

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MAY 25, 1928.

I.—GENERAL; PLANT; MACHINERY.

Heat transfer for oils and water in pipes. F. H. MORRIS and W. G. WHITMAN (Ind. Eng. Chem., 1928, 20, 234—240).—Film coefficients of heat transfer in a jacketed pipe were determined for the heating and cooling of water and more particularly of petroleum oils with a view to the rational design of pipe stills and coolers. The rate of flow was 1—20 ft./sec., flow being turbulent. The general equation for the heat-transfer coefficient, h , is $hD/k = \phi(Du\rho/z) \times \psi(cz/k)$, where D is the pipe diameter, k the conductivity, u the velocity, ρ the density, c the specific heat, and z the viscosity of the liquid; ϕ and ψ represent experimentally determined functions. The physical properties were taken at the temperature of the main body of the liquid, more concordant results being so obtained. The pipe used was long enough to minimise "end effects." The relationship between the three variables in the above equation was determined by maintaining one constant and plotting the relation between the other two graphically. Curves are thus obtained which provide data suitable for design, but the values of $\phi(Du\rho/z)$ for cooling are only about 75% of those for heating, necessitating the use of separate curves for the two cases. C. IRWIN.

Computation methods in counter-current absorption systems. W. K. LEWIS and W. H. McADAMS (Ind. Eng. Chem., 1928, 20, 253—257).—If a gas is being treated for the removal of a solute by a liquid in a column and operation is uniform $G(y - y_0) = L(x - x_0)$, when G , L are the rates of flow of gas and liquid, and y , y_0 the concentration of solute in the gas at any point and at the top, and x , x_0 concentrations in the liquid. This (linear) relationship is plotted for comparison with equilibrium curves for the systems considered (the drying of air by sulphuric acid and the scrubbing of gas for benzol). The equilibrium curve itself represents conditions at the interface between the two phases, and the rate of transfer of material is proportional to the surface of contact and the concentration gradient through each of the two films. From these facts a further series of equations is derived which are simplified if one or other film controls the transfer. These equations are integrated graphically. In the case of the system inert gas—benzene—absorbing oil Raoult's law is followed and $y = \rho x [\pi - (\rho - \pi)x]$, where ρ is the pressure of pure benzene and π the total pressure. The various diagrams are discussed in detail. C. IRWIN.

Universal tank-calibration chart. A. K. DOOLITTLE (Ind. Eng. Chem., 1928, 20, 322—323).—A chart was prepared to calibrate boiler tanks with bulged ends, whether horizontal or vertical. Two sets of curves were plotted on logarithmic paper to the equations $A =$

$\int_0^h 2x \cdot dy$; $x = \sqrt{r^2 - (y - r)^2}$; and $v = 7.4805 AL$ measurements being in inches (depth), feet (length), and gallons (volume). The abscissæ common to both sets represent the area of the segmental cross-section of liquid. A further separate set of curves gives the correction for the ends, which are treated as segments of a sphere of radius equal to the tank diameter. For simplicity the volume was integrated as an ellipsoid of revolution $v = (2 - \sqrt{3})/3 \times \pi h^2(3r - h)$, and a correction of 0.767 applied to bring the volumes so obtained to the true ones. C. IRWIN.

Boiler scale and its prevention. H. WALDE (Wiss. Veröff. Siemens-Konz., 1927, 6, 151—170).—A method of presenting the results of analyses of boiler feed-waters and of boiler scales has been elaborated in order to classify the various types of incrustation and bring them into relation with the feed water. Three ratios are calculated for the water, and three for the deposit—the ratios of silica to lime and of magnesia to lime for both, the ratio of sulphate hardness to carbonate hardness for the water, and the lime as sulphate to the lime as carbonate for the deposit. The significance of these ratios is discussed, and a classification of the different types of scale and sludge suggested. The chief types of scale are the gypsum and calcium carbonate scales, and mixtures of these, and the silica type. The sludges contain oxides of iron and aluminium, generally also silica and magnesia, and often organic matter. The various methods of preventing scale are examined, and the Cumberland electrolytic method more particularly discussed and recommended. S. I. LEVY.

Tentative standards [of specification and practice]. AMER. SOC. TESTING MATERIALS (Tentative standards, 1927, 824 pp.).—Tentative and revised specifications, tentative rules, methods of testing, and recommended practices are given for a number of commercial materials.

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[Standard specifications for] non-metallic materials. AMER. SOC. TESTING MATERIALS (Part II, Non-metallic materials, 1927, 1000 pp.).—Standard specifications, methods of testing, and definitions are given.

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PATENTS.

Heat exchangers. SUPERHEATER Co., Assees. of W. H. ARMACOST (B.P. 286,682, 24.6.27. U.S., 9.3.27).—A number of standardised parts—tubular elements with circular ends in standard headers—are constructed so as to be suitable for varying conditions. The tubular elements in the intermediate part of their length are given a pseudo-oval section by branching them into two parallel circular tubes, capable of resisting considerable internal pressure. The elements may be rotated in the

headers so that the long axis of the cross-section is more or less athwart the stream of the other fluid.

B. M. VENABLES.

Heat-exchange apparatus. C. F. BRAUN (U.S.P. 1,662,143, 13.3.28. Appl., 28.3.23).—A series of horizontal tubes gives communication between chambers situated at the front and rear of a casing. These chambers are provided with partitions so that a fluid may flow backwards and forwards through the tubes between compartments in the front and rear chambers. A second fluid flows in at one end of the casing, passes around the tubes in the space between the front and rear chambers, and leaves at the opposite end. Means are interposed between groups of tubes for alternately increasing and then abruptly decreasing the velocity of the second fluid.

F. G. CLARKE.

Apparatus for the heating and cooling of fluids. C. W. STANCLIFFE (B.P. 286,757, 12.11.26 and 20.7.27).—In a heat exchanger mechanical means are provided to disturb the stationary film on the wall of the exchanger. *E.g.*, in a gear-wheel pump the gears themselves may be heated and idler gears provided to increase the squeezing action. In a tubular form of apparatus rotary brushes may wipe the surfaces of the tubes. B. M. VENABLES.

[Water-jacket for] shaft furnaces, gas producers, etc. STETTNER CHAMOTTE-FABR. A.-G., and E. TERRES (B.P. 284,639, 1.3.27. Ger., 2.2.27).—The water-jacket is formed at the lower part by a corrugated casing and at the upper part by an enlarged dome or head, and the three parts—combustion shaft, upper and lower jackets—are easily separable. B. M. VENABLES.

Grinding mills. L. MCG. FRASER (B.P. 287,318, 12.3.27).—A grinding pan or mill for use with volatile materials is provided with a vapour-tight cover vented only through a liquid seal or condenser; the latter device may be arranged to return condensed liquid into the mill. A closed cylinder and piston is used to withdraw the ground material and has two outlets—one for returning the material if not fine enough, the other for final discharge. B. M. VENABLES.

Centrifugal grinding mill. W. A. GIBSON and J. G. BURKE, Assrs. to BRADLEY PULVERIZER CO. (U.S.P. 1,663,881, 27.3.28. Appl., 22.6.25).—A mill having a horizontal annular grinding ring, co-operating centrifugally-actuated grinding members, and a superposed air-separating chamber has selectively operated, adjustable means for admitting air above and adjacent to the grinding ring, whereby the action of the air separator is regulated by admitting the air in predetermined directions relatively to the radius of the ring.

F. G. CLARKE.

Agitators or mixers. F. EPSTEIN (B.P. 279,127, 17.10.27. Belg., 16.10.26).—The apparatus is provided with loosely-slung baffles, counterweighted so that they can be caused to remain at the surface of the liquid or be submerged to any desired extent.

B. M. VENABLES.

Separation of subdivided materials. E. C. R. MARKS. From SUSQUEHANNA COLLIERIES CORP. (B.P. 286,951, 7.7.27).—The material is allowed to enter a circular chamber in which a fluid is flowing with vortical motion of sufficient violence to keep the stratified bed

of solids in rotary motion. The lighter material (and fluid) is drawn off through a central rising pipe, whilst the heavier passes out through the bottom to another separator if desired.

B. M. VENABLES.

Separation of intermixed divided materials of different specific gravities. R. PEALE, W. S. DAVIES, and W. S. WALLACE (B.P. 287,184, 13.9.26).—A shaking table is provided with a pervious bed through which air is blown upwards, to cause stratification. One of the strata—usually the heavier—is discharged by inclined guides over each long edge, and the other stratum proceeds straight on towards the end and is collected at a central opening.

B. M. VENABLES.

Drying of disintegrated material. O. SODERLUND, and TECHNO-CHEMICAL LABORATORIES, LTD. (B.P. 286,743, 7.10.26).—The air-borne material is passed up and down through a number of heated tubes or conduits, spreading and uniform distribution of the material being effected by changing the section of the conduits. Heating is preferably effected by a liquid and its vapour, *e.g.*, water and steam.

B. M. VENABLES.

Centrifugal apparatus for the treatment of sludge. E. C. ELSMORE, H. J. HOLFORD, and A. BISHOP (B.P. 286,855, 10.2. and 27.5.27).—The sludge is fed to the interior of a horizontal rotating drum lined with gauze or other filter medium; when a considerable amount of solid matter has accumulated, the rotation is stopped and a circular scraper with flexible edge is moved from end to end of the drum to discharge the solids through one end.

B. M. VENABLES.

Mixing of gases and liquids. E. PLAYER, and PARC ENGINEERING CO., LTD. (B.P. 286,812, 23.12.26 and 23.2.27).—The gas conduit is interrupted by a gap which is surrounded by a closed vessel containing the liquid. Dipping in the pool of liquid are one or more rotating toothed wheels which are spun by impingement of the current of gas and spray a quantity of the liquid into the gas stream.

B. M. VENABLES.

Apparatus for separating liquid containing a dispersed phase of another liquid. E. W. GARD, B. G. ALDRIDGE, and H. J. MULTER (U.S.P. 1,665,164, 3.4.28. Appl., 16.11.26).—One liquid dispersed in another is separated by passing the combined liquids through a succession of foraminous plates supported by grids which divide the separating chamber into sections. The combined liquids enter at one end of the chamber, and the liquid which passes more readily through the plates passes through all the latter and leaves through an outlet at the opposite end, whilst the other liquid leaves the chamber by one or other of the outlets placed between the grids.

F. G. CLARKE.

Centrifugal thickening of mixtures and clarifying of liquids. H. M. CHANCE (U.S.P. 1,664,769, 3.4.28. Appl., 29.7.25).—The fluid mixture of liquid and comminuted solids passes through the perforated walls of a drum rotating at high speed. The solids proceed into a region of relatively slow rotation between the drum and a casing, whilst the liquid moves towards the centre of rotation. Clarified or partly clarified liquid may thus be withdrawn from the drum and the

thickened mixture from the region between the drum and the casing. F. G. CLARKE.

Preparation of filtering materials. F. H. WEIL, ASSR. to SEITZ-WERKE G.M.B.H. (U.S.P. 1,664,275, 27.3.28. Appl., 24.2.27. Ger., 22.5.24).—Natural asbestos is treated under pressure with a non-reducing gas which unites with the impurities in the asbestos forming compounds having no action on the liquids to be filtered. F. G. CLARKE.

Evaporating apparatus. AKTIEBOLAGET SVENSKA MASKINVERKEN, and E. G. ERIKSSON (B.P. 286,982, 6.9.27).—The liquid to be evaporated flows from end to end in the lower part of a fixed horizontal drum, within which is an inner, rotating, heating drum provided with disc-like projections. Within the discs the heating medium (steam) is caused to flow in spiral passages, or the discs may be formed as spiral steam coils. The steam is supplied to the larger part of the inner drum, which is provided with individual inlets to each spiral coil, and the outlets for condensate from the coils communicate with an outlet conduit co-axial with the steam drum. At the bottom of the fixed drum are screens upstanding between the heating discs and a conveyor for slime. B. M. VENABLES.

Bag filters. MASCHINENFABR. BETH A.-G. (B.P. 281,994, 27.6.27. Ger., 11.12.26).—The filter bags are divided into groups, and two fans are mounted on a common shaft, one for drawing the main stream of gas through the filter, the other for forcing reverse-current cleaning air through one group only (at a time) of the filter. The dust dislodged from this group falls into a conveyor and the air joins the main stream and therefore passes through the other groups in the filtering direction. B. M. VENABLES.

Purification of furnace gases. C. V. A. ELEY (B.P. 285,544, 15.11.26).—The discharge of smoke or noxious gases from furnaces is reduced or eliminated by passing the gases successively through a filter chamber and a tank. The filter chamber may be provided with trays containing chemical reagents for purifying the gases, or may contain a spray for supplying water or other liquid for the same purpose. The gases enter the tank below the level of the liquid and may be made to pass below a baffle plate attached to the inlet pipe before passing up through the liquid and escaping. The tank is provided with a sump containing a helical conveyor for the removal of deposited solid matter, which passes through a valve into a chamber from which it can be removed from time to time. A. B. MANNING.

Gas-cleaning apparatus. C. G. HAWLEY, ASSR. to CENTRIFIX CORP. (U.S.P. 1,663,597, 27.3.28. Appl., 4.5.25).—The gas passes from a surrounding casing into an annular tuyère consisting of vertical blades set tangentially between an upper and a lower ring member so that the overlap is adjustable. Communicating with the tuyère through the top of the casing is a conduit for the gas, and immediately below the tuyère is a central disc which deflects the dust into a receiver placed below. F. G. CLARKE.

Extraction of extraneous material from fluids or gases. J. GORDON & Co., LTD. From HAGAN CORP. (B.P. 286,853, 9.2.27).—A form of apparatus

without moving parts suitable for separating dry steam from wet steam is described. B. M. VENABLES.

Fire-extinguishing composition. AMDYCO CORP., Assees. of F. L. DUNLAP and N. T. EWER (B.P. 269,879, 1.4.27. U.S., 21.4.26).—A composition which reacts in the presence of water to produce a stable foam comprises aluminium sulphate containing not more than 14 mols. of water of crystallisation, sodium bicarbonate, a stabiliser, and an inert filler, the ingredients being ground to pass 40-mesh. The proportion of water of crystallisation in the aluminium sulphate may be reduced to 7 mols., and the weight of the inert material increased proportionately. L. A. COLES.

Manufacture of ice crystals. S. C. CARNEY, ASSR. to ROXANA PETROLEUM CORP. (U.S.P. 1,664,850, 3.4.28. Appl., 4.2.26).—Separated quantities of ice are passed through gaseous carbon dioxide under pressure and then moulded by being pressed together while impregnated with the gas. H. ROYAL-DAWSON.

Stabilisation of chemical substances. H. G. GRIMM (U.S.P. 1,664,678, 3.4.28. Appl., 29.4.24. Ger., 5.5.23).—See B.P. 240,884; B., 1925, 977.

Heat interchangers. A. E. LEEK (B.P. 270,250, 10.1.27. Ger., 1.5.26).

Separation of granular materials (B.P. 287,262).—See II. **Sublimation** (U.S.P. 1,662,056).—See III. **Drying ammonium salts etc.** (B.P. 277,652).—See VII. **Tunnel kiln** (U.S.P. 1,662,063).—See VIII. **Filling cracks in brickwork** (B.P. 286,933).—See IX. **Mixing machine** (B.P. 287,016).—See X.

II.—FUEL; GAS; TAR; MINERAL OILS.

Pressure extraction of coal with tetralin. E. BERL and H. SCHILDWÄCHTER (Brennstoff-Chem., 1928, 9, 105—113).—Two bituminous coals have been extracted with tetralin under pressure at 250° and the extracts examined. Carbonisation of the residue yielded only a small quantity of tar, showing that the constituents of the coal soluble in tetralin were the main source of the tar obtained on carbonisation. One extract, representing 20.3% of a "flaming gas coal," was treated with ether, which dissolved 54.9%, and the soluble fraction was separated into acidic and basic constituents (1.55% and 0.023%, respectively, of the total extract), asphalts precipitated by light petroleum (8.87%), and neutral oils (31.1%); the remainder of the soluble fraction consisted of solids precipitated by the acid and alkali used for extraction. Separation of the neutral oils by the Edeleanu process showed them to consist of about 20% of saturated and 80% of unsaturated hydrocarbons. From the saturated fraction solid paraffins of composition $C_{22}H_{46}$, $C_{24}H_{50}$, and $C_{30}H_{62}$ (or possibly $C_{30}H_{60}$) were separated in crystalline form. The unsaturated fraction contained hydrocarbons of the series C_nH_{2n-4} to C_nH_{2n-18} , the value of n ranging from 12 to 26. The asphalts precipitated by light petroleum were probably of aromatic nature; they could be nitrated, were oxidised to acids by alkaline permanganate, and gave Nastjukov's reaction. The extract obtained from the other coal amounted to 17% and behaved very similarly on examination, although a slightly different procedure

was adopted, the ether extraction being preceded by a separation into saturated and unsaturated constituents by treatment with liquid sulphur dioxide.

