHEELER.

Storage battery. T. A. EDISON, ASSR. TO T. A. EDISON, INC. (U.S.P. 1,651,196, 29.11.27. Appl., 14.5.23).—Separators of suitable porosity for use in cells of the nickel oxide-iron type are formed of asbestos fibre mixed with salicylic acid, which is afterwards removed by extraction of the material with potassium hydroxide solution.
T. S. WHEELER.

Electric furnace for melting or producing glass, water-glass cement, or other substances. C. E. CORNELIUS (U.S.P. 1,656,510, 17.1.28. Appl., 26.2.26. Swed., 23.3.25).—See B.P. 249,554; B., 1927, 303.

Gas detector (B.P. 273,296).—See I. **Heat-resistant compounds (B.P. 271,503).**—See VIII. **Aluminium conductors (B.P. 269,580).**—See X.

XII.—FATS; OILS; WAXES.

Detection of coconut oil in cacao butter and cocoa preparations. F. HÄRTEL (Pharm. Zentr., 1928, 69, 37—41).—The test is based on repeated determinations of the Reichert-Meissl and Polenske values, coconut oil being richer in the volatile fatty acids than cacao butter. The first determination does not give the value corresponding to the full content of volatile acids; after a first determination, the residue is treated with a further definite quantity of water and the distillation continued; after this second treatment, a third is carried out in the same way. From the series of values so obtained, adulteration of the cacao butter may be detected and roughly estimated.
S. I. LEVY.

[Extraction of oil from] oil palm in Malaya. B. BUNTING, B. J. EATON, and C. D. V. GEORGI (Malayan Agric. J., 1927, 15, 353—371).—The preparation of a palm oil of low acidity necessitates modification of the native processes of West Africa; the essential features of the process adopted in Malaya are: (a) use of only ripe, undamaged fruit, (b) sterilisation of the fruit before treatment in order to destroy the naturally occurring enzymes which promote development of free fatty acidity, and (c) rapid extraction of the oil. The sterilisation is effected by heating the fruit under slight steam pressure, after which it is transferred to a digester consisting of a vertically arranged cylindrical vessel fitted with beaters whereby disintegration of the oil cells is effected. It is then transferred to a centrifugal extractor containing a perforated basket and having a well-fitting lid provided with a jointed steam pipe whereby steam is injected into

the mash while the centrifuge is being operated, thereby facilitating the expulsion of the oil. The period of oil extraction is 15 min. and the oil is purified and freed from sludge and moisture by centrifuging in a machine of the De Laval type. Between 85 and 87% of oil in the pericarp can be recovered efficiently, and the acidity of the oil is about 3% (as palmitic acid).

H. M. LANGTON.

Atmospheric oxidation of methyl and glycol esters of β -eleostearic acid. A. B. MILLER and E. CLAXTON (Ind. Eng. Chem., 1927, 20, 43—48).—The physical and chemical changes occurring during controlled air-oxidation of β -eleostearic acid and its methyl, glycol, and glycerol esters have been examined. After 63 hrs. at 82° the methyl ester absorbed 13% O and was still fluid and oily; the glycol ester after 45 hrs. absorbed 11% O and was a sticky, elastic gel; the acid after 29 hrs. absorbed 6.5% O and formed a tough, elastic gel; and the glycerol ester formed a short, friable gel and absorbed only 3% O during 19 hrs. heating. In all cases there was an increase in hydroxy-acid content, and the results indicated the presence of polymerides containing considerable amounts of oxygen and condensation products of β -eleostearic acid. Evidence of ketol and enol formation was also noted. It is suggested that an important function of the alcohol valency is in determining the stability of the ester and preventing the liberation of free acid which, by condensation, actively influences gel formation.
E. H. SHARPLES.

Phytosteryl acetate test and the phytosterol of sunflower seed oil. J. ALLAN and C. W. MOORE (J.S.C.I., 1927, 46, 433—434 T).—This test, whilst still of use in the detection of vegetable in animal fats provided that *isocholesterol* and *bombicestrol* are known to be absent, is not suitable for the detection of animal in vegetable fats since phytosterols which yield acetates melting below 125° have been isolated (cf. Power and Browning, B., 1914, 885; Stuart, *ibid.*, 1923, 462 A). It is now shown that the *phytosterol* C₂₉H₄₉·OH, which occurs in sunflower seed oil in normal amount and constitutes the major portion of the phytosterols present yields an *acetyl* derivative, m.p. 119—119.5° (corr.) after recrystallisation from acetic anhydride and then from 95% alcohol until of constant m.p. A mixture of equal parts of this *acetyl* derivative and *cholesteryl acetate* melts at 121—122° (corr.). Sunflower seed oil also contains a small amount of a *phytosterol*, the *acetyl* derivative of which has m.p. 115.5° (corr.). The corrected m.p. of *phytosteryl acetates* prepared from other oils and fats are, from hydrogenated sunflower seed oil 119.25°, olive oil 123.5°, cotton seed oil 125.5°, palm kernel oil 127°, arachis oil 129°, hydrogenated arachis oil before deodorisation 128.5°, hydrogenated arachis oil after deodorisation 128°, coconut oil 129.5°, *sesamé* oil 129.5°, palm oil 131°, linseed oil 131°.
W. J. POWELL.

Sulphonated oils and leather. BUMCKE.—See XV.

Animal skin fat. THEIS.—See XV.

PATENT.

Emulsifying agents (B.P. 258,551 and 268,387).—See III.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Red lead problems. I. Settling and hardening of red lead. II. Colour of red lead. III. Red lead as a composite pigment. C. P. VAN HOEK (*Farben-Ztg.*, 1928, 33, 981—983, 1046—1049).—A review of the literature on the causes of settling and hardening of the sediment in red lead-oil paints, and their successful overcoming in modern "dispersed" red leads. Among the many influences discussed are:—grinding, presence or absence of turpentine or other diluent, formation of lead soaps and lead glyceroxide (an important factor in hardening), dependence of this factor on free monoxide content of the pigment and free fatty acid content of the oil, particle size, and rate of settling (a slow-settling red lead usually not hardening). A somewhat full reference to Junk's work indicates that the settling and hardening cannot be attributed to one particular cause, many varying factors being jointly responsible. Microscopical examination reveals "active" and "inactive" particles, absence of the latter in "dispersed" red lead giving it its favourable non-settling properties. This activity or non-activity is accounted for by variations in the structure of the labile combination of lead monoxide and peroxide constituting the pigment. Suggestions that water is adsorbed on the surface of "dispersed" red lead are disproved. The variation in colour of red lead is discussed with reference to the colour of different varieties of lead monoxide and peroxide, the probable constitution of red lead being a lead monoxide substratum on which are adsorbed ultramicroscopic particles of lead peroxide. Microscopical examination shows that conversion of lead monoxide into red lead is not accompanied by any appreciable increase in particle size, the heterogeneous nature of the red lead particles being demonstrated under ordinary and polarised light. Various views on the composite nature of the pigment are discussed.

S. S. WOOLF.

Pigment and vehicle. E. KLUMPP (*Farben-Ztg.*, 1928, 33, 1044—1045).—The absorption by white lead, zinc oxide, blanc fixe, and china clay of various aqueous and oily vehicles is tabulated, the value being expressed in terms of volume. The results are discussed with reference to the relation between adsorption and absorption. A white lead-linseed oil paste (67% of oil on pigment by vol.) consists of primary white lead particles separated by a thin layer of oil, but a zinc oxide-paraffin oil paste (440% of oil on pigment by vol.) consists of agglomerates or secondary particles composed of wetted primary particles. Other factors possibly tending to raise the vehicle absorption of a pigment are the presence of adsorbed gases or vapours and the formation of "vehicle-sheaths" of relatively considerable diameter around the particles.

S. S. WOOLF.

Iron-cyanogen colours, in particular Milori blue. A. MÜLLER-MAGDEBURG (*Chem.-Ztg.*, 1927, 51, 923—924).—Milori blue or steel blue is closely related to Prussian blue, and is valued for its reddish shade. It is prepared by oxidising the white paste formed by mixing sodium or potassium ferrocyanide and ferrous sulphate, the oxidising agents used being chlorine, ferric chloride, potassium dichromate and sulphuric acid, or, most commonly, potassium chlorate and hydrochloric or

nitric acid. On the large scale, using either nitric acid alone or potassium chlorate and hydrochloric acid, about 35 kg. of Milori blue are obtained from 50 kg. of potassium ferrocyanide and 45 kg. of ferrous sulphate. The chemistry of the formation and composition of Prussian blue and its related pigments is discussed.

W. T. K. BRAUNHOLTZ.

PATENTS.

Paint-removing composition. H. L. LEASK (B.P. 282,549, 8.12.26).—Wax, e.g., beeswax, paraffin wax, earth wax, is melted and coal-tar light oils are stirred in. Benzol and methylated spirit are finally added.