A. B. MANNING.

Dry distillation of lignin obtained from beech, oak, and birch. G. SZÉLENYI and A. GÖMÖRY (*Brennstoff-Chem.*, 1928, 9, 73—77).—Lignin prepared from these woods by Kürschner's process (cf. B., 1925, 912), and purified by a second hydrolysis of 24 hrs., has a methoxy-content (Zeisel) of 14.5—14.86%, 14.94—15.65%, and 14.20—14.65% for beech, oak, and birch, respectively, the yield of lignin in each case amounting to 18—19%. On dry distillation the three lignins afford qualitatively the same products as the distillation of lignin from aspen or pine wood (cf. Heuser and Skiöldebrand, B., 1919, 215 A; Heuser and Brötz, B., 1925, 585). The yield of acetic acid is practically the same in all cases, but the yield of methyl alcohol is about 66% higher with oak- and beech-lignin and 14% higher with birch-lignin than with lignin from the pine or ash. The yields of tar are lower, beech-lignin giving only 30%, oak 23%, and birch-lignin only 40% of that obtained from pine-wood lignin. Lignins from pine- and beech-wood give practically the same yield of acetone, slightly higher yields being obtained in the case of lignin derived from the oak, beech, or aspen. Only 10.15% of the original methoxy-content of the lignin is found in the liquid products in the case of beech- and oak-lignin, and only 7.9% in the case of birch-lignin. The yield of gaseous products was approximately the same in all three cases. Oak- and beech-lignin afford 53% of coke, birch-lignin 51%, these yields being about 15% higher than that obtained from pine-lignin.

R. BRIGHTMAN.

Carbonisation experiments at the Jena gas-works. GÜLICH (*Gas- u. Wasserfach*, 1928, 71, 8—10).—The formation of a layer difficultly permeable to gas at the top of the column of coal in a vertical retort, which leads to loss due to increased gas pressure in the lower part of the retort, is avoided by the use of a layer of coke on the top of the coal. Experiments on the maintenance of a gas of constant calorific value by control of the steaming and the amount of suction are briefly described.

A. B. MANNING.

Simultaneous removal of ammonia and sulphur compounds from carbonisation gas. H. BÄHR (*Gas- u. Wasserfach*, 1928, 71, 169—173, 204—210).—The recovery, from low-temperature carbonisation or coke-oven gases, of ammonia and sulphur compounds by the Feld and the Burkheiser processes is discussed, and a new process, which has been subjected to laboratory and semi-technical tests, is described. The gas from the retorts is first cooled to 20—25°, and then subjected to electrical de-tarring. The tar is separated from the condensate, which then contains most of the ammonia and hydrogen sulphide. The liquor is distilled, and the ammonia and hydrogen sulphide evolved are passed back into the de-tarred gas. About 5—6% of air is admitted, and the mixture passed over a catalyst (unspecified), whereby the hydrogen sulphide is oxidised to sulphur dioxide. By a proper arrangement of heat exchangers the oxidation reaction may be made

autothermic, the gases requiring to be heated before commencing the operation. The resulting gas is cooled, when most of the sulphur dioxide reacts with ammonia and is recovered as ammonium sulphite, the last traces of which are removed by electrical precipitation. Any excess ammonia is recovered from coolers as ammonia liquor, and is of sufficient purity to be used in an ammonia oxidation plant. Alternative methods for treating the ammonium sulphite are discussed, but in general it is proposed to volatilise it with air and then to pass the mixture through an ammonia oxidation converter, the resulting oxides of nitrogen and sulphur being removed in condensers as nitrosylsulphuric acid (subsequently converted into mixed nitric and sulphuric acids) or passed for recovery to a form of lead chamber sulphuric acid plant. A flow-sheet with estimates of capital and manufacturing costs for a plant to treat the gas from the distillation of 400 tons of coal per day is given, and it is claimed that the new method of treatment is 25—50% cheaper than the older ones.

A. E. MITCHELL.

Dry purification [of gas]. G. OFFE (*Gas- u. Wasserfach*, 1928, 71, 222—224).—When fresh bog-ore is used for purification of gas the material often forms large lumps and eventually sets up considerable back-pressure. This agglomeration is due to the high activity of some parts of the oxide, and in these places the temperature will rise and the evaporation of water will be more pronounced; the same effect is produced owing to the impossibility of filling the boxes so that the gas passes evenly throughout the purifying mass. Fresh oxide is not so useful in removing cyanogen from the gas as some which has been partly fouled; this is due to its low content of ferrous iron, which is necessary in the formation of Berlin-blue. A mixture of 3 pts. of fresh bog-ore with 1 pt. of oxide from boxes which have been in use has been found satisfactory for the removal of hydrogen sulphide and cyanogen compounds.

R. H. GRIFFITH.

Reduction of carbon monoxide and dioxide with hydrogen in the hot-cold tube at ordinary and high pressure. F. FISCHER and (Erhn.) VON WANGENHEIM (*Brennstoff-Chem.*, 1928, 9, 94—97).—Results previously obtained by Fischer and Jaeger (B., 1926, 777) have been confirmed with various binary and ternary mixtures of carbon monoxide, carbon dioxide, and hydrogen. Reduction is perceptible at 500°, and is accelerated by higher temperatures and pressures. Covering the iron spiral with the asbestos or with aluminium hydroxide had little effect. Methane is formed in amounts up to 30%, and is accompanied by higher homologues. Formaldehyde was only detected qualitatively, and methyl alcohol was not formed. Appreciable amounts of carbon were deposited. On replacement of the iron spiral by a copper spiral or foil, partial reduction of carbon dioxide to carbon monoxide was observed only at 800°; at lower temperatures no reaction occurred. Nickel wire gives similar results to iron, but the reaction is slower. Tungsten, molybdenum, and charcoal behave similarly above 550°, but the reaction velocity is considerably less than in the case of iron and nickel.

R. BRIGHTMAN.

Gas reactions in the hot-cold tube. F. FISCHER and (Führn.) VON WANGENHEIM (Brennstoff-Chem., 1928, 9, 97—98).—When heated at 600—700° and under 1—6 atm. in the hot-cold tube previously described (B., 1926, 777) in presence of steam and iron, carbon monoxide is decomposed into carbon dioxide and carbon and into carbon dioxide and hydrogen, the former reaction preponderating. The same reactions occur in the presence of methane, the latter gas being practically unchanged. Mixtures of carbon dioxide and methane are practically unchanged under these conditions. Methane alone is practically unchanged, only traces of carbon being deposited and a very little hydrogen formed, although condensation to form higher homologues may occur. Unsaturated hydrocarbons, formaldehyde, or alcohols were not formed.

R. BRIGHTMAN.

Testing laboratory of the American Gas Association. R. M. CONNER and F. E. VANDAVEER (Ind. Eng. Chem., 1928, 20, 307—310).—Small amounts of carbon monoxide are determined by the iodine pentoxide method, by thermal conductivity measurement, and by a recording apparatus depending on the liberation of heat in the selective oxidation of carbon monoxide by a mixture of copper oxide and manganese dioxide at 100°. All these methods are capable of an accuracy of 0.002%. The maximum proportions of carbon monoxide allowable in the gases of combustion of different types of gas apparatus are discussed.

C. IRWIN.

Variation in consistency of tars with temperature. (A) H. M. SPIERS (Brennstoff-Chem., 1928, 9, 77—78). (B) H. MALLISON and F. SOLTAN (*ibid.*, 78—79). (C) H. M. SPIERS (*ibid.*, 79).—(A) The formula $\log(C_1/C_2) = K(T_2 - T_1)$, expressing the relation between temperature and consistency of tars (cf. Mallison and Soltan, B., 1927, 517) is accurate only within the limits of experimental error and within a temperature range of 10°. The value of K changes suddenly at a definite temperature, depending on the nature of the tar, and this temperature corresponds with a pronounced change in the physical properties of the tar.

(B) Spiers' remarks (preceding) are criticised. On account of the limited applicability of Spiers' formula, four or six consistency determinations may be necessary for a particular tar to establish values for K , and the authors suggest making a direct determination of consistency at standard temperature (25°). The factor K , being a completely atypical quantity, is insufficient for characterisation of the consistency curve, and the mathematical treatment of the consistency curve of tars is regarded as without practical interest.

(C) A reply. In the great majority of cases the consistency curve shows no break between 24° and 34°, and this temperature range in which K remains constant is sufficiently wide to allow of calculations of consistency at 25° with reasonable accuracy. The calculation of consistency from measurements at two arbitrarily chosen temperatures affords a valuable check on the direct determination. The method offers the further advantage that technical controls can be carried out on the hot tar at temperatures above 25°. In factory work one species of tar only is usually dealt with, and, the properties of this tar being once established,

working temperatures can be chosen between which the consistency curve shows no break. The anomalous value of 55 sec. at 27°, referred to by Mallison and Soltan, disappears if the observation is made at 26° and not at 27°, the observed and calculated consistency values being then 55 and 56.6 sec., respectively.

R. BRIGHTMAN.

Utilisation of *o*-cresol as a necessary condition for the working-up of medium tar oils. F. BOVINI (Notiz. chim.-ind., 1927, 2, 688—690).—A search for new uses for *o*-cresol. Dyes were prepared from *o*-cresol (G.P. 101,541, 102,897) and from *p*-nitroso-*o*-cresol (G.P. 197,165, 205,882), and the results of variations in the methods of preparation are recorded. *p*-Nitrosophenol is best prepared by adding phenol (94 g.), 25% aqueous sodium hydroxide (80 c.c.), sodium nitrite (75 g.), and water (300 c.c.), during 2 hrs. to 93% sulphuric acid (90 g.), water (300 c.c.), and ice (400 g.), the temperature not exceeding 4°; during the addition 10% aqueous sodium hydrogen sulphite (100 c.c.) is slowly added. The mixture is agitated for at least 1 hr., filtered rapidly at 10°, washed with ice-water (100 c.c.), and dried in air at 40—50°. The yield is 75%. *p*-Nitroso-*o*-cresol was similarly prepared in 80—85% yield.

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Purification of effluent liquors from brown coal low-temperature distillation plants. D. WITT and F. SCHUSTER (Gas- u. Wasserfach, 1928, 71, 241—244).—It is pointed out that although these effluents contain ammonia, pyrocatechol, acetic acid, and phenols, together with hydrogen sulphide, the quantities involved are so small as to render unwarrantable any large capital or operating expenditure for their purification. Various proposed methods of purification are discussed, and dismissed owing to their high costs, and an account of experiments made at the Berlin gas-works is given. The electrolytic process was abandoned in favour of one in which the alkaline effluent liquor is first filtered through a bed of the coke obtained in the process to remove hydrogen sulphide. The filtrate is then acidified by treatment with the gas containing sulphur dioxide from the brown coal generators. A second filtration through brown coal coke then purifies the effluent sufficiently for it to be allowed to pass out.

A. E. MITCHELL.

Asphaltogenic substances in American lubricating oils. L. A. BASS and F. C. VILBRANDT (J. Elisha Mitchell Sci. Soc., 1927, 43, 21).—The European asphalt test indicates that American oils do not contain asphaltogenic substances in appreciable quantities; the test may be invalid.

CHEMICAL ABSTRACTS.

Solubility of paraffin wax in pure hydrocarbons. P. WEBER and H. L. DUNLAP (Ind. Eng. Chem., 1928, 20, 383—384).—The solubility of paraffin wax in pentane, hexane, heptane, octane, and *is*decane was determined at temperatures from 0° to 25°, and was found to increase from *is*decane to pentane; the increase with rise of temperature was more rapid with the solvent of higher mol. wt. It is probable from the results, expressed as g. of paraffin per mol. of solvent, that the mol. ratios of solvents to solute may be constant, and that a definite number of molecules of solvent are associated with 1 mol. of solute.

D. G. HEWER.

Heat transfer in pipes. MORRIS and WHITMAN. Counter-current absorption systems. LEWIS and McADAMS.—See I. Decay of oakwood. BRANDL.—See V. Wood preservatives. DEHNST.—See IX. Utilisation of acid sludge. LIKHUSHIN.—See XII. Nitrogen of peats and humus soils. ELLIS and MORISON. Insecticides. FRANÇOIS and SEGUIN.—See XVI.

PATENTS.

Coke ovens. R. E. ELLIS. FROM FOUNDATION OVEN CORP. (B.P. 286,776, 8.12.26. Addn. to B.P. 279,955; B., 1928, 114).—The modification consists in admitting air to the regenerators from flues disposed at the bottom thereof, and passing the air vertically upward through the regenerators to the combustion flues.

A. B. MANNING.

Separation of powdered or granular material, especially coal. R. LESSING (B.P. 287,262, 11.1.27).—A current of gas is caused to flow up a tubular vessel into which the coal is fed and down which the coarse coal drops against the gas current. Later on an increase of cross-section takes place, preferably at an inclined portion of the tube, and middlings drop out; the dust is finally separated and the gas re-used. The middlings may be retreated in a separate unit. B. M. VENABLES.

Treatment of materials with binders and briquetting said material. R. LESSING (B.P. 286,336, 3.11.26).—Finely-divided coal, coke, etc. is thoroughly mixed with dehydrated coal tar, and the mixture extracted with petroleum spirit in such a way that the pitch is precipitated on the solid particles of the material, which is then freed from any remaining solvent by steam-distillation and conveyed directly to the briquetting machine. Addition of a small proportion of sulphuric acid to the mixture before extraction facilitates the process. The petroleum spirit is recovered by distillation from the solution containing tar oils, and is used in the treatment of a further quantity of the material. The briquettes are improved by finally submitting them to a baking process.

A. B. MANNING.

Distillation of coal and similar fuels. H. M. RIDGE (B.P. 286,104, 23.8.26).—Coal, previously reduced to a suitable degree of fineness, traverses a succession of superposed horizontal hearths in an evenly distributed layer. Rabbling devices stir the coal continuously and feed it from each hearth to the one immediately below. The hearths are heated by gas-fired ducts underneath them. The coked residue finally traverses a cooling hearth, the walls of which are provided with air ducts, and is briquetted immediately on its discharge from the retort. The volatile distillation products from each hearth are separately condensed. Those from the lowermost hearths may be returned to the coke in the cooling hearth in order to facilitate the briquetting of the residue.

A. B. MANNING.

Distillation of carbonaceous or bituminous substances. W. E. EVANS. FROM KOHLENVEREDLUNG A.-G. (B.P. 286,404, 31.12.26).—The carbonaceous material is fed in the form of a powder to a nozzle of suitable form through which is blown hot generator- or water-gas directly from the producer. The intimate

mixture of gas and powdered material so produced is carried by the gas current through a chamber in which the material undergoes distillation by the sensible heat of the gas. The solid residue is separated from the volatile products of distillation and used as fuel in the gas producer. In one form of apparatus the retorts have vertical shafts, and can be arranged so that the material traverses two or more of them in succession; the retorts in another form have a number of parallel, slightly inclined or horizontal tubes, which may be heated externally if desired.

A. B. MANNING.

Distillation of solid carbonaceous materials. H. NIELSEN and B. LAING (B.P. 287,037, 4.12.26 and 21.2.27).—Solid carbonaceous materials are distilled in a current of an inert, gaseous, heating medium, the volume of which is so great that the heaviest oil fractions produced in the distillation are completely vaporised at temperatures not exceeding 450–550°. With materials such as oil shales containing a large percentage of oil, the volume of distilling medium used is such that it may be cooled to 80–100° without causing condensation of the heaviest fractions. Coking coals are subjected to a preliminary treatment by gases containing an oxidising constituent in order to reduce their caking properties. By the addition of some oxygen to the distilling medium the resin-forming constituents of the oils are polymerised and the subsequent refining processes thereby facilitated. The higher-boiling fractions of the refined neutral oils yield paraffin wax and valuable lubricating oils. The solid residue forms a free-burning fuel containing 4–15% of volatile matter.

A. B. MANNING.

Production of high-value oils from raw coal by low-temperature distillation and hydrogenation. R. FEIGE (B.P. 274,465, 11.7.27. Ger., 16.7.26).—In a process for the economical production of oils from coal, the coke produced is used in part for the generation of electric current, which is in turn used to electrolyse water. The remaining coke is gasified with the electrolytic oxygen, the hydrogen being used to hydrogenate the resulting products, which may or may not be previously mixed with gas and/or tar from the coal-distillation plant.

C. O. HARVEY.

Destructive hydrogenation of carbonaceous materials. I. G. FARBENIND. A.-G. (B.P. 272,538, 10.6.27. Ger., 11.6.26).—The gases from processes for the hydrogenation of carbonaceous materials at high temperatures and pressures are scrubbed, under pressure, with benzene, preferably that obtained in the process. The gaseous hydrocarbons are thereby scrubbed out and the residual gases can be recirculated.

A. B. MANNING.

Production of [unsaturated] hydrocarbons from coal, tars, mineral oils, etc. J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 286,825, 3.1.27).—Valuable unsaturated hydrocarbons, in particular acetylene, are produced by submitting coal, tars, oils, etc., either vaporised or in a finely-divided condition, to the action of an electric arc in the presence of less than half the quantity of water vapour requisite for the production of water-gas. The water vapour may be replaced by

hydrogen, nitrogen, carbon dioxide, etc., and the mixture fed to the arc is preferably preheated.

A. B. MANNING.

Production of carbon. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 286,845, 4.2.27).—A continuous process for the manufacture of carbon from carbon monoxide consists in adding 0.1–0.2% (by vol.) of iron carbonyl vapour to the gas and passing the mixture through a high-pressure furnace at about 400°. The effluent gas can first be freed from carbon dioxide by washing with water under pressure, and then subjected to the same process in a second apparatus. The carbon formed contains about 2% Fe.

A. B. MANNING.

Fuels for internal-combustion engines. BRIT. DYESTUFFS CORP., LTD., and S. COFFEY (B.P. 287,192, 25.10.26).—Heavy-metal derivatives of β -diketones of the type $R \cdot CO \cdot CHR' \cdot CO \cdot R''$, where R and R'' are hydrocarbons or substituted hydrocarbon radicals, and R' is the same or hydrogen, are added to motor fuels as antidetonants. Chromium, iron, cobalt, manganese, and thorium derivatives of acetylacetone, propionylacetone, and γ -methylacetylacetone are particularly claimed.

C. HOLLINS.

Manufacture of pulverulent fuel for internal-combustion engines. I. G. FARBENIND. A.-G. (B.P. 270,702, 2.5.27. Ger., 7.5.26).—Pulverulent fuels are charged with gaseous, or vaporised, or atomised liquid fuels. Their ignition temperature is thereby reduced and their speed of combustion raised.

A. B. MANNING.

Motor fuel. T. MIDGLEY, JUN., ASSR. to GEN. MOTORS CORP. (U.S.P. 1,662,323, 13.3.28. Appl., 16.5.25).—The fuel is comprised of a low-compression fuel and nickel carbonyl.

F. G. CLARKE.

Gas producer for use with wood etc. G. IMBERT (B.P. 279,475, 20.10.27. Ger., 25.10.26).—A producer suitable for use on motor vehicles.

A. B. MANNING.

Regenerator settings for use in the manufacture of gas. R. W. BROADHEAD (B.P. 286,418, 17.1.27).—The producer and regenerator are arranged externally to the retort bench, one regenerator serving a number of beds of retorts, the waste gases from which pass by insulated ducts to the regenerator, whilst preheated producer gas and secondary air are led back by other insulated ducts to the beds of retorts.

A. B. MANNING.

Production of rich gas and semi-coke from bituminous fuel. A. L. MOND. From METALBANK U. METALLURGISCHE GES. A.-G. (B.P. 285,664, 15.3.27).—The fuel is passed continuously down a column in the upper zone of which it is distilled by the sensible heat of the gas produced by the gasification in the lower zone of part of the semi-coke. The gasification process is carried out by means of oxygen, or air rich in oxygen, and steam if desired, in such a manner as to produce only the sensible heat required for the distillation in the upper zone; the remainder of the semi-coke is withdrawn without taking part in the gasification process. The gas may be enriched by injecting tar residues etc. into the hot zones of the generator.

A. B. MANNING.

Production of coal gas. W. M. CARR (B.P. 286,758, 13.11.26).—The proportion of a diluent gas added to coal gas to maintain a constant calorific value is regulated by an electrical or other device controlled by the flame temperature of a burner supplied from a by-pass through which a continuous rapid flow of the diluted gas is maintained.

A. B. MANNING.

Gas-purification process and apparatus. KOPPERS CO., and F. W. SPERR, JUN. (B.P. 261,755, 17.11.26. U.S., 18.11.25. Cf. B.P. 255,139–255,145; B., 1926, 813).—Hydrogen sulphide is removed from coke-oven or other gases by a cyclic process in which the gases are treated with wash liquors containing ammonia and a suspension of hydrated ferric oxide or similar metallic compound. If the gases contain ammonia this may be utilised in the preparation of the ferric oxide suspension from a solution of an iron compound. After absorption of the hydrogen sulphide the ammonia is recovered from the sulphided liquor by distillation; the liquor is then aerated, the ferric sulphide being converted into ferric oxide and sulphur, and, after separation of the sulphur, is recirculated, if necessary with part or all of the recovered ammonia, for further absorption of hydrogen sulphide. The process may be used in conjunction with either the "indirect" or "direct" system of ammonia recovery. In the former case the whole of the ammonia is absorbed in the wash liquor; in the latter, the temperature of the liquor is adjusted so that a part only of the ammonia is absorbed, the remainder being removed subsequently in a saturator containing sulphuric acid. Gases containing little or no ammonia are scrubbed in a tower at the top of which is introduced the liquor containing the iron compound, and at part of the way up which is introduced an ammoniacal solution. From time to time part of the liquor may be withdrawn from circulation after the aeration and oxidation stage, and treated for the recovery of ammonia present as fixed salts. Details are given of complete plants suitable for the operation of the processes.

A. B. MANNING.

Absorption of fluids from gases. Removal of tar from gases. E. PIRON, ASSR. to PIRON COAL DISTILLATION SYSTEMS, INC. (U.S.P. 1,664,483–4, 3.4.28. Appl., [A] 30.9.21, [B] 25.3.25).—(A) An absorption device consists of one or more units each comprising a chamber containing a horizontal, foraminous member across which a mentrum flows in separate streams through guide-ways from an inlet at one side to an outlet at the other. (B) Wood-distillation gases are scrubbed with a medium inert towards tar, whereby the greater part of the latter is removed. After heating the partially cleansed gases by mixing them with hot residual gases, they are scrubbed with sulphuric acid at such a temperature that no constituent other than tar condenses. Pyroigneous acid is then condensed and a sufficient quantity of the residual gases is heated to give the necessary temperature during the acid washing.

F. G. CLARKE.

[Tar] still. F. PUENING, ASSR. to KOPPERS CO. (U.S.P. 1,664,863, 3.4.28. Appl., 25.7.24).—In a vertical cylindrical still for continuous use, baffling effects are brought about by means of a diametrically placed

partition and by baffle plates, including an inner plate concentric with the still wall. C. O. HARVEY.

Separation of acid sludge emulsions. Hydrolysis of acid sludge. E. W. ROTH, Assr. to GEN. PETROLEUM CORP. OF CALIFORNIA (U.S.P. 1,665,189 and 1,665,190, 3.4.28. Appl. [A, B], 13.4.27).—(A) Emulsions of tar and dilute acid resulting from the hydrolysis of acid sludge are broken by passing through a pack of granular inert material followed by settling and separating operations. (B) Acid sludge is separated into useful constituents by digestion with a solvent oil and dilute sulphuric acid of a density initially greater than that of the sludge. C. O. HARVEY.

Fire still and oil treater. F. E. GILMORE, Assr. to F. E. GILMORE Co. (U.S.P. 1,664,920, 3.4.28. Appl., 4.2.26).—The oil is evaporated from a chamber heated by a central cylindrical flue fitted with radiating fins and having a burner at its base. C. O. HARVEY.

[Oil] evaporator. C. F. BRAUN (U.S.P. 1,662,142, 13.3.28. Appl., 14.3.22).—A casing contains a series of vertical tubes upon the surfaces of which flow films of the oil to be distilled. Steam passes up around the tubes, and oil, always at a higher temperature than the steam, is passed down through the tubes. F. G. CLARKE.

Cracking of hydrocarbon oils or their distillates by distillation under pressure. S. STRANSKY and F. HANSGIRG (B.P. 267,958, 17.3.27. Austr., 18.3.26).—In cracking hydrocarbon oils, the formation of gaseous and coke-like products is minimised by maintaining the oil at a temperature just sufficient for the generation of the necessary pressure (about 400°). Under these conditions, splitting of the paraffins and naphthenes of high mol. wt. proceeds to an equilibrium point, the reactions involved being reversible. In order to urge the cracking process towards completion, the cracked products are rapidly removed from the reaction zone by means of electrical resistance heaters situated inside the still and capable of providing a great quantity of heat per unit of time. The level of the oil in the still is also automatically adjusted, as, with an overfilled still, the vapours carry over with them entrained droplets of the heavier hydrocarbons, and, with a low level of oil, the necessary pressure cannot be attained without applying sufficient heat to vaporise the higher-boiling constituents. C. O. HARVEY.

Cracking of hydrocarbon oil. R. T. POLLOCK (B.P. 287,344, 29.4.27).—In a cracking plant consisting of a tubular still connected with an expansion chamber, a dephlegmator, and condensing apparatus, the reflux from the dephlegmator, prior to its return to the cracking still, is freed from more volatile constituents in a small still heated by the flue gases from the main still. C. O. HARVEY.

Cracking of hydrocarbons. G. H. TABER, JUN., Assr. to SINCLAIR REFINING Co. (U.S.P. 1,663,868, 27.3.28. Appl., 24.6.26).—The uncondensed vapours remaining after the passage of the products of a cracking operation through a condenser are partially retained in an absorbent medium and subsequently recovered. Unabsorbed vapours and gases are finally treated with some of the cracking stock. C. O. HARVEY.

Treatment of heavy hydrocarbons for the production of lighter hydrocarbons. C. ARNOLD. From STANDARD DEVELOPMENT Co. (B.P. 286,917, 2.5.27).—In a cracking system involving rapid passage of the oil through heating chambers, wherein the necessary temperature is attained with a minimum of actual decomposition or carbon formation, to a series of conversion chambers and thence to the vaporisation chambers, individual conversion chambers may be isolated for cleaning purposes without interfering with the continuity of the process. A lower cracking temperature than usual may be employed, as the time of passage of the oil through the series of converters is prolonged. The cleaned converters may be charged with raw material before being reintroduced into the system. C. O. HARVEY.

Cracking of oil. E. C. HERTHEL, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,660,647, 28.2.28. Appl., 23.3.25. Renewed 26.4.27).—The furnace gases employed to heat a pressure still are first led around an auxiliary coil, through which tar-free oil from the reflux is returned to the still, and are then further cooled by dilution with a portion of the waste furnace gases. Overheating of the still with consequent deposition of carbon is avoided. T. S. WHEELER.

Cracking of oils. F. PUENING (U.S.P. 1,664,263, 27.3.28. Appl., 26.5.19).—The oil is heated and cracked while circulating through the annular space between two concentric drums provided with scrapers for the continuous removal of deposited carbon. C. O. HARVEY.

Distillation of oils occurring in the petroleum, tar, and similar industries, more particularly for the production of lubricants. L. STEINSCHNEIDER (B.P. 264,476, 23.12.26. Czechoslov., 15.1.26).—An apparatus for removing from distilled lubricating oils the low-boiling and dissolved gaseous decomposition products consists of two interconnected evaporators from the first of which the volatile impurities are withdrawn. The pressure in the second evaporator, from which the purified oil is distilled, is lower than in the first. The heating of the oil to the final temperature may be carried out in stages by employing a battery of these distilling units. C. O. HARVEY.

Distillation of oil. H. L. DOHERTY, Assr. to HEAT TREATING Co. (U.S.P. 1,662,105, 13.3.28. Appl., 7.7.20).—Fractions of progressively rising b.p. are obtained by passing the oil in a downward direction through a chambered still. C. O. HARVEY.

Distillation of lubricating oils. R. W. HANNA, Assr. to STANDARD OIL Co. OF CALIFORNIA (U.S.P. 1,664,977, 3.4.28. Appl., 4.10.23).—The oil is heated to a cracking temperature and maintained in the liquid state. Before substantial cracking occurs the heated oil rapidly passes to a vaporising chamber maintained under reduced pressure, wherein the temperature of the oil drops below the cracking point. The vapours, thus freed from "mist" without substantial scrubbing, are fractionally condensed. C. O. HARVEY.

Neutralising treatment of acid oils for lubricant stocks. Treatment of lubricant stocks. G. F. OLSEN, Assr. to GEN. PETROLEUM CORP. OF CALIFORNIA

(U.S.P. 1,665,110—1, 3.4.28. Appl., [A] 31.8.26, [B] 15.10.27).—(A) Oils containing organic and mineral acid are agitated and treated with sufficient pulverised solid alkali to neutralise only the mineral acid, the addition of water being avoided throughout. (B) In treating viscous petroleum oils with liquid sulphur dioxide, the oil is previously diluted with lighter petroleum products.

C. O. HARVEY.

Manufacture from bituminous shales of a product adapted for dry distillation. PATENTAKTIEBOLAGET GRÖNDAL-RAMÉN (B.P. 278,378, 30.9.27. Swed., 2.10.26).—Shales high in bitumen are finely crushed, thoroughly mixed to give a homogeneous product, and the latter is formed into balls by further admixture with water and passage of the moist material through a revolving drum. The balls are dried and strengthened by treatment with hot fuel gases in a second drum.

A. B. MANNING.

Purification of the liquid complex hydrocarbon products of the destructive hydrogenation of carbonaceous materials. I. G. FARBENIND. A.-G. (B.P. 257,270, 18.8.26. Ger., 20.8.25).—The crude liquid hydrogenation products are treated with one of the lower aliphatic alcohols (*e.g.*, crude synthetic methyl alcohol obtained by the catalytic hydrogenation of carbon monoxide). The treatment is preferably carried out by a counter-current method, the solid paraffins having first been separated from the crude oils at -10° . The impurities dissolve in the alcohol and highly-refined products are obtained, suitable for use as lubricating oils etc.

A. B. MANNING.

Recovery of paraffin [from slack wax]. W. A. GRUSE and W. F. FARAGHER, Asss. to GULF REFINING Co. (U.S.P. 1,663,592, 27.3.28. Appl., 28.10.24).—Hard paraffin wax is recovered from solidified slack wax by passing the latter through a mass of water maintained at a suitable temperature and repeatedly cutting the wax so as to enable the oil to separate therefrom and rise to the surface.

C. O. HARVEY.

Disposal of spent clay in [oil]-refining plants. J. M. CORY and F. H. BUNKE, Asss. to SOLAR REFINING Co. (U.S.P. 1,660,434, 28.2.28. Appl., 27.1.26).—A mixture of the clay and a hydrocarbon oil is claimed as a liquid fuel.

T. S. WHEELER.

Washing apparatus for coals. P. WOLF (U.S.P. 1,666,189, 17.4.28. Appl., 28.6.26. Fr., 3.7.25).—See B.P. 254,709; B., 1927, 161.

Degasification of coal. N. YOUNG (U.S.P. 1,664,723, 3.4.28. Appl., 30.10.19. Ger., 1.11.18).—See B.P. 134,529; B., 1921, 74 A.

Fuel for use in internal-combustion engines. S. ISERMAN, W. VERNET, and E. Q. MOSES, Asss. to F. J. HALL and L. B. RATTERMAN (Re-issue 16,937, 17.4.28, of U.S.P. 1,654,259, 27.12.27).—See B., 1928, 222.

Purification of illuminating or like gas. HUMPHREYS & GLASGOW, LTD., Asses. of W. H. FULWEILER and C. W. JORDAN (B.P. 273,250, 26.3.27. U.S., 24.6.26).—See U.S.P. 1,632,758; B., 1927, 770.

Drying of gases. R. E. SLADE and V. E. PARKE, Asss. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,664,997,

3.4.28. Appl., 28.1.26. U.K., 28.1.25).—See B.P. 240,350; B., 1925, 957.

Production of stable [oil] emulsions. R. MEZGER (U.S.P. 1,665,105, 3.4.28. Appl., 1.4.27. Ger., 1.7.25).—See B.P. 254,701; B., 1927, 423.