S. S. WOOLF.

Manufacture of red lead. J. A. SCHAEFFER, J. H. CALBECK, and J. R. CRENSHAW, Assrs. to EAGLE-PICHER LEAD Co. (U.S.P. 1,652,217, 13.12.27. Appl., 9.5.25. Renewed 21.10.27).—Ground litharge obtained by the oxidation of molten lead is mixed with litharge condensed from its vapour, and the mixture is oxidised in a reverberatory furnace.

T. S. WHEELER.

Manufacture of a non-dusting carbon [black] pigment. S. E. SHEPPARD and L. W. EBERLIN, Assrs. to EASTMAN KODAK Co. (U.S.P. 1,651,733, 6.12.27. Appl., 26.11.26).—Powdered carbon black is impregnated with a benzene solution of a wax, and the solvent is evaporated.

T. S. WHEELER.

Preparation of lacquers from tung oil. W. SCHMIDDING (B.P. 282,574, 28.2.27. Addn. to B.P. 247,599; B., 1926, 988).—Lacquers are prepared in an installation of three vessels located one above the other and connected by pipes, tung oil being heated in the middle vessel and additional material being run in, at a suitable stage in the cooking of the tung oil, from the upper vessel (also provided with heating apparatus). The mixture is eventually run off into the lowest vessel, where cooling and thinning take place. The outlet closure of the middle vessel is located within the vessel to avoid trapping of gelatinised oil. The three vessels are connected to a common vapour exhaust.

S. S. WOOLF.

Nitrocellulose lacquer composition. C. BOGIN, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,651,578, 6.12.27. Appl., 1.6.25).—*cyclo*Hexanol is claimed as an ingredient.

T. S. WHEELER.

Production of a composition for use in the manufacture of, or for use as, varnishes etc. H. W. MATHESON, Assr. to CANADIAN ELECTRO PRODUCTS Co., LTD. (U.S.P. 1,650,899, 29.11.27. Appl., 19.8.22).—The products obtained by heating acetylene-phenol resins with hexamethylenetetramine or paracetaldehyde are dissolved in suitable solvents.

T. S. WHEELER.

Production of condensation products of phenols with aldehydes. G. PETROFF (B.P. 283,002, 1.2.27).—Resols are obtained by condensing a phenol with an aldehyde in presence of the sodium salt of *p*-toluene-sulphonchloroamide ("Chloramine T") as catalyst instead of the usual basic or acid catalysts. The resols may be hardened by heat, or by means of acid catalysts dissolved in an alcohol, chlorinated hydrocarbon, or fatty acid, or by alkaline catalysts, dissolved as glycerol or phenol salts in an alcohol.

C. HOLLINS.

Preparation of fluid condensation products from phenols and formaldehyde. G. PETROFF (G.P. 442,211, 23.7.21).—Phenols are condensed with formaldehyde in the presence of lead acetate (in amount at least 10% of the amount of phenol used), the mixture being boiled. The condensation proceeds under acid conditions, partial decomposition of the lead acetate occurring and giving rise to lead salts of "novolacs." After removal of the water layer the condensation is continued until the product has the required density. It is soluble in alcohol, benzol, and turpentine, etc., and may be blended with drying oils, natural resins, etc. S. S. WOOLF.

Manufacture of condensation products of carbamide and its derivatives and formaldehyde. I. G. FARBENIND. A.-G. (B.P. 259,950, 6.10.26. Ger., 16.10.25).—Condensation products of carbamide and its derivatives and formaldehyde are hardened by exposing them while in the gelatinous state to the action of sulphur dioxide and, if desired, heat. S. S. WOOLF.

Synthetic resin composition. H. L. BENDER, Assr. to BAKELITE CORP. (U.S.P. 1,650,109, 22.11.27. Appl., 10.11.26).—The resin formed by the action of *p*-toluenesulphonamide on formaldehyde is of value as a plasticising agent for phenol-formaldehyde resins. T. S. WHEELER.

Preparation of solvents etc. from resins. H. G. HJERPSTED (F.P. 618,740, 10.7.26. Denm., 11.7.25).—Resins are subjected to dry distillation with calcium or barium, and the distilled products are hydrogenised, e.g., in an autoclave, in the presence of catalysts, e.g., nickel, platinum, if necessary. S. S. WOOLF.

Production of resinous condensation products of the urea-formaldehyde type. ROHM & HAAS Co., Asses. of F. LAUTER (B.P. 256,248, 28.7.26. U.S., 28.7.25).—See U.S.P. 1,633,337; B., 1927, 788.

Cellulose nitrate solutions (B.P. 282,172).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Brittleness tests for rubber and gutta-percha compounds. G. T. KOHMAN and R. L. PEEK, JUN. (Ind. Eng. Chem., 1928, 20, 81—83).—India-rubber and gutta-percha compounds, e.g., insulating materials, become brittle at a definite temperature, which is characteristic of the individual material. An apparatus is described for determining the highest temperature (the "brittle temperature") at which a sample undergoes fracture when suddenly bent through an angle of 45°. The test may also be applied when the sample is under high hydrostatic pressure. The "brittle temperature" probably represents a temperature range in which the physical properties of the material undergo an abrupt change. D. F. TWISS.

Reduction of period of vulcanisation of thin dipped goods in sulphur chloride vapour. R. DITMAR and G. BALOG (Gummi-Ztg., 1928, 42, 858—859).—Vulcanisation of films (thickness 0.03 mm.) in sulphur chloride vapour (1.5 c.c. in space of 31.02 c.c.) proceeds rhythmically with alternating aggregation and dispersion in a series of successive changes. Contrary to previous views, a period of 2 min. is sufficient to give a

well-vulcanised product with good ageing. The presence of zinc oxide, lithopone, or carbon black is favourable to good ageing behaviour, but certain organic colours have an unfavourable influence. D. F. TWISS.

PATENTS.

Manufacture of compositions of matter containing caoutchouc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 282,617, 10.6.26; cf. B.P. 282,172; B., 1928, 155).—Solutions of rubber, with or without colouring matters and cellulose nitrate, are prepared using as solvent an oily product of the catalytic hydrogenation of oxides of carbon or a liquid produced by the hydrogenation, acylation, or condensation of such oily product. D. F. TWISS.

Compounding and vulcanising rubber and products obtained therefrom. O. H. SMITH, Assr. to NAUGATUCK CHEMICAL Co. (U.S.P. 1,651,737, 6.12.27. Appl. 6.7.25. Renewed 3.5.27).—Sheet rubber is dipped in benzene containing sulphur, an amine, e.g., dibenzylamine, and a metallic salt of a carbon disulphide derivative, e.g., zinc butylxanthate, and is then heated at 100° for 2 hrs. T. S. WHEELER.

Vulcanisation of rubber. C. W. BEDFORD, Assr. to B. F. GOODRICH Co. (U.S.P. 1,650,975, 29.11.27. Appl., 28.6.24).—The additive products of metallic salts, e.g., zinc sulphate, and primary amines are used. T. S. WHEELER.

Production of vulcanised rubber. H. W. ELLEY, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,652,101, 6.12.27. Appl., 2.12.26).—Cyclic thiouram mono- and di-sulphides are claimed as accelerators. T. S. WHEELER.

Renewal of india-rubber and chiefly that contained in the covers of worn-out pneumatic tyres. C. DANIER, Assr. to SYNDICAT FRANCO-NEERLANDAIS (U.S.P. 1,656,694, 17.1.28. Appl., 16.9.26. Fr., 8.4.26).—See B.P. 269,127; B., 1927, 635.

Treatment of fabrics (U.S.P. 1,651,751).—See V.

XV.—LEATHER; GLUE.

Batch B. 14 hide powder. II. D. BURTON (J. Soc. Leather Trades' Chem., 1927, 11, 386—393; cf. B., 1927, 757).—B. 14 hide powder treated with a buffer solution of p_H 5.4 gave non-tan results closely agreeing with those given by B. 13 for liquid-blended extracts and for most liquid quebracho extracts. The non-tan values given by B. 13 were obtained with some extracts using B. 14 chromed with chrome alum. By using B. 14 chromed with a more acid chromium chloride solution, e.g., B. 14 chromed with a liquor containing 15 g. of anhydrous sodium carbonate per 100 g. of chromium chloride, results agreed most closely with those given by B. 13. Paessler's powder chromed with chrome alum gave lower non-tan values than B. 14 chromed with chrome alum or a more acid chromium chloride solution. D. WOODROFFE.