Gas producers (B.P. 284,639).—See I. Separation of sulphonic acids (B.P. 284,859).—See III. Filling cracks in brickwork (B.P. 286,933). Asphaltic or tarry emulsions (B.P. 264,496).—See IX. Insecticides (B.P. 287,371).—See XVI. Ammonium sulphoichthyolate (U.S.P. 1,664,376). Medicinal liquids (U.S.P. 1,660,655).—See XX.

III.—ORGANIC INTERMEDIATES.

Synthesis of methyl alcohol from carbon monoxide and hydrogen. W. K. LEWIS and P. K. FROLICH (Ind. Eng. Chem., 1928, 20, 285—290).—Factors governing the catalytic synthesis of methyl alcohol are studied. Among the possible simple reactions between carbon monoxide and hydrogen the formation of methyl alcohol is accompanied by the largest decrease in volume, and hence is favoured by increase in pressure. Even with the best catalysts it is not possible to reduce the temperature of reaction below 300° , and whilst with a pressure of 1000 lb./in.² practically 100% methyl alcohol may be obtained at 300° , it is desirable to work at higher pressures to prevent the formation of methane. It has been found advantageous to use as a catalyst mixtures of two or more metallic oxides, of which one at least is non-reducible in carbon monoxide and hydrogen at atmospheric pressure at the temperature employed (*e.g.*, oxides of copper, zinc, and aluminium). Owing to the exothermic character of the reaction (26,150 g.-cal. per mol. of gaseous methyl alcohol at 327°) the catalyst should be supported on a good heat conductor, *e.g.*, granulated metallic copper, to prevent local overheating. The catalyst is prepared by precipitating the hydroxides by ammonia from a solution of the mixed nitrates at 85° , and is obtained as a gel into which the supporting copper is mixed prior to drying at 110° . It is sensitive to poisons, but by connecting several chambers in series it is possible to dispose of the catalyst poisons in the first chamber, thus protecting the contact material in the others. As the temperature is raised the yields of methyl alcohol pass through a maximum at $300-350^{\circ}$, and then fall off considerably owing to the decreased activity of the catalyst. Whilst the quantity formed per unit time increases with the rate of gas flow through the reaction chamber, the percentage conversion of carbon monoxide decreases owing to the shorter time of contact between the reacting gas and the catalyst. A pure alcohol is produced at about 3000 lb. pressure and at a relatively moderate temperature, and has a sp. gr. corresponding to 99—100% methyl alcohol. An apparatus suitable for the synthesis is described.

W. J. POWELL.

Application of the U.S.P. methyl alcohol test. W. L. O. WHALEY (Ind. Eng. Chem., 1928, 20, 320—322).—The method as given is suitable only for alcoholic solutions, hence when it is applied to flavours or medicinal preparations, the latter should be distilled and the distillates tested with the modified Schiff's reagent. It is important to prepare a sample of 5% total alcoholic

strength, as with stronger solutions the colour formation may be inhibited. The test is more sensitive if the sample is oxidised with dilute potassium permanganate and phosphoric acid, the excess of permanganate being decomposed with oxalic acid solution before distillation; under these conditions, however, many other substances yield similar colorations.

W. J. POWELL.

Use of *o*-cresol. BOVINI. **Cracking of hexadecane.** GAULT and SIGWALT.—See II.

PATENTS.

Production of formaldehyde by catalysis. E. A. BARBET (U.S.P. 1,661,063, 28.2.28. Appl., 23.10.23. Belg., 30.10.22).—In the oxidation of methyl alcohol to formaldehyde the air used is led in an inner tube through the catalyst chamber, and thus preheated is mixed with methyl alcohol vapour and passed to the catalyst.

T. S. WHEELER.

Manufacture of ethylene glycol [and ethylene oxide]. BRIT. DYESTUFFS CORP., LTD., K. H. SAUNDERS, and H. WIGNALL (B.P. 286,850, 8.2.27).—A mixture of about 40 pts. of ethylene chlorohydrin vapour and 60 pts. of steam, obtained for example by passing steam through a tower against a counter-current of crude chlorohydrin, is led up a packed tower against a downflowing stream of sodium carbonate solution. The glycol so produced dissolves in the salt solution and forms at the bottom of the tower a layer through which the chlorohydrin vapour and steam bubble. By a suitable heating of this layer, concentrated glycol may be drawn off through a constant-level trap, and its salt content allowed to crystallise out. If concentrated sodium hydroxide solution is used in place of sodium carbonate, ethylene oxide is produced and passes from the top of the tower to be dried and collected in the usual manner.

C. HOLLINS.

Manufacture of halogenated alcohols. I. G. FARBENIND. A.-G., and J. CALLSEN (B.P. 286,797, 13.12.26. Addn. to B.P. 235,584; B., 1925, 738).—A secondary alcohol is used as solvent in place of the primary alcohol of the prior patent. Bromal, dissolved in *isopropyl* alcohol, is reduced by aluminium ethoxide at 115° in a stream or atmosphere of hydrogen to tribromoethyl alcohol, m.p. 80°.

C. HOLLINS.

Manufacture of primary aliphatic and cyclic amines. I. G. FARBENIND. A.-G. (B.P. 265,960, 7.2.27. Ger., 9.2.26).—Ketones, aldehydes, or their ammonia compounds are passed at 100–200° with hydrogen and ammonia over hydrogenation catalysts (reduced nickel and silica on pumice) preferably with the addition of dehydrating catalysts (aluminium phosphate, alumina) and activators (zinc oxide, magnesium oxide). *cyclo*-Hexylamine, *n*-butylamine, and *isobutylamine* are thus obtained together with only insignificant amounts of secondary and tertiary amines.

C. HOLLINS.

Concentration of aqueous solutions of fatty acids. F. E. LICHTENTHAELER (U.S.P. 1,660,756, 28.2.28. Appl., 21.1.26).—The dilute solution is treated with anhydrous aluminium ammonium sulphate (cf. U.S.P. 1,492,717; B., 1924, 671), ethyl alcohol, and an esterification catalyst, and the mixture is heated at about 90° to give ethyl acetate and the hydrated salt. The ester is recovered by distillation and hydrolysed to yield the concentrated acid.

T. S. WHEELER.

Manufacture of new aralkylated unsaturated fats and fatty acids and their sulphonic acids. W.

CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 286,796, 13.12.26).—Unsaturated aliphatic acids or fats are condensed with aralkyl halides, especially in presence of a catalyst such as zinc chloride, iron powder, aluminium chloride, to give compounds which are easily sulphonated, forming wetting-out or dispersing agents unaffacted by acids or salt solutions. *E.g.*, oleic acid and rape oil are condensed with benzyl chloride in presence of zinc chloride, and sulphonated.

C. HOLLINS.

Manufacture of new derivatives from lignin-sulphonic acid (sulphite-cellulose waste). BRIT. DYESTUFFS CORP., LTD., and A. J. HALLWOOD (B.P. 286,808, 21.12.26).—Sulphite cellulose pitch is heated with aqueous ammonia at 110–120° in a closed vessel, to form an aminated derivative which is a highly-protective colloid suitable as a drying-down agent, and has tanning properties.

C. HOLLINS.

Preparation of vanillin and *isovanillin*. F. BOEDECKER (B.P. 285,156, and Addn. B.P. 285,551, [A] 12.11.26, [B] 6.11.26).—(A) The fission of the methylenedioxy-group of safrole or *isosafrole* by alcoholic alkali, followed by methylation with methyl sulphate and boiling with alcoholic hydrogen chloride, leads to a mixture of *isoeugenol* and *isochavibetol*, which may be oxidised in the usual manner to vanillin and *isovanillin*, separable by known means. Or the *isochavibetol* may be frozen out, methylated to 4-propenylveratrol (*isoeugenol* methyl ether), and converted by alcoholic alkali into a mixture of *isoeugenol* and *isochavibetol* from which the latter is again frozen out and added to the next batch for methylation. The *isoeugenol* is purified by crystallisation of its benzoyl or nitrobenzoyl derivative and subsequent hydrolysis; or it may be converted by way of its methyl ether into the mixture of *isoeugenol* and *isochavibetol*, and hence into *isovanillin*, if desired. (B) The mixture of *isoeugenol* and *isochavibetol* obtained is separated by taking advantage of the sparing solubility of sodium *isoeugenol*.

C. HOLLINS.

Manufacture of *isoeugenol*. SOC. ANON. PROD. CHIM. COVERLIN, Assecs. of R. H. BOTS (B.P. 271,819, 2.2.27. U.S., 25.5.26).—Potassium eugenol is heated in aniline, *o*-toluidine, or other suitable amine at the b.p. to give *isoeugenol*, which may be oxidised to vanillin with nitrobenzene and alkali without isolation.

C. HOLLINS.

Separation of *m*-dinitrobenzene from its isomerides. H. J. WEILAND and I. GUBELMANN, ASSTS. to NEWPORT Co. (U.S.P. 1,665,005, 3.4.28. Appl., 19.8.26).—Technical *m*-dinitrobenzene is treated in the presence of water with an alkali-forming substance, which converts the *o*- and *p*-isomerides into water-soluble derivatives without decomposing the *m*-dinitrobenzene. The latter may then be separated in a pure condition.

F. G. CLARKE.

Alkylation of carbazole. R. W. HESS and J. C. SIEMANN, ASSTS. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,662,061, 13.3.28. Appl., 25.1.22).—*N*-Alkyl derivatives of carbazole are produced by the interaction of an alkylating agent with carbazole in the presence of an acid-binding agent.

F. G. CLARKE.

Manufacture of indophenols. R. W. HESS and O. D. CUNNINGHAM, ASSRS. to NAT. ANILINE AND CHEM. Co., Inc. (U.S.P. 1,662,062, 13.3.28. Appl., 25.3.22).—An indophenol is produced by condensing carbazole or a derivative thereof with excess of *p*-nitrosophenol in the presence of sulphuric acid. With *N*-carbazole derivatives the reaction is commenced at -5° and then maintained below 0° .

F. G. CLARKE.

Manufacture of new quaternary ammonium compounds from halogen-substituted tertiary aromatic amines. W. CARMAEL. From I. G. FARBENIND. A.-G. (B.P. 286,984, 10.12.26).—Alkylating agents are obtained by interaction of alkyl sulphates or alkyl arylsulphonates and halogenated tertiary amines of the benzene or naphthalene series. 3-Chloro-*p*-tolyltrimethylammonium *p*-toluenesulphonate, m.p. 154° , 1-chloro- β -naphthyltrimethylammonium methyl sulphate, m.p. 98° , *o*-chloro- (with 2:4-dichloro-)phenyltrimethylammonium methyl sulphate, and the methosulphates of 3:3'-dichloro- [m.p. 218° (decomp.)] and 3:3'-dibromo-4:4'-tetramethyldiaminodiphenylmethanes are described.

C. HOLLINS.

Separation of mono- and di-alkyl derivatives of aromatic amines. SILESIA VER. CHEM. FABR. (B.P. 280,877, 21.10.27. Ger., 19.11.26).—The mixture of secondary and tertiary amines is treated with a slight excess of phthalic anhydride in an inert solvent (benzene etc.). The phthalamic acid formed from the secondary amine dissolves in the benzene, from which it is removed by shaking with aqueous alkali and hydrolysed with boiling acid; the tertiary amine is recovered from the benzene solution by distillation.

C. HOLLINS.

Preparation of disulphide compounds. GOODYEAR TIRE & RUBBER Co., Asses. of C. MCK. CARSON (B.P. 277,338, 6.9.27. U.S., 7.9.26).—Mercaptans (in particular, 1-mercaptobenzthiazole) are converted into corresponding disulphides by treatment in a solvent (e.g., carbon tetrachloride) with sulphur chloride, sulphur and hydrogen chloride being produced. The disulphide, m.p. 175° , from 1-mercaptobenzthiazole is a vulcanisation accelerator.

C. HOLLINS.

Separation and purification of sulphonic acids of high mol. wt. G. PETROFF (B.P. 284,859, 1.2.27).—Sulphonated oils and other sulphonic acids of high mol. wt. (sulphonated black naphtha, octahydroanthracene-sulphonic acid, Twitchell's reagents, etc.) are mixed with cellulosic material (sawdust, wood-powder, hydrocellulose), dried, and extracted successively with mineral oil (benzine), benzene, and alcohol. The acids are recovered from the extracts by washing with water, aqueous alcohol, or alcohol, or by conversion into salts.

C. HOLLINS.

Manufacture of *o*-aminoaldehydes and *o*-amino-ketones of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 267,163, 8.3.27. Ger., 8.3.26).—The anthraquinoneoxazoles obtained by the action of fuming sulphuric acid on 1-nitro-2-alkylantraquinones (B.P. 147,001; B., 1922, 50 A) are reduced with ammonia and sodium hyposulphite, or with ferrous sulphate, to 1-aminoanthraquinone-2-aldehydes or corresponding ketones. The oxazole reaction mixture may be diluted and reduced with ferrous sulphate without

isolation. By these two reactions 1-nitro-2-methylanthraquinone is converted into 1-aminoanthraquinone-2-aldehyde, m.p. 239° , 1-nitro-2-ethylanthraquinone into 1-aminoanthraquinonyl methyl ketone, m.p. 220° , 1:5-dinitro-2-methylanthraquinone into 5-nitro-1-aminoanthraquinone-2-aldehyde, m.p. 228° , 1:5-dinitro-2:6-dimethylanthraquinone into 1:5-diamino-2:6-dialdehydoanthraquinone, m.p. 340° , and 1:8-dinitro-2:7-dimethylanthraquinone into the 1:8:2:7-compound. C. HOLLINS.

Manufacture of *o*-aminocarboxylic acids of the anthraquinone series and substitution products thereof. I. G. FARBENIND. A.-G. (B.P. 267,164, 8.3.27. Ger., 8.3.26).—Anthraquinoneoxazoles (B.P. 147,001; B., 1922, 50 A) are converted by hot dilute alkalis into 1-aminoanthraquinone-2-carboxylic acids. The preparation of 1-aminoanthraquinone-2-carboxylic acid, its 5-nitro-derivative, m.p. 325° , 1:5-diaminoanthraquinone-2:6-dicarboxylic acid, and the 1:8:2:7-isomeride, is described.

C. HOLLINS.

Manufacture of polycyclic compounds containing a carbonyl group. I. G. FARBENIND. A.-G. (B.P. 263,163, 15.12.26. Ger., 15.12.25).—Aromatic and heterocyclic ketones containing halogen in positions *ortho* or *peri* to the carbonyl group are cyclised by heating with a slightly alkaline salt or acid-binding agent (sodium carbonate, calcium carbonate, sodium acetate, magnesium oxide), especially in presence of catalysts (copper, copper carbonate, iron carbonate).

C. HOLLINS.

Manufacture of catalysts and their application in the reduction or hydrogenation of organic compounds. I. G. FARBENIND. A.-G. (F.P. 621,434, 20.7.26. Ger., 22.7.25).—Further to B.P. 255,884 (B., 1927, 528) are the following. For the preparation of a nickel catalyst, dust-free air is passed for 4 hrs. at 70° through aqueous nickel nitrate solution containing 25% ammonia and finely divided silica gel, the precipitate being dried and reduced in hydrogen first at $100-130^{\circ}$ and then at 550° ; the catalyst is finally cooled in hydrogen or nitrogen and transferred to the liquid (water, pyridine, decalene) for use. The precipitate obtained by adding nickel nitrate solution at 70° dropwise to a very dilute sodium carbonate solution may be similarly worked up. These catalysts are used in the reduction of various nitro-compounds with hydrogen at $60-100^{\circ}$ to give amines and diamines, namely, α -naphthylamine, *m*- and *p*-phenylenediamines, cresidine, *o*-chloroaniline, 4-chloro- α -naphthylamine (m.p. 95°), aniline-*o*-sulphonic acid, *o*-aminoacetanilide (m.p. $120-121^{\circ}$: hydrochloride, m.p. 132°), *p*-aminoacetanilide, *p*-aminobenzoic acid, ethyl 3-amino-4-hydroxybenzoate, naphthastyril and 5-amino-1-naphthoic acid (from 1:5[8]-nitronaphthoic acid), 2:5-dichloroaniline (m.p. 50°), 2:4:6-triaminotriphenyl-1:3:5-triazine, 5-aminoacenaphthene, methyl 5-*m*-nitrobenzoylsalicylate (m.p. $130-131^{\circ}$), 3-amino-*p*-tolyl carbonate (m.p. $146-147^{\circ}$), 4-chloro-2-aminophenol; benzeneazosalicylic acid gives 5-aminosalicylic acid, and azoxybenzene yields aniline. Reduction of nitriles under these conditions gives a larger proportion of primary and less secondary amine. From phenylacetonitrile are obtained 63% of β -phenylethylamine and 18% of secondary base; from *p*-nitrophenylacetonitrile *p*-aminophenylacetonitrile and

β -*p*-aminophenylethylamine, b.p. about 147°/11 mm., result at 50 and 90°, respectively. Similarly α -naphtho-nitrile gives (1-naphthylmethyl)amine (70%) and di-(1-naphthylmethyl)amine (20%), acetonitrile ethylamine, and propionitrile *n*-propylamine (80%). Other reductions described are: crotonaldehyde to *n*-butaldehyde and thence to *n*-butyl alcohol; cinnamonitrile to β -phenylpropionitrile and thence to γ -phenyl-*n*-butylamine; acetophenone to phenylmethylcarbinol and thence to ethylbenzene; diethyl ketone to diethylcarbinol (γ -hydroxypentane); ethyl acetoacetate to ethyl γ -hydroxybutyrate; 2-*p*-toluoylbenzoic acid to 4-methyldiphenylmethane-2'-carboxylic acid, and the nitro-compound to 3-amino-4-methyldiphenylmethane-2'-carboxylic acid, m.p. 130—136°; benzophenone to diphenylmethane, b.p. 260—262°; *m*-nitro-*p*-tolyl sulphonyl ketone to *m*-amino-*p*-tolylsulphonyl-1-naphthylmethane; 2:2'-dinitrodi-*p*-anisyl ketone to 2:2'-diaminodi-*p*-anisylmethane, m.p. 110°; phenyl *m*-nitro-*p*-tolyl ketone to 3-amino-4-methyldiphenylmethane (hydrochloride, m.p. 218°); 3-nitrodi-*p*-tolyl ketone to 3-aminodi-*p*-tolylmethane (hydrochloride, m.p. 210—211°); acetoxime to isopropylamine; quinaldine to tetrahydroquinaldine at 80°, or decahydroquinaldine at 130°; pyridines to piperidines.

C. HOLLINS.

Manufacture of carboxylic acids of acenaphthene.

I. G. FARBENIND. A.-G. (B.P. 274,894, 22.7.27. Ger., 24.7.26).—The mono- or di-acyl derivatives, obtained by condensing acenaphthenes with acetyl or chloroacetyl chloride in presence of aluminium chloride, are oxidised with alkaline hypochlorite to mono- or dicarboxylic acids (and chloroform). The methylene groups of the *peri*-ring are not attacked. The 5-carboxylic acid, m.p. 217°, 5:6(?)-dicarboxylic acid (from bischloroacetylacenaphthene, m.p. 180°), and 5-bromo-6-carboxylic acid (from the bromination product, m.p. 164°, of 5-acetylacenaphthene), are described.

C. HOLLINS.

Method of sublimation.

C. FIELD, Assr. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,662,056, 13.3.28. Appl., 22.3.20).—Substances such as anthracene or anthraquinone in a finely-divided condition are charged into a preheated receptacle and stirred until they are plastic. A heated gas or vapour is then introduced to promote sublimation.

F. G. CLARKE.

Manufacture of paraformaldehyde. E. KUSS, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,666,708, 17.4.28. Appl., 18.6.26. Ger., 24.6.25).—See B.P. 267,768; B., 1927, 428.

Production of vinyl acetate. E. BAUM, H. DEUTSCH, W. O. HERRMANN, and M. MUGDAN, Assrs. to CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (U.S.P. 1,666,482, 17.4.28. Appl., 15.6.22. Ger., 23.6.21).—See B.P. 182,112; B., 1923, 861 A.

Stearic acid (U.S.P. 1,659,790).—See XII. **Pyridine derivatives** (B.P. 259,982 and 265,167).—See XX.

IV.—DYESTUFFS.

Lake pigments. WAGNER and others.—See XIII.

PATENTS.

Manufacture of new vat dyes of the anthraquinone series. W. CARMAEL. From I. G. FARB-

ENIND. A.-G. (B.P. 285,555, 16.11.26).—A 3-halogenopyrazolanthrones, particularly the 3-bromo-compound prepared from 3-bromo-1-aminoanthraquinone by diazotisation, reduction, and cyclisation, is heated with an acid-binding agent (potassium acetate) and a catalyst (copper acetate) in boiling nitrobenzene to give a blue vat dye. C. HOLLINS.

Manufacture of vat dyes of the benzanthrone series. I. G. FARBENIND. A.-G. (B.P. 263,200, 20.12.26. Ger., 18.12.25. Addn. to B.P. 242,620; B., 1927, 101).—The sulphoxides or sulphones obtained by oxidising 3-benzanthronyl thioethers, e.g., with persulphates, hydrogen peroxide, or nitric acid (the last also introduces nitrogen), give vat dyes when treated with alcoholic alkali. 3-Benzanthronyl methyl sulphide thus gives a violet vat dye. 3:3'-Dibenzanthronyl sulphide is oxidised and dinitrated by mixed acid, and the diamino-3:3'-dibenzanthronyl sulphone obtained by subsequent reduction yields with alcoholic alkali a violet-blue vat dye. C. HOLLINS.

Manufacture of condensation products of the anthracene series. I. G. FARBENIND. A.-G., Asses. of MEISTER, LUCIUS, & BRÜNING (B.P. 261,400, 12.11.26. Ger. 13.11.25).—Anthranols, oxanthranols, or anthrones are condensed with crotonaldehyde in presence of sulphuric acid in the absence of an oxidising agent; e.g., anthrone gives 1-methylbenzanthrone, m.p. 113—114° (cf. B.P. 244,120; B., 1927, 326). C. HOLLINS.

Manufacture of derivatives of naphthaquinones.

R. LANTZ and A. WAHL, Assrs. to SOC. ANON. DES MAT. COL. & PROD. CHIM. DE ST.-DENIS (U.S.P. 1,666,241, 17.4.28. Appl., 21.1.26. Fr., 21.1.25).—See B.P. 246,482; B., 1927, 39.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Transmission of ultra-violet radiation by various fabrics. H. R. HIRST, P. E. KING, and P. N. LAMBERT (J. Soc. Dyers and Col., 1928, 44, 109—113).—Samples of various fabrics (wool, silk, cotton, and artificial silks) were exposed to the radiation from a mercury arc filtered through a screen of Chance's ultra-violet glass. All fabrics cut off a large proportion of the radiation, the chief controlling factors being the weave and texture of the material, and not the kind of fibre of which it is composed. For equal thicknesses of material, wool has generally the highest transmissive power, whilst dyeing diminishes this power in the case of all fabrics.

B. P. RIDGE.

Cleaning of fur and leather garments. M. H. GOLDMAN and C. C. HUBBARD (U.S. Bur. Stand. Tech. Paper, 360, 1927, 22, 183—197).—Fur and leather goods retain their colour, appearance, and pliability during dry-cleaning if a small percentage (1.25% for leather and 2.5% for furs) of paraffin wax, m.p. 45°, is added to the naphtha. If, as has been found the case in works' practice, it is undesirable to add the paraffin wax to the cleaning liquor, the material, after cleaning in the usual way, may be soaked in the paraffin wax-naphtha mixture for about 5 min., centrifuged, and exposed to warm air at a temperature not above 50°. A standard practice for cleaning fur and leather materials is suggested.

D. J. NORMAN.

Viscose. XIII and XIV. G. KITA, R. TOMIHISA, K. NAKAHASHI, and J. ONOHARA (J. Cellulose Inst. Tokyo, 1928, 4, 25—30; 31—34).—By the addition of dextrose, viscose may be spun after a longer ripening than when ammonium sulphate is used in the bath, whilst increasing the quantity of dextrose slightly shortens the necessary time of ripening. Over-ripe viscose cannot be spun from a bath containing a large quantity of dextrose, the action of the latter being affected by the degree of ripening. For viscose spun from such a bath the tensile strength of the fibres is increased but the extensibility is diminished. In a bath containing 8% of sulphuric acid a less ripe viscose may be spun than in one containing much sodium sulphate, whilst if 12% of sulphuric acid is used the opposite is the case. Sodium sulphate has no effect on the tensile strength and extensibility of the fibres. No special influence is exerted by magnesium sulphate of 6% concentration, whilst with under-ripe viscose, in the initial stages of spinning, a 12% solution increases the tensile strength.

Fibres from a bath containing zinc sulphate are at first coloured yellow, but afterwards decolorised. When 1—3% of the salt is present a less ripe viscose may be spun than when no zinc sulphate is added, but for a 5% solution the opposite holds, *i.e.*, a less ripe viscose is more difficult to spin than when the salt is absent. The influence of zinc sulphate on the properties of the fibres depends on the properties of the viscose, but with a moderately ripened viscose fibres of increased extensibility and tensile strength may be obtained.

B. P. RIDGE.

Decay of oak-wood. A BRANDL (Brennstoff-Chem., 1928, 9, 89—94).—Examination of samples of decayed oak-wood by Odén's alkaline-extraction method (B., 1926, 82, 568) or by the acetyl bromide process (Grosskopf, B., 1926, 939) shows that much of the decayed wood is already humified, a further proportion being converted into decomposition products of lignin, as lignic acid. Values for lignin determined by the sulphurous acid method are about 5% high. The soluble matter (%) extracted from decayed (young) wood from the bark of the tree and from older wood from the trunk by means of cold water, hot water, 5% sodium carbonate solution, 5% sodium hydroxide solution, and 10% sodium hydroxide solutions, respectively, are 4.5, 4.8; 17.1, 10.0; 29.5, 33.1; 25.1, 26.0; 7.3, 3.4; these woods contained 9.3% and 6.9% of cellulose, respectively, and 16.5% and 22.7% of insoluble residue. Precipitation of the sodium hydroxide-soluble fraction with alcohol affords a humic acid closely resembling that obtained by the acetyl bromide process, the chief difference being in the high methoxy content, which is due to absorbed alcohol. The carbonyl content is a little higher than that of the original decayed wood (2.66—2.68; methoxyl 10.95). The calcium salt has equivalent 318, corresponding with that of the humic acid isolated by Odén from coal. Nitration of the humic acid affords a product (impure) containing 47.81% C, 3.73% H, 3.99% N, and closely resembling the corresponding product from the humic acid of coal, together with a yellow "nitrophenolic" substance, 42.38% C, 2.53% H, resembling that described by Tropsch and Schellenberg

(B., 1924, 323). The fraction soluble in sodium hydroxide and in alcohol (59.84% C, 4.76% H) has acid value 70.73 (cold), 88.86 (hot), carbonyl value 2.63, methoxyl value 11.34 (due to absorbed alcohol), and is probably a humatmelanic acid, contaminated with tannins and other soluble products. It decomposes slowly on drying at 70—80%, more rapidly at 105—110° (cf. Eller and Schöppach, B., 1926, 257). The heats of combustion of the products isolated by the alkaline extraction process fall fairly regularly with the carbon content. The methoxy-content of the products tends to decrease, the carbonyl value to increase as compared with the original decayed wood, and the ease with which these products undergo oxidation or condensation and polymerisation is attributed to the presence of ketonic or aldehydic groups. These results indicate that a part of the lignin has already been converted into humic acid in the decayed wood, and support the view advanced by Fischer and Schröder that coal is essentially a decomposition product of lignin. R. BRIGHTMAN.

Position of researches on [the constitution of] lignin. K. G. JONAS (Papier-Fabr., 1928, 26, 221—229).—A review of the constitutional formulae ascribed to lignin by various authors. The formula proposed by Klason (A., 1923, i, 187), which represents lignin as a condensation product of coniferylaldehyde, is rejected on the grounds that it does not explain why one of the two methoxyl groups is very readily hydrolysed whereas the other is difficult to attack; that a substance so constituted should yield large quantities of aromatic substances on oxidation, whereas only very small yields of such products are obtainable from lignin; and that it is not in accordance with the fact that most analyses of lignin indicate the presence of oxygen atoms (usually 3 per 20 carbon atoms) other than those in hydroxyl, alkoxy, and carboxyl groups. The author favours the formula of Schrauth (A., 1923, i, 443) and regards lignin as a complex structure which contains tetrahydrofuran and cyclohexenol rings and is derived from a hexose by dehydration and subsequent reduction. The formula proposed—a modification of Schrauth's—explains more satisfactorily the difference in the rates of hydrolysis of the two methoxyl groups and includes an acetyl group which has been shown to be present in natural lignin. W. J. POWELL.

Edible cellulose. HARDING.—See XIX.

PATENTS.

Protection of animal fibres etc. against the effects of alkaline or acid media. BRIT. DYESTUFFS CORP., LTD., R. S. HORSFALL, and L. G. LAURIE (B.P. 285,554, 16.11.26).—A soluble condensation product of carbamide with an aldehyde, *e.g.*, dihydroxydimethyl-carbamide [dimethylolurea], is added to acid or alkaline baths to protect wool, silk, fur, etc. from injury in the bath. Examples are the scouring of wool, dyeing silk with vat dyes, "killing" furs, dyeing worsted with acid colours, and mordanting wool. C. HOLLINS.

Sizing of fibres. G. M. CLARK. FROM AGASOTE MILLBOARD Co. (B.P. 286,948, 2.7.27).—A more efficient and even distribution of the sizing agent over the surface of the fibres is obtained if tannic acid is added to the

beater before precipitation of the size by the addition of alum. Thus, in the manufacture of waterproof pulp boards, the stock may be sized with 11—13% of sodium linoleate to which has been added 10—20% of its weight of tannic acid, preferably in the form of a commercial tanning extract. D. J. NORMAN.

Manufacture of waterproof compositions. C. G. A. LUNDBERG (B.P. 271,076, 10.5.27. Swed., 11.5.26).—Roved wool or cotton, or waste material from wool and cotton manufacture, is first purified, e.g., by treatment with dilute sulphuric acid for about 12 hrs., and, after washing and drying, is impregnated at about 80° for, e.g., 30 min. with an acid solution containing a zinc salt, e.g., a solution containing 78 pts. of zinc chloride, 14 pts. of zinc sulphate, and 8 pts. of nitric acid, which has been boiled for about 2 hrs. before use. The treated material, after removing the excess of liquor, is dried, disintegrated, and mixed, optionally after the addition of fillers, with a rubber solution prepared by adding a solution of waste rubber in linseed oil to molten asphalt, rosin, etc. The resulting mixture is moulded under heat and pressure and is finally vulcanised.

D. J. NORMAN.

Method of laundering. J. TAKAMINE and J. TAKAMINE, JUN., Assis. to TAKAMINE FERMENT CO. (U.S.P. 1,660,458, 28.2.28. Appl., 17.12.21).—Starch, gums, oils, etc. are removed from the goods by treatment with tepid water containing diastatic and proteolytic enzymes derived by extraction of a vegetable fungus, e.g., *Aspergillus oryzae*. T. S. WHEELER.

Production of cellulose and other chemical products. H. WALLIN (U.S.P. 1,660,696, 28.2.28. Appl., 28.9.25. Sweden, 8.10.24).—In the manufacture of cellulose by cooking wood with an alkali silicate solution, the spent liquor is regenerated by evaporation and calcination of the residue at 700°, sodium carbonate and silicic acid being added to make up loss of alkali silicate. T. S. WHEELER.

Opening-up of materials containing cellulose. I. G. FARBENIND. A.-G. (B.P. 279,036, 26.8.27. Ger., 13.10.26. Addn. to B.P. 274,892; B., 1928, 154).—The material, e.g., moist fir wood, is treated for one or more hours with a mixture of air and oxides of nitrogen at 40—50°, and is then exposed to the action of air alone for about 24 hrs. at the ordinary temperature or at about 50°. D. J. NORMAN.

Treatment of celluloid scrap. RHEINISCHE GUMMI-CELLULOID-FABR. (B.P. 277,626, 9.7.27. Ger., 18.9.26).—The comminuted or pulverised material is intimately mixed with, e.g., 2% of a non-volatile solvent for nitro-cellulose (triphenyl phosphate, ethyl phthalate, acetanilide, ethylacetanilide, ethyl-*p*-toluenesulphonamide) and is then moulded under heat and pressure. The moulding may be done at relatively low temperatures and the resulting products are homogeneous and of but slightly reduced hardness. D. J. NORMAN.