Influence of temperature on the tannin yield of pyrogallol tans. J. G. PARKER and J. A. GILMAN (J. Soc. Leather Trades' Chem., 1927, 11, 374—385).—Samples of myrobalans, valonia, sumae, and divi divi were scalded with boiling water prior to extraction, or heated for a time in an air oven at 103°, extracted, and

analysed. Both processes of preheating caused an increase in the yield of tan from myrobalans, and a diminution in the non-tans. The solutions were practically clear, showing little sign of deposition as usually occurs in the official method. Scalding overnight improved the colour of the liquors, whereas heating prior to extraction darkened the colour. The effect of both processes on *divi divi* was to decrease the tannin content and increase the insoluble matter. Scalding improved the colour and yield of tan from valonia and diminished the non-tans and insoluble matter, but was not suitable for sumac as it destroyed the tannin and increased both the insoluble matter and non-tans. Preheating caused a distinct increase in both the total soluble matter and the tans, with a slight increase of non-tans; the colour was not affected. In the official method of analysis, some hydrolysis must occur during the soaking period, which is retarded by preheating. Greater concordance in analytical results is obtained by preheating.

D. WOODROFFE.

Wattle barks. II. F. A. COOMBS, W. MCGLYNN, and M. B. WELCH (J. Proc. Roy. Soc. New South Wales, 1926, 60, 360—371).—The tannin content of wattle bark stored for thirty years was much the same as that of the original fresh bark, but the ratio of tannins to non-tannins had apparently increased, suggesting a possible increase in tannin at the expense of the non-tannins. Although the barks had become dark red in colour, there had been no apparent decrease in the solubility of the tannins. No evidence could be obtained of the existence in the old bark of fissures rendering it more readily penetrated by water. It appears probable that tannin reds do not actually occur in fresh wattle bark, but are formed in the extraction with water above 40°, which is necessary for the complete removal of the tannins, since boiling solutions of wattle tannins did not result in any loss of tannin. The losses which occur when solutions of the tannins in contact with partly spent bark are subjected to high temperatures seem to be due to the formation of a starch-tannin compound, partly soluble near the b.p., but separating out on cooling.

R. CUTHILL.

Sulphonated oils and their reaction on leather. G. BUMCKE (J. Amer. Leather Chem. Assoc., 1927, 22, 621—635; cf. B., 1915, 1214).—Two highly sulphonated cod oils *A* and *B* which proved unsatisfactory for fat-liquoring leather and two oils *C* and *D* which were satisfactory, have been analysed. The solubility in water, dark colour, and strong odour of *A* and *B* indicated that they were not made from pure Newfoundland cod oil. *C* contained a large amount of liquid unsaponifiable matter, and this reduced the other values. When the analytical data are calculated on the basis of oil free from moisture and unsaponifiable matter, the values for *C* agree with those for *A* and *B*, and indicate that the base in *C* is a fairly highly sulphonated oil, probably cod oil, though less sulphonated than *A* or *B*. The high content (5.5%) of unsaponifiable matter in *D*, excessive for a pure cod oil, might be due to shark liver, sperm, or other fish oil. It also contained a high percentage of ammonia soap (1.81%) not present in the other oils. It is suggested that the high percentages of oxidised fatty acids in *A* and *B* were responsible for

their unsatisfactory fat-liquoring properties. The base of *C* might have caused the same trouble but for the dilution with mineral oil which reduced the amount of oxidised and sulphonated fatty acids almost to the same quantity as in *D*. It is concluded that high degree of sulphonation alone or a high value for combined SO₃ is no criterion for the good quality of a sulphonated oil; further, evaluation of the oil from its so-called "sulphonation value" is quite erroneous. D. WOODROFFE.

Procter-Searle method of determining free mineral acid in leather. D. WOODROFFE (J. Soc. Leather Trades' Chem., 1927, 11, 493—397).—Procter-Searle determinations were made on samples of vegetable-tanned leathers which had been respectively treated with various products containing sulphonic groups, e.g., sulphonated oil, sulphite-cellulose, and synthetic tan. Positive results were given in every case (except that of a sulphonated vegetable oil which had been neutralised with soda) in which the sulphonated oils had been neutralised with ammonia. Samples of sulphonated cod oils, neutralised to Methyl Red, still gave positive results by the Procter-Searle method. A positive Procter-Searle value is therefore not always indicative of the presence of free mineral acid, but may be due to combined sulphuric acid. The *p*_H value of the water extract of a leather should be determined as well as its Procter-Searle value, to ascertain whether the product in the leather is deleterious or not.

D. WOODROFFE.

The "by difference" method of determining acid absorption by pelt in one-bath chrome-tanning experiments. F. HUDSON (J. Soc. Leather Trades' Chem., 1927, 11, 398—404).—Tanning tests were made with a pure chromium sulphate, violet and green chrome alum, glucose chrome liquor, and a chromium chloride liquor. Analysis of the leather showed that the "by difference" method of determining acid absorption by the pelt from the chrome-tanning liquors was not accurate. The "total sulphate" method was also useless, since it included any sodium sulphate absorbed. This value may, however, be useful for certain control purposes.

D. WOODROFFE.

Effect of temperature on the hydrolysis of skin and hair in saturated lime water. H. B. MERRILL and J. W. FLEMING (Ind. Eng. Chem., 1928, 20, 21—23).—Samples of skin and degreased hair from calfskins were treated with saturated lime water. After 3 days at temperatures below 35° the hair was more hydrolysed than skin; above this temperature the reverse was the case. The hydrolysis of hair was a nearly linear function of lime at any temperature. The hydrolysis of skin proceeded linearly until a certain amount of decomposition had taken place, after which the digestion was very rapid. The skin was dissolved completely in a few hours at 45°, in 1—2 days at 40°, in 4—5 days at 35°, in 35—40 days at 30°, but not in 60 days at lower temperatures. These results confirm the difference in the hydrolysis of hair and skin. Hair is hydrolysed by the breaking of a primary valency linking, probably the —S—S— linking of cystine. The hydrolysis of skin is a stepwise process. Skin consists of comparatively simple polypeptide-like aggregates linked together by

secondary valency forces. During the hydrolysis of skin, the secondary valency linkings are disrupted first, and then the component aggregates are hydrolysed into progressively simpler molecules until a compound is obtained which is more soluble in hot lime water than in cold.

D. WOODROFFE.

Further characteristics of animal skin fat. E. R. THEIS (J. Amer. Leather Chem. Assoc., 1928, 23, 4—9; cf. McLaughlin and Theis, B., 1925, 602).—Portions of heavy-cured steer hide cut up into 0.125-in. cubes, were soaked for 24 hrs. and 96 hrs. at ordinary temperatures, 48 hrs. at 37.5°, 24 hrs. and limed for 120 hrs., and 96 hrs. and limed for 120 hrs., respectively. The fat was extracted in each case after the experiment and analysed. The fat from soaked hide was dark brown, and that from limed hide a light golden-yellow. The saponification and iodine values of the fat diminished with increased time of soaking, but increased sharply with liming. The amount of unsaponifiable matter was inversely proportional to the saponification value of the extracted fat. The acid value increased with the length of soak period. Steer skin fat consisted of 2% of phospholipin, 24% of solid neutral fat, and 74% of liquid neutral fat, and the fatty acids of these ingredients were not highly unsaturated. It is suggested that soaking removes the lower glycerides from animal skin fat, leaving only the higher members. Liming may possibly saponify some of the cholesteryl esters in skin, thus increasing the saponification value of the fat extracted. The decrease in iodine value during soaking is attributed to bacterial oxidation of the unsaturated fatty acids, and the increase after liming must be due to selective saponification by the lime.

D. WOODROFFE.

Behaviour of agar towards iodine. ROSENTHALER.—See XX.

PATENTS.

Preservation of hides and skins. SALT UNION, LTD., W. CLAYTON, and W. E. GIBBS (B.P. 282,128, 13.8. and 31.12.26).—0.08% of lead as lead chloride or 0.0053% of cadmium as cadmium chloride is added to a neutral natural brine (d 1.20), which may or may not be dyed, and the solution allowed to crystallise so as to produce a solid salt containing less than 1% of a lead or cadmium compound distributed throughout the crystal structure. The product is bactericidal, and is used for preserving hides and skin, thus preventing salt stains.

D. WOODROFFE.

Printing of chrome leather. ANC. ÉTABL. A. COMBE & FILS & CIE. SOC. ANON. (B.P. 256,195, 28.6.26. Fr., 31.7.25).—The colouring of chrome leather by dyeing or printing is satisfactory provided that the leather is not dried between tanning and dyeing. Chrome leather loses the greater part of its affinity for dyes when dried, and this affinity cannot be restored by steeping in water or steaming. After tanning, the chrome leather is wrung so that it contains 50—70% of moisture at the time of dyeing or printing. Alternatively, the wet leather is impregnated with an aqueous solution of glycerin and then dried so that it contains not less than 20% of glycerin; such leather behaves towards dyes similarly to leather which has been maintained wet.

A. J. HALL.

Manufacture of solutions of gelatin in organic solvents. I. G. FARBENIND. A.-G. (B.P. 279,443, 15.10.27. Ger., 20.10.26).—Gelatin is dissolved in water by the aid of an alkali or alkaline-earth hydroxide, in the absence of any other electrolyte, and an organic diluent is added; the resulting solution is alkaline. A gelatin solution of any desired p_H value may be prepared by mixing this product with a gelatin solution in which an acid has been used as peptising agent, or by addition of an organic acid.

B. FULLMAN.

Impregnation of [moulded] leather with wax etc. W. C. MATTHEWS (B.P. 282,256, 29.3.27).

XVI.—AGRICULTURE.

Study of base exchange in soils with the aid of the quinhydrone electrode. F. O. ANDEREGG and R. P. LUTZ (Soil Sci., 1927, 24, 403—412).—Using Donnan's membrane equilibrium theory, equations expressing base exchange in soils have been worked out. These have been verified by substituting the values obtained by electrometric titration of a suspension of clay "acid" (clay practically freed from cations other than hydrogen by electro-dialysis) with solutions of salts in the presence of a quinhydrone electrode. The clay behaved like a monobasic acid in contact with solutions of neutral salts. The ionisation constants for the sodium, potassium, magnesium, and calcium salts of the clay are calculated, respectively as 1.70, 1.15, 0.73, and 0.76, all multiplied by 10^5 ; the exchange constants between these cations and hydrogen are also evaluated.

C. T. GIMMINGHAM.

Quinhydrone electrode and soil reaction. N. A. CLARK and E. R. COLLINS (Soil Sci., 1927, 24, 453—463).—Investigations are reported on some points in the application of the quinhydrone electrode to the measurement of p_H in soils. Various methods for preparation of the mixture of soil and water were tested, and reproducible values were obtained by stirring equal parts of soil and water, shaking for a few seconds, and allowing to settle for $\frac{1}{2}$ min., the electrode being placed in the soil paste (cf. Biilmann and Tovborg-Jensen, B., 1927, 887). There does not appear to be a definite soil-water equilibrium, even at a ratio of 1 : 1. Differences between the p_H of the supernatant liquid and the settled soil are accentuated if the soil is passed through a colloid mill. Temperature corrections are given for the quinhydrone electrode used with the saturated potassium chloride-calomel half-cell.

C. T. GIMMINGHAM.

Comparison of the quinhydrone and hydrogen electrodes for determining the hydrogen-ion concentration of soils. E. F. SNYDER (J. Agric. Res., 1927, 35, 825—834).—Measurements of p_H values of soils varying from p_H 4.0 to 9.0 by means of a modified Gillespie electrode showed the quinhydrone electrode to be generally satisfactory. Agreement with results obtained by the hydrogen electrode was close except on the alkaline side, where irregularities were more common. Using buffer solutions, results from quinhydrone and hydrogen electrodes were in close agreement except for the range p_H 8.0—9.0. When using suspensions having a soil-water ratio of 1 : 2, equilibrium with hydrogen electrodes was usually attained in 10 min.

Substitution of palladium or iridium black for platinum in the making of the electrode was without effect when soil suspensions were examined by means of the hydrogen electrode. Gold-foil electrodes were superior to platinum in quinhydrone electrodes used with buffer solutions.

A. G. POLLARD.

Relative proportions of exchangeable bases in some Scottish soils. A. M. SMITH (*J. Agric. Sci.*, 1928, 18, 68—75).—The changes effected by treatment of a number of soils from East Scotland with dilute solutions of sodium, potassium, and calcium chloride have been studied. The effect of 0.02*N*-solutions was very marked, the amount of exchangeable potassium in one soil being increased about 17 times by contact with 0.02*N*-potassium chloride solution. Changes caused by 0.002*N*-solutions were, however, very small, and comparable with what might be expected in practice when fertilisers are used. The content of exchangeable bases and their relative proportions vary considerably in different soils, and should prove useful as an additional characteristic of soil types.

C. T. GIMINGHAM.

Reaction, exchangeable calcium, and lime requirement of certain Scottish soils. W. G. OGG and W. T. DOW (*J. Agric. Sci.*, 1928, 18, 131—158).—Some of the results of a survey of the soils of a wide area in South-East Scotland, including a detailed study of a single farm, are discussed. The majority of the cultivated soils had a p_H value between 5.0 and 6.5, a lime requirement of 0.05—0.25% $CaCO_3$, and an exchangeable calcium content of 0.1—0.45% CaO . There was general agreement between the three sets of figures, though with many exceptions. Woodland, hill, and heath soils were characterised by a p_H usually below 5.0, exchangeable calcium usually below 0.1% CaO , and a high lime requirement. Cultivation had frequently brought about great changes in reaction. The results support the view that Scottish soils in general belong to the podsol group. Relationships between the data given and the natural vegetation are noted.

C. T. GIMINGHAM.

Fate of fractional incorporations of burnt lime in two soil zones. W. H. MACINTIRE (*Soil Sci.*, 1927, 24, 475—485).—Analyses are reported of the leachings from lysimeters filled with soil with which calcium hydroxide was incorporated at various rates, either in the upper or lower half (zone) of the soil. Over a period of 4 years, the losses of calcium from soil which received small additions of calcium hydroxide were practically the same as from the untreated soil. With larger amounts, losses of calcium were specially great when the lime was added to the lower zone of soil. Losses of calcium and increases of nitrates in the leachings were greatest during the first year. The relation of rate of liming to calcium conservation is discussed.

C. T. GIMINGHAM.

Distribution of phosphoric acid and potassium in soils. S. GERICKE (*Z. angew. Chem.*, 1928, 41, 52—56).—The absorption of phosphoric acid and potassium by plants from the soil depends not only on the presence of these materials in a readily assimilable form, but also on the physical properties of the soil itself. The variation in size of the soil particles is

an important factor; this may be determined by shaking the soil with water, allowing to settle for a definite time, and pouring off the liquid containing the smaller particles in suspension, thus dividing the sample into fractions of decreasing particle size. The results obtained depend largely on the method of preparation of the analysis sample, which must be shaken or boiled with water or rubbed with a pestle to separate the particles. Phosphoric acid is found in all fractions, but chiefly in the fraction containing particles of 0.02—0.2 mm., whilst potassium salts are concentrated in the smaller particles (less than 0.002—0.02 mm.). Both substances are more readily assimilated by germinating roots from the fractions of larger particle size than from the closely packed soils of small particle size.

W. J. POWELL.

Retention of phosphorus by soil colloids. C. A. ROSZMANN (*Soil Sci.*, 1927, 24, 465—474).—A pure colloidal clay, freed from exchangeable bases by electro-dialysis, absorbed a maximum amount of phosphoric acid, in presence of calcium or sodium salts, at p_H 3—4. There was no appreciable absorption above p_H 9. Iron and aluminium phosphates were not formed, since at the p_H at which maximum absorption occurred the salts of these metals are practically all soluble; nor was absorption due to formation of insoluble calcium salts, because soluble phosphorus only was taken into account. Some evidence is given that organic matter was responsible for some or all of the absorption of phosphorus.

C. T. GIMINGHAM.

Determination of the phosphate requirement of soils. F. TERLIKOWSKI, S. MICHNIEWSKI, and M. KWINICHIDZE (*Rocz. Nauk Rolniczych i Lesnych*, 1927, 17, 309—335; *Bied. Zentr.*, 1928, 57, 8—10).—In a critical examination of methods for estimating soil fertility the importance of adapting the experimental process to suit local soil conditions is emphasised. Fresh soil samples are essential in determining phosphate requirements. Drying alters the proportion of soluble phosphate in soils to an extent peculiar to each soil. The amount of soil phosphate assimilable by plants bears no definite relationship either to the total, water-soluble, or citric-soluble phosphate present. Many soils, showing no phosphate requirement by the Mitscherlich method, were deficient according to the Némec standard. There is a definite inverse relationship between the humus content of soils and their water-soluble phosphate content.

A. G. POLLARD.

Relation of organic matter and nitrogen content to series and type in virgin grassland soils. J. C. RUSSEL and W. G. MCRUER (*Soil Sci.*, 1927, 24, 421—452).—A study of virgin grassland soils in Nebraska indicates that texture is the chief factor determining the nitrogen content of any soil type. Textural variations can be expressed in terms of the hygroscopic coefficient, and the ratio of the coefficient to the percentage of nitrogen is used to investigate the distribution of nitrogen in various series and types. Some soil types are very homogeneous in content of nitrogen; others are decidedly heterogeneous. In a series of homogeneous types, the nitrogen content varies with the rainfall and with topography. The amounts of organic matter and nitrogen bear a fairly constant ratio to one another

both in extremes of rainfall and of texture. The application of the results to soil classification and mapping is discussed.
C. T. GIMINGHAM.