Manufacture of gut strings. T. SARRA (U.S.P. 1,664,323, 27.3.28. Appl., 22.3.26).—Bovine intestines are treated with caustic alkali solution to separate the inner and outer layers; the inner layer, after further treatment with caustic alkali solution to strengthen it, is then twisted into gut strings. D. J. NORMAN.

Sizing of paper. J. A. DE CEW (U.S.P. 1,663,976, 27.3.28. Appl., 22.9.26).—The rosin size is precipitated with aluminium sulphate that has been dissolved in water and made basic. D. J. NORMAN.

Production of permanent-finish vegetable [fibrous] material. Vegetable yarn and fabric with wool-like effects. A. BODMER, Assr. to HEBERLEIN PATENT CORP. (U.S.P. 1,666,082—3, 17.4.28. Appl., [A] 11.4.23, [B] 12.4.23. Ger., 15.4.22).—See B.P. 196,298; B., 1924, 332.

Production of artificial silk. W. H. GLOVER and C. F. TOPHAM, Assrs. to COURTAULDS, LTD. (U.S.P. 1,665,958, 10.4.28. Appl., 29.10.26. U.K., 7.1.26).—See B.P. 268,455; B., 1927, 472.

Production of artificial silk filaments by drawing. K. GRUNERT (U.S.P. 1,665,453, 10.4.28. Appl., 4.10.26. Ger., 8.10.25).—See B.P. 263,462; B., 1927, 138.

After-treatment of freshly-spun artificial silk. F. J. GAHLERT (U.S.P. 1,666,090, 17.4.28. Appl., 28.7.26. Ger., 9.6.25).—See B.P. 283,752; B., 1928, 228.

Production of cellulose by decomposition of vegetable fibres. O. C. STRECKER (B.P. 284,846, 13.1.27).—See U.S.P. 1,658,213; B., 1928, 295.

Hydrolysis of cellulosic substances. SOC. DES BREVETS ÉTRANGERS LEFRANC & CIE. (B.P. 271,410, 11.2.27. Fr., 20.5.26).—See F.P. 616,424; B., 1928, 187.

Manufacture of a cellulose-derivative film. H. J. HANDS, Assr. to SPICERS, LTD. (U.S.P. 1,666,377, 17.4.28. Appl., 25.1.27. U.K., 24.9.26).—See B.P. 281,803; B., 1928, 187.

Stencil sheet. S. HORII (U.S.P. 1,664,777, 3.4.28. Appl., 14.1.27).—See B.P. 250,798; B., 1926, 534.

Manufacture of a composite adhesive [for sand paper]. F. G. OKIE, Assr. to MINNESOTA MINING & MANUF. CO. (U.S.P. 1,659,611, 21.2.28. Appl., 4.3.22).—See B.P. 219,341; B., 1924, 905.

Machines for [stretch-spinning] manufacture of artificial textile threads etc. H. KINDERMANN (B.P. 287,602, 23.11.26).

Derivatives from ligninsulphonic acid (B.P. 286,808).—See III.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Colouring of leather. LAMB.—See XV. **Dye absorption of soils.** CROUCHER.—See XVI.

PATENTS.

Dyeing, printing, or stencilling of materials composed of or containing cellulose ethers. H. DREYFUS (B.P. 285,104, 8.10.26).—The methods of the prior patents for the dyeing etc. of acetate silk are extended to the dyeing etc. of cellulose ethers, such as methyl-, ethyl-, and benzyl-celluloses. C. HOLLINS.

Dyeing, printing, and stencilling of materials made with or containing cellulose derivatives. BRIT. CELANESE, LTD., and H. C. OLPIN (B.P. 285,641, 9.2.27).—Cellulose esters and ethers, including immunised

cotton, are dyed etc. by means of β -sulphatoethyl derivatives of aminoanthraquinones. 1- β -Sulphatoethyl-aminoanthraquinone gives a scarlet, and the corresponding derivatives of 1-amino-2-methyl- (scarlet), 1-amino-4-hydroxy- (violet), 1:4-diamino- (mono-, blue-violet; di-, pure blue), 2-bromo-1:4-diamino- (mono-, blue-violet), 1-amino-4-methylamino- (pure blue), and 1:5-diamino- (di-, bluish-red) -anthraquinones are also described.

C. HOLLINS.

Dyeing of fur. A. WINOGRADOFF, Assr. to INECTO, INC. (U.S.P. 1,660,826, 28.2.28. Appl., 1.4.26).—The fur is treated with dilute silver nitrate solution, and, after exposure to light, is immersed in *p*-phenylenediamine solution to develop opalescent colours. T. S. WHEELER.

Toning colours in fabrics. I. B. FUNK (U.S.P. 1,659,598, 21.2.28. Appl., 15.9.24).—A coloured fabric comprising a wool nap and a cotton warp is treated with a paste of diatomaceous earth and dilute sulphuric acid. The capillary attraction of the nap removes the acid from the paste, and the warp is not attacked.

T. S. WHEELER.

Treatment of printed or dyed fabrics. R. W. JACOBY, Assr. to DELTEX Co. (U.S.P. 1,663,845—6, 27.3.28. Appl., [A] 3.5.26, [B] 13.8.27).—(A) In the ageing or reduction of printed or dyed fabrics, moisture is supplied in the vicinity of the exothermic reactions so that its evaporation absorbs heat and maintains the heat-carrying medium as a saturated vapour. (B) Suitable apparatus is described.

F. G. CLARKE.

Colour printing on textile fabrics. SOC. ANON. DES ÉTABL. PETITDIDIER (ANC. MAISON JOLLY-BELIN) (B.P. 261,399, 12.11.26. Fr., 12.11.25).—Immunised cotton, with or without viscose silk, with a backing of cotton, wool, silk, etc. is vinted with a cellulose acetate dye, or an acid dye having affinity for immunised cotton, or a basic dye in conjunction with tannin or acetanol, and steamed to fix the colour on the immunised cotton. The viscose and the backing are then discharged to white by rinsing with water followed by 0.1% sodium hyposulphite at 75°. The last traces of colour may be removed from a silk backing by means of dilute acid.

C. HOLLINS.

Production of pattern effects in fabrics. H. MÜLLER, Assr. to A.-G. CILANDER (U.S.P. 1,660,042, 21.2.28. Appl., 25.2.26. Ger., 20.1.26).—A mixed fabric containing animal and vegetable fibres is printed with an alkaline paste and steamed to destroy the animal fibres.

T. S. WHEELER.

Dyeing preparation for cellulose esters. R. METZGER, Assr. to GRASELLI DYESTUFF CORP. (U.S.P. 1,666,715, 17.4.28. Appl., 4.3.26. Ger., 7.5.25).—See B.P. 271,550; B., 1927, 553.

Preparation of colour pastes for colour printing. M. FREIBERGER and A. HOLTSMANN (U.S.P. 1,665,162, 3.4.28. Appl., 25.10.24. Ger., 14.12.23).—See B.P. 226,164; B., 1925, 879.

[Machines for] the fluid treatment of artificial silk yarns. J. BRANDWOOD (B.P. 287,731, 19.4.27).

Protection of animal fibres (B.P. 285,554).—See V. **Coloured rubber** (U.S.P. 1,663,436).—See XIV.

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

Determination of the caustic value of lime. Two **filtration devices.** W. M. SHAW, W. H. MACINTYRE, and J. E. UNDERWOOD (Ind. Eng. Chem., 1928, 20, 312—314).—The slow settling of magnesia suspensions in sucrose solution renders the following method of filtration without contact with carbon dioxide preferable in the analysis of magnesian limes. The ground lime is shaken with sugar solution in a 500 c.c. Erlenmeyer flask, the stopper of which carries a soda-lime tube and a siphon connexion. To the latter and within the flask is connected a filter cone made from a cut-down pipette and packed with cotton covered by macerated filter paper. Aliquot parts are drawn for titration by the siphon. Any moisture in the filter is removed by the first aliquot. Another type of apparatus consists of a flask with protected air connexion and measuring pipette with 3-way stopcock at each end. Connected to the latter and within the flask is an alundum filtering thimble. The filtrate is drawn up into the pipette by suction (cf. following abstract). C. IRWIN.

Effects of certain impurities on the determination of causticity of limes by modifications of the sugar and Scaife methods. W. M. SHAW and W. H. MACINTYRE (Ind. Eng. Chem., 1928, 20, 315—319).—The filtration devices described in the preceding abstract were used in this investigation. Magnesia carefully freed from lime was without effect on the determination of caustic lime with sugar, but lowered the value found by the Scaife method. Added silica decreased the values found in both cases, no doubt by silicate formation, but greater decreases occurred in the sugar method. If a mixture of lime, magnesia, and silica is calcined, some of the silica always passes into combination with lime. A similar result was obtained by igniting natural magnesium silicate (serpentine) with lime. Addition of calcium silicate was without effect in the time required for determination, but reduced causticity in 24 hrs.' contact. Alumina also causes some reduction in causticity, whilst added calcium aluminate increases it by hydrolysis.

C. IRWIN.

Determination of free alkali in hypohalogenite solutions. E. RUPP and F. LEWY (Z. anal. Chem., 1928, 73, 283).—The determination of free alkali in solutions of hypochlorite or hypobromite is carried out by titration with standard acid in the presence of methyl-orange after first destroying the hypohalogenite by addition of hydrogen peroxide.

A. R. POWELL.

Determination of small amounts of carbon monoxide in air. G. M. EDELL (Ind. Eng. Chem., 1928, 20, 275).—Samples of air containing known amounts of carbon monoxide from 0.001% to 0.06% were analysed by the iodine pentoxide method. It was found that only the purest iodine pentoxide could be used. Even then with pure air a constant though small liberation of iodine occurred, but it could be corrected for and the results obtained were of fair accuracy.

C. IRWIN.

Ammoniacal nitrogen of peats and humus soils. ELLIS and MORISON. **Dusting mixtures.** SWINGLE.—

See XVI. **Fumigation.** BUTTENBERG and GAHRTZ.—
See XXIII.

PATENTS.

Manufacture of sulphuric acid. SOC. GÉN. MÉTALL. DE HOBOKEN (B.P. 284,208, 16.12.27. Fr., 24.6.27).—The consumption of nitric acid is reduced when using cold sulphurous gases to manufacture sulphuric acid having d 1.7 or less by introducing the cold gases into the plant beyond the denitration apparatus, the denitration of the acid being effected by hot sulphurous gases in a tower filled with irregularly-shaped filling material of smaller diameter than 40 mm. W. G. CAREY.

Manufacture of sulphuric acid. A. SHARP. FROM SOC. GÉN. MÉTALLURGIQUE DE HOBOKEN (B.P. 286,865 and 286,980, [A] 19.2.27, [B] 23.8.27).—(A) When manufacturing sulphuric acid from sulphur dioxide of varying temperature and concentration a single reaction chamber is used of such shape that maximum volume is obtained for minimum surface and is filled with packing having a high heat capacity, the temperature being kept constant by varying the temperature of the circulating acid. (B) Gases with a varying sulphur dioxide content are first treated by any known contact process and the remaining sulphurous gases are treated in towers, which are rinsed with nitrosylsulphuric acid. W. G. CAREY.

Preparation of vanadic acid. H. G. C. FAIRWEATHER. FROM SELDEN CO. (B.P. 287,401, 14.9.27).—Impure alkali vanadate solutions are treated with alkali carbonates and with oxidising agents, such as chlorine, bromine, or peroxides, to convert all the vanadium into the quinquevalent state and to precipitate metals which form insoluble carbonates and peroxides. If arsenic and phosphoric acids are present magnesium chloride is added to the acidified filtrate and the solution is further oxidised with nitric acid or a halogen, made just ammoniacal, and again filtered after 8–10 hrs. The precipitate contains arsenic, phosphorus, iron, and silica; the filtrate is heated to boiling, saturated with ammonium chloride, and cooled, and the ammonium metavanadate which crystallises out is collected, washed, and ignited at 200–300° in air, then at 300–400° in oxygen to obtain pure vanadium pentoxide free from lower oxides. A. R. POWELL.

Drying, neutralising, and delivering ammonium salts and other granular substances. INTERNAT. CEMENT-GUN CO., G.M.B.H., Assees. of F. BINSWANGER (B.P. 277,652, 8.9.27. Ger., 14.9.26).—The substance is fed into a drying chamber having superposed trays and is caused to fall from tray to tray by rotating, gradually descending scrapers; heated air mixed with neutralising gases if necessary is passed upward in a zig-zag path. The dried material is thrown by centrifugal action on to the rim of a rotating disc and is discharged by an air blast. W. G. CAREY.

Magnesia [from dolomite]. Refractory magnesia compounds. W. KOEHLER (U.S.P. 1,664,630—1, 3.4.28. Appl., [A] 21.12.23, [B] 15.3.26).—(A) A mixture of dolomite and magnesium chloride is calcined to obtain magnesia, calcium chloride, and carbon dioxide. The calcium chloride is leached out, the solution treated with the oxides obtained by calcining a further quantity of

dolomite alone and saturated with carbon dioxide, and the magnesium chloride thus regenerated separated from the calcium carbonate precipitate and used again in the process. (B) Finely-divided raw dolomite is treated with magnesium chloride equivalent to its lime content, the mixture heated until it is dehydrated, and the calcium chloride leached out. The residual magnesia is calcined to obtain a refractory. A. R. POWELL.

Production of high-grade magnesium oxide. W. KOEHLER (U.S.P. 1,661,043, 28.2.28. Appl., 15.10.27).—Dolomite is heated with magnesium chloride at 700° to yield a mixture of calcium chloride and magnesium oxide which is extracted with water. T. S. WHEELER.

Recovery of zinc from solutions [of zinc salts]. S. C. SMITH (B.P. 287,186, 18.9.26).—Purified zinc chloride or sulphate solutions obtained from the leaching of zinc ores or products are treated with an excess of ammonia, and carbon dioxide is passed in with agitation until the zinc is precipitated as a basic carbonate containing 3–4% of ammonia. This precipitate is granular and may readily be separated from the mother-liquor; on boiling with water it slowly loses all its ammonia and on calcination is converted into a pure white zinc oxide. A. R. POWELL.

Manufacture of aluminium chloride. G. S. TILLEY (U.S.P. 1,661,100, 28.2.28. Appl., 10.7.24).—Anhydrous aluminium sulphate is mixed with charcoal and treated at 500° with hydrogen chloride. T. S. WHEELER.

Decomposition of zirconium ores. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G. (B.P. 287,424, 2.11.27. Addn. to B.P. 282,023; B., 1928, 337).—The sintered mass obtained by heating zirconium ores with lime and calcium chloride or fluoride is treated with less than the theoretical quantity of hydrochloric acid required to combine with all the bases, and the concentrated solution so obtained is filtered and treated with an excess of concentrated hydrochloric acid to precipitate zirconium oxychloride. A. R. POWELL.

Manufacture of hydrogen peroxide. A. E. VIDAL. FROM J. D. RIEDEL A.-G. (B.P. 287,281, 4.2.27).—Hydrogen peroxide is distilled from an acid persulphate solution etc. by introducing it through a capillary tube into the lower end of a narrow vertical tube connected at the other end with a vacuum. The tube, or system of tubes, is made of chromium-nickel alloy or other chemically-resistant heavy metal, and heat is supplied internally or externally by steam, hot gases, or electrical means. W. G. CAREY.

Production of phosphorus and aluminous cement. I. G. FARBENIND. A.-G. (B.P. 287,036, 2.2.28. Ger., 19.3.27).—A mixture of bauxite, rich in iron, and lime in the proportion of 70–90 pts. of alumina to 30–10 pts. of lime is fused under reducing conditions in an electric or shaft furnace, and the easily fusible, almost iron-free slag obtained is then fused under reducing conditions with crude phosphates. W. G. CAREY.

Recovery of bromine [from saline solutions]. G. EDGAR, Assr. to ETHYL GASOLINE CORP. (U.S.P. 1,662,305, 13.3.28. Appl., 13.10.25).—The solutions

are chlorinated and the liberated bromine is converted into an insoluble compound. F. G. CLARKE.

Isolation of argon. G. R. FONDA, Assr. to GEN. ELECTRIC Co. (1,664,205, 27.3.28. Appl., 19.3.24).—Air is partially liquefied, the liquid being less than a half of the whole, and the liquid rectified so as to obtain liquid oxygen and a distillate rich in argon. The latter is re-liquefied and rectified, in order to obtain an argon concentrate. F. G. CLARKE.

Manufacture of catalysts. H. NIELSEN, Assr. to B. LAING (U.S.P. 1,665,175, 3.4.28. Appl., 7.3.25. U.K., 3.12.21).—See B.P. 198,385; B., 1923, 760 A.

Zinc oxide [for rubber compounding]. F. G. BREYER, E. H. BUNCE, and J. H. WEIKEL, Assrs. to NEW JERSEY ZINC Co. (U.S.P. 1,664,767, 3.4.28. Appl., 18.6.25).—See B.P. 253,875; B., 1927, 107.

Separation of hafnium and zirconium. [A] D. COSTER and G. VON HEVESY, [B] A. E. VAN ARKEL and J. H. DE BOER, [C] J. H. DE BOER, Assrs. to N. V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,666,440, 1,666,800, and 1,666,811, [A—C] 17.4.28. Appl., [A] 15.7.24, [B] 11.11.24, [C] 10.8.26. [A] Denm., 18.7.23, [B] Holl., 12.12.23, [C] 12.9.25).—See B.P. 219,327, 226,180, and 258,243; B., 1925, 499; 1926, 273; 1927, 482.

Catalysts (F.P. 621,434).—See III.

VIII.—GLASS; CERAMICS.

Antimony enamels. K. BECK and W. A. SCHMIDT (Z. Unters. Lebensm., 1928, 55, 1—24).—The use of antimony compounds, especially sodium metantimonate (Leukonin), in the enamel industry is discussed. The solubility of antimony from the frit and from the enamel of cooking ware in 3% tartaric acid solution was measured, a process for the accurate determination of small quantities of mixed ter- and quinque-valent antimony being used. For enamelled ware the method is as follows:—200 c.c. of 3% tartaric acid solution are added for each 1 litre volume of the vessel, the latter is covered with a round-bottom flask filled with cold water, and the liquid boiled for $\frac{1}{2}$ hr. over a free flame with frequent shaking. After cooling, the solution is diluted to 200 c.c. To 50 c.c. are added 20 c.c. of hydrochloric acid (*d* 1.126) and 1 drop of methyl-orange solution (1 in 500), and the solution is titrated with 0.001N-potassium bromate solution till colourless. The titration, less a blank, multiplied by 0.0609 gives the trivalent antimony in mg. per 50 c.c. For the total antimony the solution (50 c.c.) is mixed with 20 c.c. of hydrochloric acid (*d* 1.126)—or the solution already titrated may be employed—and heated nearly to boiling. After removal of the flame 10 drops of 10% phosphotungstic acid are added, followed by sufficient approximately 0.1N-titanous chloride solution to give a cobalt-blue colour persisting for 2 min. After 3 min. 2 drops of 0.01% copper sulphate solution are added, and the solution should then be colourless. After a further 5 min. 1 drop of methyl-orange solution is added and the red-coloured solution titrated till colourless with 0.001N-potassium bromate. The titration, less a blank, multiplied by 0.0609 gives the total antimony in mg. per 50 c.c. J. R. NICHOLLS.

Effect of firing on the chemical and physical properties of refractories. W. MIEHR, J. KRATZERT, and H. IMMKE (Tonind.-Ztg., 1928, 52, 280—282, 298—301, 323—325).—The relation between chemical composition of refractories and mullite formation at or above 1000° is discussed briefly. The mullite formation between 1000° and 1600° was determined by chemical means in 12 clays of different chemical and mineralogical composition, and the relation between mullite content and the temperature and duration of firing, grain size of the material, clay-substance content, and alkaline flux content was studied. The results were confirmed and extended by microscopical and X-ray investigations. Mullite is formed most readily, *i.e.*, at relatively low temperatures, in clays rich in fluxes and poor in clay substance; in this case its formation can be facilitated by fine grinding of the material. A long firing period at a very high temperature is necessary to produce mullite in clays rich in clay substance and poor in fluxes; an increase in the flux content of such clays lowers the temperature of mullite formation somewhat. The effect of mullite formation and the accompanying structural changes on the crushing strength, thermal expansion (resistance to spalling), and the resistance to chemical action (slags) of refractories is discussed. F. SALT.

Properties of magnesia bricks. H. HIRSCH (Arch. Eisenhüttenw., 1927, 1, 439—444; Stahl u. Eisen, 1928, 48, 371—372).—The physical, mechanical, and refractory properties of magnesite bricks from 32 different sources have been determined, and from the results obtained the samples have been divided into three groups, *viz.*, those containing more than 7%, those with 3—7%, and those with less than 3% Fe₂O₃. These groups may again be subdivided into nine sub-groups the characteristics of which are tabulated. Magnesite bricks containing only silica as an impurity have a softening point under load of about 1790°, which approaches that of bricks made of sintered magnesia; they have a high true sp. gr. and low porosity, but a relatively high coefficient of expansion. Although the behaviour of the bricks under load varied greatly with the composition, the refractoriness varied only slightly, all the samples withstanding exposure to a temperature above cone 42. X-Ray examination of well-burnt bricks showed that practically the whole of the magnesia has been converted into periclase, and therefore no further transformation should take place even after prolonged heating. Bricks containing 5—7% Fe₂O₃ and very small amounts of silica have the best sintering properties and the greatest mechanical strength.

A. R. POWELL.

Composition of "Kochi-stone." FUKUCHI and WATANABE (J. Geol. Soc., 1925, 32, 131—141).—This refractory material contains SiO₂ 24.68, Al₂O₃ 61.80, Fe₂O₃ 0.60, CaO 0.60, MgO 0.10, F 5.18, Cl 2.91, H₂O 6.15%.

CHEMICAL ABSTRACTS.

PATENTS.

Kilns for firing pottery, tiles, etc. T. SIMPSON (B.P. 285,935, 21.10.26).—The fire mouths of the kiln extending underneath the kiln floor are each divided by a short central midfeather, and have a set of up-

flues at the front and back to convey the heat from each mouth to the kiln crown. Passages in the midfeathers direct the heat along the crown to the back down-flues, which convey the heat underneath the kiln, whilst up-flues at the front of the kiln convey the heat to a conduit leading to a chimney. A damper on the outside wall of the kiln facilitates cooling and allows the kiln to be drawn in a shorter time than usual. W. G. CAREY.

Apparatus [tunnel kiln] for firing ceramic and other products. C. T. HOLCROFT and C. E. DOLL (U.S.P. 1,662,063, 13.3.28. Appl., 1.12.24).—In a tunnel kiln of the muffle type the air is circulated in direct contact with the articles as they advance through the kiln. The air supply, which is under pressure, is near one end of the tunnel. F. G. CLARKE.

Manufacture of appliances and articles of highly refractory material such as oxide of zirconium, of thorium, and of the rare earths. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (B.P. 280,907, 12.11.27. Ger., 22.11.26).—Articles of zirconium oxide, thorium oxide, etc. are improved in solidity and density if the mixture of sintered and unsintered material is heated as rapidly as possible to 1300° or above. W. G. CAREY.

Crucibles [for molten metals]. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of M. UNGER (B.P. 278,367, 28.9.27. U.S., 1.10.26).—Leakage of molten metal through porous fireclay crucibles is prevented by impregnating them, preferably *in vacuo*, with a liquid, e.g., coal-tar pitch, containing a refractory substance in suspension or solution, and slowly heating them to drive off the volatile components and leave carbon in the pores of the crucible. W. G. CAREY.

Manufacture of chamotte materials. L. KERN (U.S.P. 1,664,689, 3.4.28. Appl., 13.10.25).—A grog material suitable for use in chamotte is produced by burning at a strong glowing heat a plastic mass of kaolin, combustible organic matter, and a binder, all in a finely-divided state, and then pulverising the product. H. ROYAL-DAWSON.

Compositions for heat-insulating and heat-resisting purposes. L. F. TOOTH (B.P. 287,363, 22.6.27).—The composition, which can be shaped into bricks or used *in situ*, comprises Portland cement or fireclay, slagwool, and crushed burnt firebrick (grog), e.g., in equal pts. by wt., with the addition of 0.33–1.0% of aluminium powder or similar material, which on heating causes the evolution of gas and the formation of a porous product. L. A. COLES.

Manufacture of pottery etc. J. W. MELLOR (U.S.P. 1,666,828, 17.4.28. Appl., 14.1.26. U.K., 9.3.25).—See B.P. 253,184; B., 1926, 667.

Glass furnaces. HARTFORD-EMPIRE Co., Assees. of P. G. WILLETTS (B.P. 275,555, 22.3.27. U.S., 3.8.26).

Manufacture of imitations of frosted glass, glass stainings, etc. R. GOLDSCHIEDER and I. TUSCHINSKY (B.P. 286,012, 29.11.26).

Ovens of the direct flame continuous tunnel type. J. WILLIAMSON (B.P. 287,623, 22.12.26).

Treatment of sludge (B.P. 286,855).—See I. **Magnesia refractory** (U.S.P. 1,664,631).—See VII.

IX.—BUILDING MATERIALS.

Electrical method for measuring the setting time of Portland cement. Y. SHIMIZU (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 85–92).—On measuring the electrical conductivity of gauged cement during setting and hardening, the specimen being maintained at constant temperature, the resistance suddenly changed at the moment corresponding to the final set, as defined by the maximum temperature change. The change in resistance is used to determine the time of setting, the mixed cement being contained in a small tube in a thermostat; two platinum electrodes are immersed in the cement and connected to a Kohlrausch alternating-current bridge. The rate of setting and hardening is influenced greatly by temperature, but the method is considered better adapted and more accurate than the mechanical and thermal methods. C. A. KING.

Formation and dissolution of calcium hydroxide crystals in Portland cement. J. O. DRAFFIN (Ind. Chem., 1928, 20, 311).—After the setting of Portland cement, crystals of calcium hydroxide gradually form, originating from the hydrolysis of tricalcium silicate. In a study of this process on microscope slides such crystals later were found to redissolve, apparently owing to slight dissolution of the glass of the slide. No such dissolution could be produced with natural soda-lime felspar. C. IRWIN.

Mechanism of the action of wood-preserving agents. DEHNST (Z. angew. Chem., 1928, 41, 355–358).—To test Bateman's theory (Proc. Amer. Wood Preservers' Assoc., 1920–1924) that the fungicidal action of coal-tar oil or creosote oil is due to the low-boiling hydrocarbons and the high-boiling tar acids and bases and is not influenced in any way by the 40% of neutral oil usually present, a very pure German coal-tar oil was freed from acids and bases and separated into several fractions by distillation, and the action of the various products on four of the ordinary varieties of wood fungus was examined. All the products obtained had a strong fungicidal action on *Polyporus vaporarius* and *Coniophora cerebella*, but the neutral oil boiling above 288° had little action on *Lenzites abietina* and *Fomes annosus*, although the remainder of the oil had a strong fungicidal action on these varieties. A. R. POWELL.

PATENTS.

Burning of cement. A. ANDREAS (B.P. 263,166, 15.12.26. Ger., 16.12.25).—Air is blown at a relatively high pressure into a kiln for burning cement at a point directly below the clinkering zone and also, at the same pressure, at lower points, including one below the grinding rollers, if desired. The air is introduced through pipes surrounding the kiln at suitable heights, fitted with branch pipes at regular intervals leading to the interior of the kiln. The kiln tapers towards the grinding rollers or outlet at the lower end. L. A. COLES.

Brick making. G. H. HADFIELD (B.P. 286,772, 7.12.26).—Clinker relatively free from carbonaceous material recovered from boiler ash, house refuse, etc. is used as opener-out in the manufacture of bricks from plastic clay. L. A. COLES.

Manufacture of acid-proof cementing compositions. I. G. FARBENIND, A.-G. (B.P. 281,689, 1.12.27. Ger., 1.12.26. Addn. to B.P. 258,616; B., 1927, 333).—In place of silicon an alloy of silicon with, *e.g.*, magnesium, aluminium, or calcium, which consists partly of pure silicon or of silicon containing other constituents in solid solution, is used. L. A. COLES.

Filling cracks in the brickwork of ovens. G. CAPIAU, M. GAUQUIER, and L. LAHAUT (B.P. 286,933, 7.6.27).—A dry, finely-powdered mixture of refractory cement, a flux (*e.g.*, dextrin or borax), litharge, sodium chloride, and sodium carbonate is forced into cracks in the brickwork of coke ovens etc. while the brickwork is still hot. L. A. COLES.

Manufacture of asphaltic, bituminous, or tarry emulsions. SOC. ANON. "LA TRINIDAD" (B.P. 264,496, 11.1.27. Fr., 12.1.26).—An anhydrous mastic prepared by adding a mineral powder, *e.g.*, cement or powdered limestone, to products such as asphalt, bitumen, or pitch, is emulsified by mixing with a suitable agent such as soap. The product is suitable for the construction of roads. A. B. MANNING.

Preservation of timber. H. C. GARDINER (B.P. 286,886, 19.3.27).—The portion of wooden posts etc. buried in the ground is protected against rot by placing in contact with it a slightly soluble powdered preservative containing arsenic, *e.g.*, arsenic trioxide or arseniferous smelting residues, and surrounding it with a pervious covering so that the slow percolation of water causes continuous impregnation of the wood and sterilises the surrounding soil. Deliquescent substances, *e.g.*, sodium chloride, magnesium chloride, etc., or compounds capable of increasing the solubility of the material, *e.g.*, sodium carbonate, may be added to the preservative. L. A. COLES.

Preservation of wood, timber, etc. G. GUNN (B.P. 286,892, 25.3.27. Addn. to B.P. 273,007; B., 1927, 655).—The timber, treated as described in the prior patent, is drained and exposed to the action of steam. L. A. COLES.

Manufacture of burnt building materials etc. of clay. H. GROŃROOS (U.S.P. 1,664,815, 3.4.28. Appl., 4.8.25. Denm., 6.8.24).—See B.P. 238,229; B., 1926, 632.

Production of artificial stone and artificial wood. F. TRACHSEL, J. WIEDMER, and P. ZIGERLI (U.S.P. 1,665,148, 3.4.28. Appl., 3.5.26. Switz., 1.5.26).—See B.P. 280,104; B., 1928, 93.

Making of road surfaces. M. LÉVY, and PRODORITE S.A. (B.P. 273,759, 2.7.27. Ger., 2.7.26).

[Emulsification] apparatus for dealing with bitumen for use as a road material etc. H. E. WARSOP (B.P. 287,257, 1.1.27).

Aluminous cement (B.P. 287,036).—See VII.

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

Magnetic concentration of iron ores of Alabama. High-silica red ores. High-silica grey hæmatite of Talladega county. Flue dust of the Birmingham

district. O. LEE, B. W. GANDRUD, and F. D. DE VANEY (U.S. Bur. Mines, Bull. 278, 1927, 1—33, 35—56, 61—75).—Results with high-silica red ores closely approached theoretical limits. Flow-sheets are given. Roasting in a reducing atmosphere at 500—600° converts the hæmatite into magnetite, often eliminating the necessity for fine grinding. The ores are mostly oolitic hæmatites. Treatment of the grey ore, a mixture of hæmatite and magnetite, is described; 94—95% of the iron is extracted. The treatment of flue dust is described. CHEMICAL ABSTRACTS.

Magnetic concentration of iron ores of Alabama. Flotation of limestone from siliceous gangue. O. LEE (U.S. Bur. Mines, Bull. 278, 1927, 57—59).—The results are tabulated; equal quantities of oleic acid and cresol are used as flotation agents. CHEMICAL ABSTRACTS.

Influence of occluded oxygen in steel on the carburising quality of steel. K. INOUE (Mem. Coll. Eng. Kyushu Imp. Univ., 1928, 5, [1], 69 pp.).—Samples of mild steel, prepared by melting the metal *in vacuo* and giving products containing 0.04—0.23% O as determined by Oberhoffer's method, were carburised. The critical points of steel were not affected by change in the oxygen content, but the specific resistance increased with increasing oxygen, though not following Mahler's formula. Grain growth was noted in steels containing 0.116% O or more. The carburising experiments were made to test the view that a larger grained structure facilitates the penetration of carbon; the results supported the theory and failed to confirm the view that the presence of oxygen generally induces shallow case-hardening. C. A. KING.

Equilibria in the reduction, oxidation, and carburisation of iron. R. SCHENCK (Stahl u. Eisen, 1928, 48, 269—271, 274—276). E. MAURER and W. BISCHOF (*ibid.*, 271—274, 276).—Polemical. Schenck replies to criticisms of his work (A., 1927, 939, 1030) by Maurer and Bischof (Stahl u. Eisen, 1928, 48, 15) and gives fuller experimental details. The latter authors maintain and amplify their previous criticisms. A. R. POWELL.