Milorganite—a new fertiliser material. V. H. KADISH (Ind. Eng. Chem., 1928, 20, 9—10).—Sewage waste of a uniform character, which is marketed at Milwaukee under this name, is a dry, evenly ground solid containing 4.08% of moisture, 86.13% of available, water-insoluble, organic nitrogen (by neutral permanganate method), and 2.43% of available phosphoric acid. It compares satisfactorily with other artificial fertilisers.

R. H. GRIFFITH.

Hot fermentation of stable manure by Krantz' process. H. GLATHE (Landw. Versuchs-Stat., 1927, 107, 65—129).—Examination of the drainage liquor from hot-fermenting manure heaps showed that the addition of peat mould to the heap not only reduced the drainage in quantity, but also lowered its content of phosphorus and potassium. During the making of ordinary cold-fermented manure the losses of nitrogen and dry matter were 2—3 times those from hot-fermented stacks. Rapidity of heating of manure stacks depended mainly on their water content and permeability to air. The latter was adequately regulated by loose stacking. Best results were obtained in hot-fermenting stacks by thorough trampling down and covering with earth immediately the maximum temperature was reached. In field trials the nitrogen of hot-fermented manure performed the same function as that from ordinary cold-fermented stacks. Its activity differed with soil type. Only in rich soils was the water-insoluble nitrogen effective on the first season's crop. Crop increases resulting from the use of hot-fermented manure were in some cases nearly twice those from cold-fermented manure. The better conservation of nutrients obtaining in hot-fermented stacks was formerly possible only by storage in deep stalls under cattle which consolidated the heap by trampling.
A. G. POLLARD.

More effective dust fungicides by the use of oxidising agents with sulphur. H. A. LEE and J. P. MARTIN (Ind. Eng. Chem., 1928, 20, 23—28).—The fungus *Helminthosporium sacchari*, which causes "eye-spot" in sugar cane, is not checked at all by such dust fumigants as Bordeaux mixture, calcium hypochlorite, and hydrated lime, and only slightly by sulphur. The addition of 1% of creolin to the sulphur, extra fine grinding, and more frequent applications caused no improvement. It was found, however, that the addition of oxidising agents to the sulphur, which promote the formation of pentathionic acid, greatly increased its efficiency. 1% of potassium permanganate as an addition reduced the disease by 90%; $\frac{1}{4}$ % of nitric acid and 5% of β -naphthol produced less improvement. These dusts also stimulated growth. Trials at another plantation on different soil gave much less successful results with these dusts. In this case Chloramine-T in a lime carrier was the most effective dust tried. Quantitative figures of degree of infection were carried out by counts of the numbers of spots per leaf.
C. IRWIN.

Differential response of barley varieties to manuring. F. G. GREGORY and F. CROWTHER (Nature, 1928, 121, 136).

PATENTS.

Manufacture of fertilisers. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G. (B.P. 273,291, 20.6.27. Ger., 28.6.26. Addn. to B.P. 235,860; B., 1925, 731).—The amount of silica required in the process of the parent patent is reduced to the proportion of 1 mol. of SiO_2 to 2 mols. of P_2O_5 , if sodium sulphate or chloride, or a mixture of the two, is used as the alkali salt. If the raw phosphate contains calcium not combined with phosphoric acid (e.g., as calcium carbonate or fluoride), sufficient silica must be added to convert this calcium into calcium orthosilicate. C. T. GIMINGHAM.

Fertiliser. SYNTHETIC AMMONIA & NITRATES, LTD., and A. E. MITCHELL (B.P. 282,463, 19.6.26).—Powdered alkali sulphate, preferably potassium sulphate, added to a mixture of ordinary crumbled superphosphate and powdered ammonium nitrate yields a mixed fertiliser which does not rot the bags in which it is stored nor corrode metal. By adjusting the proportions of the ingredients, a fertiliser containing predetermined amounts of nitrogen, phosphorus, and potassium can be prepared; as little as 2% of added sulphate gives a satisfactory mixture.
C. T. GIMINGHAM.

Production of a fertiliser product. G. BARSKY, ASSR. to AMER. CYANAMID Co. (U.S.P. 1,651,363, 6.12.27. Appl., 29.12.23).—An intimate mixture of calcium cyanamide and phosphate rock is treated with sulphuric acid.
T. S. WHEELER.

Manufacture of briquettes containing phosphate rock. J. A. BARR, ASSR. to INTERNAT. AGRICULTURAL CORP. (U.S.P. 1,655,981, 10.1.28. Appl., 9.4.24).—About 6 pts. of ground phosphate rock are mixed with 2 pts. of powdered coal and 2 pts. of silica, and the product is made into briquettes and heated at approximately red heat. The final product is substantially waterproof.
H. ROYAL-DAWSON.

Insecticide and method of applying same. W. L. TANNER, ASSR. to GRASSELLI CHEMICAL Co. (U.S.P. 1,652,291, 13.12.27. Appl., 1.10.21).—A sublimable, solid, organic, trivalent arsenic compound, e.g., phenarsazine chloride, is packed in a container with a core of celluloid, which is ignited to vaporise it.
T. S. WHEELER.

XVII.—SUGARS; STARCHES; GUMS.

Barium compound of technical interest from starch. E. STERN (Z. angew. Chem., 1928, 41, 88—91).—The precipitation of starch by means of its barium oxide compound avoids the necessity of evaporating large quantities of water in preparing soluble starches and starch pastes. The solutions obtained by treating starch with dilute sodium hydroxide solutions yield the whole of their starch content as a precipitate on addition of the equivalent quantity of barium chloride; the precipitate is readily collected on a filter and dried, and on treatment with a soluble sulphate (e.g., sodium or aluminium sulphate) in presence of water regenerates the soluble starch compound with precipitation of barium sulphate. The probable compositions of the various starch preparations and compounds are discussed.
S. I. LEVY.

PATENTS.

Manufacture of fructose [lævulose] from inulin. CHEM. FABR. AUF ACTIEN (FORM. E. SCHERING) (B.P. 272,876, 23.5.27. Ger., 15.6.26).—Aqueous inulin pulp is heated with volatile organic acids, especially formic (also acetic, carbonic, etc.). The resulting syrup, treated with charcoal and concentrated *in vacuo*, yields pure crystalline lævulose, the acid being simultaneously driven off.
B. FULLMAN.

Starch conversion products. W. D. STEIN (B.P. 282,178, 28.9.26).—Substantially dry starch (containing, *e.g.*, not more than 12% of water) is treated with acetic acid (or with a salt such as calcium hydrogen acetate, which evolves acetic acid on heating) and a small amount of a catalyst, *e.g.*, ammonium persulphate, and heated with agitation to about 163°. The product contains about 13% of unaltered starch; 62% of amylolysis products, including mono- to hexa-acetate of starch, and tri-, tetra-, and hexa-amylose; 20% of sugars, *e.g.*, dextrose, maltose, raffinose, etc.; and about 0.4% of acid. When present in flour (1–20%) it improves the baked products by increasing the volume and preventing "ropiness" (by increasing acidity).
B. FULLMAN.

XVIII.—FERMENTATION INDUSTRIES.

Preservative principles of hops. VIII. Modification of the gravimetric method for the evaluation of hops. J. J. H. HASTINGS and T. K. WALKER (J. Inst. Brew., 1928, 34, 9–13).—10 g. of minced hops are extracted for 3 hrs. in a Soxhlet apparatus with 200 c.c. of ether which should not be heated above 55°. After removal of the ether by distillation, the last portion being removed under diminished pressure, the resinous residue is extracted with successive portions (20 c.c.) of warm (50–60°) methyl alcohol, and the mixed extracts are made up to 100 c.c. and filtered. To determine the α -resin, the α -resin lead salt is precipitated at 60° in 40 c.c. of the filtrate by the addition of a 1% solution of lead acetate in methyl alcohol, an excess of 1–2 c.c. of the lead acetate solution being permissible. After keeping for 30 min., the lead salt is collected on a filter in a tared Gooch crucible, washed, and dried at 100° for 1 hr. Then, weight of α -resin lead salt $\times 15.75 = \%$ of α -resin. The total soft resins are determined by diluting 20 c.c. of the remainder of the methyl alcohol solution with 40 c.c. of water containing a pinch of sodium chloride. The resins are extracted with four successive volumes, each of 50 c.c., of light petroleum (b.p. below 40°), and, after distillation of the petroleum from the combined extracts, the residue is dried at 100° for 1½ hrs. The weight of total soft resins $\times 50 = \%$ of total soft resins. The content of β -resin is obtained by difference from the total soft resins and the α -resin. (Cf. B., 1927, 24.)
C. RANKEN.

Extraction of β -resin in the determination of hop resins. A. H. BURGESS and H. MARTIN (J. Inst. Brew., 1928, 34, 13–15).—Extraction of β -resin from the methyl alcohol solution by light petroleum, although uninfluenced by an excess of sulphuric acid, is affected by the amount of water present. The quantity of

resin extracted increases until the ratio of methyl alcohol to water reaches 20 : 15, after which a decrease takes place. The additional resin extracted after dilution of the methyl alcohol possesses preservative properties.
C. RANKEN.

Yeast cell and yeast cake. N. C. BEETLESTONE (J. Inst. Brew., 1928, 34, 22–38).—The moisture in the yeast cell has been determined experimentally and by calculation. Errors due to the effects of diffusion through the cell walls are negligible and necessitate no correction in the determination of the cell moisture. In the experimental methods, which are modifications of the original "dilution" method, the quantity of drainings present in the yeast mass is ascertained from the amounts of alcohol contained in the yeast mass and the drainings. This value, together with the sp. gr. and the residual gravity of the drainings, and the dry solids of the yeast, permit the calculation of an average moisture content in the yeast cell of 51.6%. Using results published by Tait and Fletcher, the average moisture of the cell is calculated to be 47%, and by adapting the formula devised by Hehner and Richmond for the determination of the total solids of milk, a value of 58.7% is obtained. From the various figures, the moisture of a normal yeast cell is finally chosen as 54%. Yeast cake, however dry it may appear, contains free liquid varying according to the method of separation of the yeast. Under ideal conditions the pressed cake, which consists of undistorted cells arranged in hexagonal packing, contains approximately 74% by vol. of cells and retains in the intracellular space approximately 26% by vol. of the liquid from which the cells have been separated. The cells may be distended as a result of hydrostatic or osmotic pressure.
C. RANKEN.

Water content of yeast cells in suspension in brewery worts. R. H. HOPKINS (J. Inst. Brew., 1928, 34, 39–41).—Assuming that 0.35% is the average quantity of yeast increase (as dry solids) during fermentation, that 60% of those solids consists of nitrogenous and mineral matter, that 1 g. of yeast solids utilises 1.19 g. of maltose by assimilation, and that the differences in degrees of gravity lost, for a definite spirit indication, are proportional to the yeast increase, expressions are deduced for the original sp. gr. of the yeast cells and for the difference between the degrees of gravity lost, for 6° of spirit indication, with worts of 1045° and 1099° original gravity. By this method, it is calculated that the original gravity of the yeast cell is 1270°, and that 0.35 g. of yeast solids is associated with 0.52 g. of water, from which it follows that 60% of water is present in the yeast cell suspended in wort.
C. RANKEN.

Alcohol content of wash-water of carbonic acid from closed fermentation vessels. E. LÜHDER (Z. Spiritusind., 1928, 51, 1–2).—Since the greater part is condensed in the upper part of the fermentation vessel, the amount of alcohol carried over is small. In most cases it is only about 0.4% of the total alcohol produced. The variation with volume of liquid in the fermentation vessel is illustrated by a test in a 10,000 litre vessel, using 3000, 6000, and 9000 litres, when 400 litres of wash-water afterwards contained 0.0, 0.8, and 1.5% of alcohol, respectively.
F. E. DAY.

Oxalic acid and its adsorption compounds in beers. C. GEYS (Woch. Brau., 1928, 45, 1—7).—The association of calcium oxalate with the flocculent sediment of beers is very intimate, probably owing to adsorption of oxalate ions by the colloids. The greater part of the oxalic acid is derived from the malt, and as acidity increases during fermentation the colloids tend to coagulate, calcium oxalate crystals being formed in association with the flocks. Conditions such as warmth, shaking, or presence of gas bubbles, which favour flocculation, in general increase crystal formation, which is inhibited by heating the beer, as the incipient crystals which act as centres of crystallisation are dissolved. Pale beers deposit more oxalate than dark beers, since their colloids are in a higher state of dispersion and hydration and more readily adsorb the oxalate ions. The approximate solubility of calcium oxalate in distilled water, pale wort (12%), pale new beer (12%), and pale finished beer (12%) is given as 6.6, 60, 30, and 20 mg. per litre, respectively.

F. E. DAY.

Identification of lactic acid. GRÜSS.—See III.
Fermentation of stable manure. GLATHE.—See XVI.
Water for brewing. STRELL.—See XXIII.

PATENTS.

Increasing the activity of yeast and activator for yeast. A. W. HIXSON and A. K. BALLS, Assrs. to FLEISCHMANN CO. (U.S.P. 1,651,027, 29.11.27. Appl., 12.12.22).—The yeast prior to incorporation in the dough is stirred for 15 hrs. at 20° with an aqueous solution of sodium stearate containing 6.7×10^{-7} — 4×10^{-5} g.-mol./g. of yeast.

T. S. WHEELER.

Production of acetone and butyl alcohol by fermentation. E. F. PIKE and H. F. SMYTH, Assrs. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,655,435, 10.1.28. Appl., 29.5.23).—A culture of *Clostridium butyricum* is added in the absence of amylaceous materials to a sterilised mash of molasses and water, and the fermentation allowed to proceed to completion.

C. RANKEN.

XIX.—FOODS.

Importance of particle size in manufacture of flour and baking. G. VAN DER LEE (Chem. Weekblad, 1928, 25, 34—40).—Very fine grinding of flour is not practised by reason of the cost, the danger of heating, and the difficulty of sifting. Silk gauze does not give accurate separation according to particle size, the material passing the sieve being much finer than corresponds to the size of the holes. The finer fractions have lower nitrogen content and higher diastatic power; the medium fractions are more suitable for baking than the fine and the coarse, but this is due to the differences in chemical composition rather than to particle size. The differences in suitability for baking are, in any case, small; where an unsuitable flour is found to be finely ground, the overheating in grinding, rather than the fineness attained, is found to be the determining cause.

S. I. LEVY.

Method of humanising cow's or goat's milk. O. FERRIER and V. CHENARD (J. Pharm. Chim., 1927, [viii], 6, 562—563).—To 500 c.c. of cow's or goat's milk diluted with 500 c.c. of boiled water there are added

45 g. of lactose and variable small quantities of tricalcium and magnesium phosphates, disodium hydrogen phosphate, iron glycerophosphate, and sodium fluoride. Cream or fresh butter (25 g.) is added, and sometimes cod-liver oil etc. The mixture is boiled for 15 min. It is efficacious in the case of infants suffering from gastro-enteritis.

B. FULLMAN.

Bromocresol Green as indicator in the manufacture of grain-curd casein. A. G. BENTON (Ind. Eng. Chem., 1928, 20, 15).—Control of hydrogen-ion concentration is essential in the preparation of technical casein, and Methyl Red has been generally used for this purpose. As this indicator is not stable, the substitution of Bromocresol Green, of which standards can be preserved much longer, is suggested.

R. H. GRIFFITH.

Destruction of essence of mustard in its preparations. E. LASAUSSE (J. Pharm. Chim., 1927, [viii], 6, 97—107).—Fresh mustard preparations contain no free sulphur, but a gradual decomposition of the allylthiocarbimide takes place on keeping with liberation of sulphur. This reaction also occurs in aqueous-alcoholic solution in the cold after a few hours, and more rapidly on heating. When determining the essence content of a mustard preparation in the usual way by slowly distilling the aqueous-alcoholic solution into ammoniacal silver nitrate solution, part of the free sulphur present is carried over with the distillate and forms silver sulphide, thus increasing the apparent content of allylthiocarbimide. No liberation of sulphur from the allylthiocarbimide occurs during the actual process of distillation, probably because the bulk of this essence distils over in the early stages, and consequently accurate results are obtainable by this method when applied to freshly made mustard preparations.

F. R. ENNOS.

Necessity for the evaluation of mustard flour. LEBRUN (J. Pharm. Chim., 1927, [viii], 6, 109—110).—Four samples of mustard flour, stated to conform to a standard of 0.70% of allylthiocarbimide, on examination were found to contain 0.37, 0.32, 0.64, and 0.838%.

F. R. ENNOS.

Preservative action in catsup of salt, sugar, benzoate, and acid. C. S. PEDERSON and R. S. BREED (New York Agric. Exp. Sta. Bull., 1926, No. 538, 3—15).—Sugar is ineffective. Sugar (15%) with salt (3.5%) is effective. Acetic acid (1.0%) with salt (5.0%) and sodium benzoate (0.2%) stops the growth of all the bacteria tested.

CHEMICAL ABSTRACTS.

Relation of *d*-gossypol to the toxicity of some cotton-seed products. W. D. GALLUP (Ind. Eng. Chem., 1927, 20, 59—63; cf. Sherwood, B., 1926, 564; Gallup, 1927, 883).—During the heating of cotton-seeds the gossypol is converted into *d*-gossypol, which is insoluble in ether. The extent of the change depends on the temperature, time of heating, and the amount of water present, and has been observed chemically and pharmacologically over a period of 16 hrs. *d*-Gossypol is toxic but less so than gossypol, and the toxicity of the seeds decreases with heating. Continued heating, especially in the presence of excess of water, destroys the *d*-gossypol, finally giving a product free from both forms of gossypol and almost non-toxic. Cottonseed meal

contains only traces of gossypol but relatively large amounts of *d*-gossypol, to which the toxicity of the meal is due. Heating the meal in the presence of water destroys the toxic properties. The insoluble gossypol formed by heating the dry seeds is much more toxic than the insoluble form found in the meal or in seeds which have been steam-heated. The existing chemical methods for the determination of the gossypols are not reliable for measuring the toxicity of cotton-seed products.

E. H. SHARPLES.

Tinplate. KOHMAN and SANBORN.—See X.
Coconut oil in cocoa preparations. HÄRTEL.—See XII.

PATENTS.

Manufacture of artificial sausage skins. J. SAMUEL (U.S.P. 1,656,681, 17.1.28. Appl., 21.9.25. Ger., 7.8.25).—See B.P. 262,202; B., 1927, 171.

Manufacture of thick gravies. E. M. ERSLEV (JØRGENSEN) (B.P. 282,475, 17.5.27).

Mixing apparatus (B.P. 281,904).—See I. **Starch conversion products** (B.P. 282,178).—See XVII.
Increasing activity of yeast (U.S.P. 1,651,027).—See XVIII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Detection of arsenic in [medicinal] iron preparations by means of hypophosphite. G. WALLRABE (Pharm. Zentr., 1928, 69, 33—37).—The employment of Thiele's reagent, obtained by treatment of sodium hypophosphite with concentrated hydrochloric acid, in place of stannous chloride in the detection of arsenic in medicinal preparations of iron compounds, as recommended by the new German pharmacopœia, is found to lead to less accurate results, and the new test is described as definitely less sensitive. Different results are obtained with different iron preparations; this is traced back to the presence of varying proportions of ferrous and ferric compounds, and it is established that ferric compounds are not completely reduced, even by large excess of Thiele's reagent. The addition of stannous chloride or of potassium iodide increases the sensitiveness of the test.

S. I. LEVY.

Chemical characteristics of drugs. Behaviour of agar towards iodine. L. ROSENTHALER (Pharm. Ztg., 1928, 73, 77—78).—A series of experiments on the reaction of agar in aqueous suspension towards iodine is described. The colour produced is dependent on the amount and concentration of iodine solution, the age, temperature of heating, temperature of the agar solution after cooling, and the method of addition of the iodine solution. The following method is suggested: 0.1 g. of powdered agar and 50 c.c. of water are boiled for 5 min.; 10 c.c. of this liquid are rapidly cooled (under 3 min.) to 15°, and 1 c.c. of 0.05*N*-iodine solution is added all at once; a bluish-violet colour is formed. The remainder of the solution is cooled to 15° and, after 1 hr., 10 c.c. are mixed with 1 c.c. of the iodine solution; no coloration should be formed.

E. H. SHARPLES.

Bactericidal power of some preparations of iodine. P. SUIFFET (J. Pharm. Chim., 1927, [viii], 6, 398—401).—The bactericidal power of various iodine solutions, ointments, etc. is determined by the time taken to kill *B. pyocyaneus*, *B. coli*, staphylococci, and streptococci. The iodine content of the preparations was 0.005%, but of the oils and ointments 0.1%. *B. coli* is the most vulnerable, followed in order by streptococci, staphylococci, and *B. pyocyaneus*. Aqueous iodine has the highest bactericidal power, followed by iodine in potassium iodide. Then follow in order tincture of iodine with water, and iodinised preparations of gum, tannin, starch, agar-agar, glycerol, and gelatin. The power of preparations of olive oil, vaseline, etc. depends on their iodine content.

B. FULLMAN.

Titration of ipecacuanha extract. E. LÉGER (J. Pharm. Chim., 1927, [viii], 6, 501—505).—Ipecacuanha extract is dissolved in water, treated with powdered pumice, and the mixture evaporated to dryness and powdered. The powder is digested with alcoholic ammonia solution, ether is added, and the digestion continued. The liquid is filtered (the volume *V* of the filtrate being noted) and distilled, and the residue dissolved in acidified water, precipitated by ammonia, and extracted with chloroform. The residue left on distillation of the latter is purified by repeated treatment with ether and distillation. The residue is dissolved in alcohol, water is added, and the liquid titrated with 0.1*N*-sulphuric acid (hæmatoxylin as indicator). From the quantity of acid used and the factor *V* the percentage of alkaloids is calculated. Alternatively, the residue is dissolved in 0.1*N*-acid and titrated with potassium hydroxide (Methyl Red). Determinations should be referred to the dry extract, which should contain 8—10% of alkaloids.

B. FULLMAN.

Insolubility of adrenaline in oils. F. MARTIN (J. Pharm. Chim., 1927, [viii] 6, 248—250).—Adrenaline base is insoluble in vaseline and in olive and castor oils. A stable, homogeneous emulsion of adrenaline hydrochloride suitable for therapeutic use may be prepared by dissolving 1 g. of adrenaline in 10 g. of alcohol with the aid of the smallest possible quantity of hydrochloric acid, adding the solution to 100 g. of castor oil, and making the mixture up to 1 litre by the addition of olive oil.

E. H. SHARPLES.

ψ-Ephedrine from *Ephedra alata*. O. F. BLACK and J. W. KELLY (Amer. J. Pharm., 1927, 99, 748—751).—ψ-Ephedrine was obtained in about 1% yield by extracting the dried twigs of *E. alata*, from Morocco, with chloroform.

S. COFFEY.

Production of ergot extract. RAPP and R. LECHLER, JUN. (Pharm. Ztg., 1928, 73, 76—77).—From experiments with pure ergotamine it is shown that, in the neutralisation of the alcoholic extract previous to extraction of the alkaloids, if a *p_H* value exceeding 7.7 is attained there is a large loss of ergotamine. Neutralisation to *p_H* 8.0 caused a loss of 18% of the alkaloid. Thus litmus and phenolphthalein are not satisfactory indicators for the neutralisation, and the authors suggest the use of a Folien colorimeter. For the extraction of ergot, a

modified percolation method, which is shorter, gives a higher yield, and decreases the volume of solvent necessary, is described.

E. H. SHARPLES.

Terpeneless lemon oil. W. G. DALTON (Perf. Ess. Oil Rec., 1928, 19, 7—8).—From the examination of a large number of genuine terpeneless lemon oils, the following limits for the physical and chemical constants are given; $n_D^{15.5}$ 0.8935—0.8980, n_D^{20} 1.4807—1.4820, α -3° to -8° , citral 42—52%, esters 18—22%; soluble in 80% alcohol in all proportions. The aldehyde/ester ratio in all the samples examined was roughly 5 : 2, and all the oils were soluble in 4—5 vols. of 78% alcohol, but in no case was any sample soluble in 75% alcohol. The addition of citral from lemongrass oil together with an appropriate amount of linalyl acetate rendered the oil soluble in 75% alcohol, even when the original oil contained a comparatively low percentage of aldehydes. The values given by four commercial oils, all of which contained lemongrass citral, did not agree with the above specification. The significance and detection of added lemongrass citral in terpeneless lemon oils is discussed.

E. H. SHARPLES.

Lignite generator tar and oil of camomile. RUHEMANN and LEWY.—See II.

Determination of sodium borate. FRANÇOIS and SEGUIN.—See VII.

PATENTS.

Manufacture of compounds [salts] of acetamidosalicylic acid with aliphatic amines. G. W. RAIZISS and A. PROSKOURIAKOFF, Assrs. to ABBOTT LABORATORIES (U.S.P. 1,652,796, 13.12.27. Appl., 15.10.25).—The acetamidosalicylic acid, e.g., 5-acetamidosalicylic acid, is dissolved in an alcoholic solution of the amine, e.g., *n*-propylamine or hexamethylenetetramine, and the salt is precipitated with ether.

T. S. WHEELER.

Manufacture of cyclohexylresorcinol. M. PHILLIPS (U.S.P. 1,650,036, 22.11.27. Appl., 28.4.26).—A cyclohexane derivative containing a negative substituent, e.g., cyclohexanol, is heated with resorcinol at 150° in presence of a condensing agent, e.g., zinc chloride, to give cyclohexylresorcinol, m.p. 124° .

T. S. WHEELER.

Manufacture of physiologically-active substances from female internal secretive organs. M. HARTMANN, Assr. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,651,770, 6.12.27. Appl., 22.1.27. Switz., 2.2.26).—See B.P. 265,567; B., 1928, 107.

Electrode for therapeutic application (U.S.P. 1,655,783).—See XI.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

PATENTS.

Obtaining photographic images [light-sensitive diazo compounds]. E. GAY (B.P. 282,894, 30.9.26).—The diazo compounds of ethers of *o*-aminophenols or *o*-aminonaphthols are suitable for production of light-sensitive layers on paper etc. Examples are: *o*-anisidine (developed with 2 : 4-tolylenediamine for orange-brown),

o-phenetidine (developed with phloroglucinol for deep brown), cresidine (developed with 2-amino-8-naphthol-3 : 6-disulphonic acid for purple-brown), and, in particular, 2-aminoquinol dimethyl ether [2 : 5-dimethoxyaniline] (developed with 1-amino-8-naphthol-3 : 6-disulphonic acid for violet).

C. HOLLINS.

Photographic and etching process and product. M. C. BEEBE, Assr. to WADSWORTH WATCH CASE CO. (U.S.P. 1,655,127, 3.1.28. Appl., 28.8.25).—A dyed substratum for applying between the sensitive coating and the metal plate consists of a solution of about 35 g. of dye in about 1300 g. of alcohol containing a slow-drying constituent.

W. CLARK.

Reduction of photographic silver image layers. P. PLAGWITZ, Assr. to E. TAESCHNER CHEM.-PHARM. FABR. (U.S.P. 1,656,235, 17.1.28. Appl., 6.5.26).—See B.P. 260,892; B., 1927, 61.

[Production of offset printing plates for] photoengraving processes. DEPTH-O-TONE CORP., Asses. of F. STOCKINGER (B.P. 271,468, 16.5.27. U.S., 18.5.26).

Carbon pigment (U.S.P. 1,651,733).—See XIII.

XXII.—EXPLOSIVES; MATCHES.

PATENTS.

[Permitted] explosive. H. H. CHAMPNEY, Assr. to HERCULES POWDER CO. (U.S.P. 1,650,186, 22.11.27. Appl., 8.5.25).—A mixture of nitroglycerin, ammonium nitrate, and ground oat husks is claimed.

T. S. WHEELER.

Production of a cartridge for use with liquid oxygen. C. MOTT, Assr. to PUROX CO. (U.S.P. 1,650,766, 29.11.27. Appl., 13.8.23).—Dehydrated molasses mixed with powdered charcoal (1—3 pts.) is employed.

T. S. WHEELER.

XXIII.—SANITATION; WATER PURIFICATION.

Testing a carbon monoxide gas-mask. F. HEINRICH and E. PETZOLD (Chem. Fabr., 1928, 19—22).—The Degea gas-mask for use in atmospheres containing carbon monoxide consists essentially of a filter impregnated with a catalyst, which promotes the oxidation of the carbon monoxide to dioxide by the oxygen in the air, and is provided with an indicator which liberates acetylene as a warning when the efficiency of the filter begins to be impaired. In air containing 1.5% CO the life of the filter is about 40 min., and in air containing 4.4% about 10 min. To test the efficiency of the mask the gases are passed through at a definite velocity, thence through potassium mercuric iodide to remove acetylene, concentrated sulphuric acid to absorb hydrocarbons, calcium chloride and phosphorus pentoxide to remove moisture, and soda-lime and phosphorus pentoxide to absorb carbon dioxide, and finally over copper oxide at 300° , any carbon dioxide formed being absorbed in soda-lime after drying in the usual way. For qualitative detection of minute traces of carbon monoxide the blood test gives the most dependable

results; the ammoniacal silver solution and palladous chloride tests are uncertain in the presence of hydrogen, acetylene, and carbon dioxide. A. R. POWELL.

Germicidal efficiency of sodium hydroxide and sodium hydroxide-carbonate mixtures at the same hydrogen-ion concentration. M. LEVINE, E. E. PETERSON, and J. H. BUCHANAN (Ind. Eng. Chem., 1928, 40, 63—65; cf. B., 1928, 70).—The germicidal efficiency of different solutions was measured by the time required to kill 99.9% of the spores of a given bacterium. At p_H 13.0—13.2 the presence of sodium carbonate increased the efficiency, but at p_H 13.2—13.4 it diminished it. The p_H value is, therefore, not a measure of germicidal efficiency, neither is the alkalinity by titration with Methyl Orange. The difficulty of the accurate determination of p_H value at high alkalinity is pointed out, and it is suggested that the determining factor in germicidal action may be undissociated sodium hydroxide. C. IRWIN.

Scientific sewage disposal at Milwaukee. R. CRAMER and J. A. WILSON (Ind. Eng. Chem., 1928, 20, 4—9).—A brief description of this activated-sludge plant (diffused air system) and its operation, including sludge disposal, is followed by a discussion of the mechanism of the process, which is claimed to be essentially non-bacterial. A comparison of the various reagents used for "conditioning" the sludge prior to filtration indicates the superiority of ferric chloride, reasons for which are discussed. The lines of present and future research are indicated. C. JEPSON.

Odours and sewage sludge digestion. I. Effect of sea water on hydrogen sulphide production. W. RUDOLFS and P. J. A. ZELLER (Ind. Eng. Chem., 1928, 20, 48—50).—Sea water is shown to retard but not prevent the digestion of sewage sludge. The presence of hydrogen sulphide in the gaseous products is due to the utilisation of sulphates present in sea water, though less than 6% of the sulphur utilised was recovered in this form. C. JEPSON.

Coagulation studies [in water purification] at the Washington suburban sanitary district. R. B. MORSE, C. A. HECHMER, and S. T. POWELL (Ind. Eng. Chem., 1928, 20, 56—59).—The water supply is treated at two rapid sand-filtration plants, one at Hyattsville and the other at Burnt Mills. At the former it was found that with a constant addition of 0.2 grain of sodium aluminate per gallon the alum, lime, and soda ash to be materially reduced with a slight saving in cost but a manifest improvement in the quality of water supplied. At Burnt Mills, which is the larger of the two plants, the p_H value of the raw water is usually higher, and apparently little advantage is to be gained by the use of sodium aluminate except at times of high turbidity. C. JEPSON.

Effect of salts on rate of coagulation and optimum precipitation of alum floc [in water purification]. B. H. PETERSON and E. BARTOW (Ind. Eng. Chem., 1928, 20, 51—55).—The salt content of the water to be treated is more of a controlling factor governing floc production than the hydrogen-ion concentration. Sul-

phates reduce the time required to produce the alum floc and extend the p_H range over which this may occur. Oxalates and phosphates probably form complex compounds which have properties different from the true floc. C. JEPSON.

Biological purification of water for brewing. M. STRELL (Woch. Brau., 1927, 44, 607—613).—The micro-organisms can be removed by filtration or destroyed by chemical or electrochemical methods. Restricted supplies of water may be purified by Berkefeld, Bühring, Delphin, Chamberland, Sukro, or Seitz filters, which differ only in the composition of the filter material. The absolute removal of the bacteria cannot be guaranteed, although the pores in such filters, which vary between 0.41 μ and 0.16 μ in diameter, are smaller than the bacteria. Sand filters are used for greater volumes of water. The removal of the bacteria is effected in the uppermost sand layer and in the filter film, which forms during filtration from animal, plant, and mineral debris. Bacteria smaller than 2 μ —0.5 μ would not be kept back but for the filter film. To ensure absolute purity, two filters in series are used, or the water is sterilised by chemical means. In the chemical method part of the water is treated with chlorine gas or ozonised air, and subsequently mixed with the remainder of the water. A treatment of 0.2—0.3 g. of chlorine per m.³ of water is sufficient. The apparatus for purification of the water by electro-osmosis is built up of a series of cells, each divided into three compartments by porous diaphragms, the electrodes being hung in the outer compartments. The water flowing through the inner compartments is electrolysed, and the ions derived from the salts in solution pass through the diaphragms into the outer compartments, with the result that not only are the bacteria destroyed by the nascent ions, but the hardness may also be wholly or partly removed from the water. From 50 to 100 litres per hour of water free from bacteria and salts can be obtained from a water giving 400 g. of residue per m.³, and having a hardness of 12.4° by means of a current of 30 kw.-hrs. C. RANKEN.

Milorganite. KADISH.—See XVI. **Bactericidal power of iodine preparations.** SUIFFET.—See XX.

PATENTS.

Refuse-consuming furnaces. WOODALL-DUCKHAM (1920), LTD., E. W. SMITH, and J. W. REBER (B.P. 282,262, 13.4.27).—The refuse is burnt in a shaft furnace (with blast), which increases in diameter upwards, and the material is fed through a sleeve which can be lowered more or less into the shaft, thus regulating the upper level of the refuse. B. M. VENABLES.

Purification and softening of water. W. PATERSON (B.P. 282,165, 17.9.26).—The water is treated with an excess of caustic lime and then carbonated before passing through a zeolite bed, by adding carbon dioxide directly or as a solution of predetermined strength. The deleterious effect on zeolites of caustic lime and fine particles of calcium carbonate is thus avoided. W. G. CAREY.