Duralumin change in iron-carbon alloys. G. MASING and L. KOCH (Wiss. Veröff. Siemens-Konz., 1927, 6, 202—210).—A change similar to that which occurs with duralumin and other aluminium alloys after rapid cooling from about 500° and subsequent keeping at ordinary temperature or heating again to about 150°, occurs also with iron-carbon alloys. The change which occurs is distinguished from the result of ordinary quenching, and is a modification with time of the properties of the alloy, particularly of the hardness. The observations were made on various irons which were first heated to 660—700°, quenched, and then kept for several weeks, the hardness being measured at intervals; it was found that an increase, amounting to about 80% of the original hardness, took place during two to three weeks. S. I. LEVY.

Theory of steel hardening. K. HONDA (Arch. Eisenhüttenw., 1927—8, 1, 527—536; Stahl u. Eisen, 1928, 48, 263—263).—The changes taking place when steel is cooled from above the A1 point to 20° may be summarised as follows: austenite (tetragonal lattice $c/a =$

1.414)→ α -martensite (tetragonal lattice $c/a = 1.04$) → β -martensite (cubic body-centred lattice) → pearlite (precipitation of carbon). With very slow cooling these changes proceed to completion; with slightly more rapid cooling part of the martensite remains undecomposed and the steel is partly hardened; with a still greater rate of cooling the change to pearlite is suppressed, the structure consists of ($\alpha + \beta$)-martensite, and the steel is completely hardened; and, finally, with a greater rate of cooling, austenite remains undecomposed on the outside of the specimen and the steel is then overhardened. Annealing of hardened steel at 150° causes the α -martensite to be converted into the β -modification with an increase in the intensity of magnetisation, a decrease in the electrical resistance, and a decrease in volume; at 250° β -martensite is converted into pearlite with evolution of heat but without change of volume. At a somewhat higher temperature a considerable contraction in volume takes place accompanied by a change in the magnetic properties and by absorption of heat. As cementite is the only endothermic phase that can exist at this temperature, these facts can be explained only on the assumption that the decomposition of the solid solution (β -martensite) takes place in two stages, in the first of which carbon atoms are displaced from the lattice and in the second of which these atoms combine with iron to form the carbide, cementite, with absorption of heat and a decrease in volume. The greater hardness of martensite compared with ferrite, although both have a similar lattice structure, is ascribed to the finer grain of martensite, the presence of internal stress due to a state of metastable equilibrium, and the presence of carbon atoms in the lattice in such a way that the lines of force emanating from them affect six neighbouring iron atoms. Assuming that ferrite has a Brinell hardness of 225 the increase due to the above-named factors would be 80, 150, and 225 respectively, giving the martensitic hardened steel a Brinell hardness of 680.

A. R. POWELL.

Quenching of steels in a high-temperature bath.

K. HONDA and K. TAMARU (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 69—83).—Quenching of steels in low-temperature media, *e.g.*, water and oil, is often accompanied by cracking, visible or microscopic, in either case harmful, and according to the theory of quenching advanced by Honda (B., 1919, 821 A) there is no reason why troostitic and sorbitic structures should not be obtained by a mild quenching in a hot bath. Austenite is transformed into pearlite through martensite irrespective of the cooling velocity, and when this change occurs at a high temperature the martensite is converted again into troostite or sorbite. Since the change austenite → martensite → troostite involves an expansion and a contraction, the two phases altering side by side causes no abrupt change in volume. A mixture of 65% of potassium nitrate and 35% of lithium nitrate (m.p. 132°) was used as a quenching medium above 150°, for lower temperatures mercury was utilised, when quenching a steel containing 0.89% C. Maximum hardness was obtained at about 110°, and impact-resistance increased gradually up to 320°, decreasing at higher temperatures. Specimens quenched in the salt bath below 530° were weaker than under normal treatment,

but had a higher tensile strength when quenched at above 530°. Quenching cracks occurred always at temperatures below 120°, but any desired structure could be obtained without any risk of cracks if the bath was above 150°, martensitic up to 200°, troostitic 300—400°, and sorbitic above 400°.

C. A. KING.

Diffusion phenomena of zinc and tin in steel and soft iron.

H. HONIGL (Mikrochem., 1928, 6, 22—27).—Tin begins to diffuse into iron and steel at 300° with the formation of a thin layer of solid solution which at this temperature appears to hinder further diffusion. At 500—750°, however, diffusion of tin into steel proceeds rapidly with the formation of several layers of intermetallic compounds interspersed with crystals of solid solutions; at the same time the carbon content of the steel diffuses towards the central portions, leaving a layer of decarburised steel inside the layers of tin-iron compounds. At 900° there is a pronounced tendency for the carbon to diffuse back towards the edges of the test-piece. The rate of diffusion of zinc into iron increases rapidly with the temperature above 500° with the formation of three distinct layers of differently oriented mixed crystals, accompanied by an enrichment of the carbon content of the inside portions of the test-piece.

A. R. POWELL.

Effect of a short period of annealing on the tensile strength and electrical conductivity of copper, bronze, aluminium, aludur, and aldrej.

H. BÖHNER (Z. Metallk., 1928, 20, 132—141).—Experiments on hard-drawn wires (99% reduction) of copper (99.95% Cu), bronze (containing small quantities of magnesium and tin in solid solution), aluminium (containing 0.17% Fe and 0.14% Si), aludur (aluminium containing 0.27% Fe, 1.07% Si, and 0.43% Mg), and aldrej (aluminium containing 0.33% Fe, 0.49% Si, and 0.51% Mg) are described in which the electrical conductivity and tensile strength after passing a heating current through them for varying short periods of time were determined. For metals and alloys consisting wholly of solid solution at the ordinary temperature complete recrystallisation, accompanied by minimum tensile strength and maximum electrical conductivity, is obtained in a fraction of a second at a sufficiently high temperature, *e.g.*, 700° for copper, 550° for the above bronze. This also applies to alloys which require to be aged at a high temperature to obtain the maximum tensile strength, *e.g.*, aldrej. On the other hand, alloys of which the strength and conductivity depend on the form in which the constituents are present require a relatively long period of annealing when the annealing temperature necessary to produce minimum strength and maximum conductivity is low; this is due to the slow rate of diffusion of the constituents.

A. R. POWELL.

Influence of dissolved gases on the soundness of 70:30 brass ingots.

G. L. BAILEY (Inst. Metals, March, 1928. Advance copy, 12 pp.).—Ingots cast from brass treated in the molten state with nitrogen, hydrogen, and sulphur dioxide were cast in a series of moulds giving a wide range of cooling velocities. In no case was evidence obtained of unsoundness caused by gas evolution, though slow speeds of cooling were associated with extensive shrinkage cavities. It is considered that the high vapour pressure of zinc in molten brass

precludes the dissolution of gases, and that the spherical cavities occurring in brass ingots are the result of gases trapped mechanically within the mould. The conclusion is opposed to that of Bamford and Ballard (B., 1921, 84 A) as brass can be cast safely by special methods from only 30° above the liquidus. C. A. KING.

Quenching and tempering of brass, bronze, and "aluminium bronze." T. MATSUDA (Inst. Metals, March, 1928. Advance copy. 42 pp.).—When quenched from high temperatures the hardness of ($\alpha + \beta$)-brass increased while the electrical resistance decreased, but no abnormal change corresponding to the β -transformation was observed. No appreciable change in the properties of a bronze containing 10–12% Al occurred if quenched below 600°, but the hardness and electrical resistance increased steadily due to redissolution of α in β . A bronze containing 18–20% Sn behaved similarly to the aluminium bronze, showing, however, another decrease in hardness at the eutectoid transformation at 590°; in all cases the rate of cooling had a pronounced effect. The temper-hardening is due probably to the straining of space-lattice produced by the separation of α from β or γ , or the decomposition of β or γ into eutectoid. C. A. KING.

Determination of antimony in bronze, brass, and similar alloys. S. A. TSCHERNICHOF (Z. anal. Chem., 1928, 73, 265–270).—The alloy (5 g.) is dissolved in nitric acid and the precipitate of metastannic and antimonious acids is collected, ignited, and dissolved by heating with concentrated sulphuric acid and a few crystals of sugar. When the acid becomes colourless it is cooled and diluted with water, hydrochloric acid is added, and the antimony titrated as usual with bromate. If the alloy contains no tin the addition of a small quantity of this metal to the flask in which the dissolution is effected is necessary in order to ensure that the antimony is completely precipitated.

A. R. POWELL.

Thermal and electrical conductivity of some aluminium alloys and bronzes. E. GRIFFITHS and F. H. SCHOFIELD (Inst. Metals, March, 1928. Advance copy. 36 pp.).—A series of seven copper alloys containing 0.5–12.4% Sn and 2% Zn showed thermal conductivities at room temperature varying from one fifth to one tenth of that of copper, increasing considerably with rise of temperature in contrast to the conductivities of pure metals. An admixture of 10% of aluminium was comparable in its effect with that due to 10% of tin, and minute traces of phosphorus gave rise to appreciable reduction in conductivity. All the alloys followed Lorenz's law of the relation between thermal and electrical conductivity and absolute temperature. In the series of light aluminium alloys, the two containing 4.5% and 8% Cu, respectively, possessed a thermal conductivity equal to 82% of that of aluminium. Alloys containing copper, nickel, iron, and manganese in increasing quantities gave decreasing conductivity values, the lowest value of the series being given by an alloy containing 13% Zn and 3% Cu. The Lorenz coefficients of aluminium alloys were consistent but of lower value than those for copper alloys. C. A. KING.

Recrystallisation of the silver[–copper] alloy containing 80% Ag. R. GLOCKER and H. WIDMANN

(Z. Metallk., 1928, 20, 129–131).—The tensile strength of the 20:80 copper–silver alloy after cold-rolling to 98% reduction is about 90 kg./mm.² compared with 40 and 50 kg./mm.² for similarly treated silver and copper respectively. The tensile strength of the worked alloy decreases slowly with rise in annealing temperature to 85 kg./mm.² at 200°, then more rapidly and almost linearly to 35 kg./mm.² at 600°, and, finally, slowly again to 30 kg./mm.² at 800°. The tensile strength of pure silver, on the other hand, decreases suddenly at 200° to a constant value of 30 kg./mm.² Annealing the alloy at 800° produces new grains which in 30 min. are 2–5 times as great as those produced in 13 hrs. at 780°. Microscopical examination with a high-power objective fails to disclose any sign of recrystallisation below 500°, and X-ray examination by the Debye–Scherrer method shows that the characteristic rolled structure persists up to 780°, and that recrystallisation is complete only after 1 hr. at 800°. The broadening of the outer rings in the X-ray photographs, however, begins to disappear on annealing at 300°, and at 400° these rings become quite sharp, showing that distortion of the lattice caused by the cold-work has been relieved. Hence this method affords a delicate test for determining the temperature at which the preliminary rearrangement of the atoms prior to recrystallisation takes place. A. R. POWELL.

Dilatometric study of light metals. M. HAAS (Inst. Metals, March, 1928. Advance copy. 16 pp.). Technical improvements in the working of the Oberhoffer–Esser dilatometer are described. The chief heating tube of the furnace consists of nickel with a network of chrome–nickel heating strip. Copper heads at the end of the tube serve as heat reservoirs, and the whole is surrounded by a second nickel tube wound for uniform heating. The use of a test bar having rounded ends and also of delicate pressure contact springs is recommended to minimise the increasing plasticity of a test-piece at higher temperatures. Curves of several light alloys are reproduced. C. A. KING.

Tin bronzes. M. HANSEN (Z. anorg. Chem., 1928, 170, 18–24).—Bands round the edges of the crystal grains may appear after etching in alloys of copper with 15–25% of tin when quenched even from below 590°. The formation of the bands is, therefore, not connected with the transition point at 590°, and is not a consequence of a polymorphic transformation of the β -crystals, as Stockdale has suggested (B., 1925, 809). The bands are the wider the slower is the quenching, and the nearer the temperature from which the alloy is quenched approaches to 520°. It thus appears likely that they consist of a crystal species stable at lower temperatures, and formed in spite of the quenching. This could only be the δ -crystal, formed at 520°, but this cannot be the only constituent of the bands, since crystals and bands may be present together. It seems, therefore, highly probable that it is the ($\alpha + \delta$)-eutectoid in a very highly dispersed state which is involved, and it has in fact been possible to reproduce various intermediate stages between the bands, apparently consisting of only one phase, and the visibly two-phase eutectoid. R. CUTHILL.

Volume change in certain type metals during solidification. Y. MATUYAMA (Sci. Rep. Tôhoku Imp.

Univ., 1928, 17, 1—25).—The change in volume of certain metals and alloys used in casting type has been measured by means of a thermo-balance. Pure antimony showed a volume expansion during solidification of 0.95% (approximately one third that of bismuth), but a contraction was observed in every trial with standard type alloys. As this is contrary to general belief it is pointed out that the surface tensions of antimony and lead, which are the chief components of type metals, are smaller than those of many other pure metals, and so a good fluidity of the melt is produced. Also it is usual to apply pressure on the molten metal in the type mould which aids in securing "sharp" type. C. A. KING.

Reversed segregation [in mixed-crystal alloys].

II. G. MASING and C. HAASE (Wiss. Veröff. Siemens-Konz., 1927, 6, 211—221; cf. Bauer and Arndt, B., 1922, 220 A).—The various explanations of the fact that the first-cooled portions of a casting of an alloy of mixed crystal structure are found to be richer than the remainder in the most fusible component are considered. S. I. LEVY.

[Standard specifications for] metals. AMER. SOC. TESTING MATERIALS (Part I, Metals, 1927, 871 pp.).—Standard specifications, methods of testing, definitions, and rules are given, together with certain recommended practices. CHEMICAL ABSTRACTS.

PATENTS.

Carrying-out of the Bessemer process [for steel].

A. E. WHITE. From S. G. ALLEN (B.P. 286,745, 30.8.26).—The "net blow-heat," *i.e.*, the gross heat developed by oxidation in the molten charge by the oxygen in the blast less the quantity of heat absorbed in raising the temperature of the waste gases, of any particular charge is determined and the proportion of oxygen in the blast is regulated to minimise loss of heat in the waste gases. [Stat. ref. to B.P. 20,468 of 1904 and 13,952 of 1900.] C. A. KING.

Furnace for treating iron with a low proportion of carbon. F. WÜST (B.P. 275,646, 5.8.27. Ger., 6.8.26).—Scrap iron or steel is melted in a cupola furnace connected to a continuously working reverberatory furnace. A substantial proportion of the heat required for melting is supplied from the reverberatory furnace and the coke charge to the cupola is regulated within the carbon limit of the iron, which is superheated in the second furnace to ensure the formation of eutectic graphite. C. A. KING.

Apparatus for purifying and de-aerating molten iron. J. DECHESNE (B.P. 271,100, 13.5.27. Ger., 14.5.26. Addn. to B.P. 265,827; B., 1927, 302).—During the pouring of molten iron from a furnace into the ladle the latter is mechanically agitated; *e.g.*, the ladle may be tiltably journalled and jiggled by means of cams underneath. C. A. KING.

Production of synthetic pig iron. A./S. NORSK STAAL (ELEKTRISK-GAS-REDUKTION), and E. EDWIN (B.P. 286,067, 12.2.27).—Spongy iron, produced by reducing iron ore and separating the gangue before melting, is melted with substantially pure carbonaceous material, together with additions of silicon and man-

ganese, in an electric furnace at 1100—1300° under acid conditions. C. A. KING.

Manufacture of commercially pure iron alloys.

L. F. REINARTZ and J. H. NEAD, Assrs. to AMER. ROLLING MILL Co. (U.S.P. 1,665,146, 3.4.28. Appl., 4.10.23).—A bath of molten iron low in carbon and metalloids is treated in an electric furnace with a degasifying agent, then with a reducing slag, and finally with an alloying element substantially free from carbon. A. R. POWELL.

Protection of ferrous metals against corrosion.

G. B. ELLIS. From T. E. MURRAY (B.P. 287,194, 8.11.26).—Iron and steel are electroplated with nickel on which a layer of chromium is subsequently applied. The product is then heated at 980—1380° in an oxidising atmosphere. Partial alloying occurs and the outer surface is covered with a thin tenacious film of oxide. C. A. KING.

Galvanising or coating by dipping of steel.

E. A. ATKINS, and RYLANDS BROS., LTD. (B.P. 287,201, 19.11.26).—In a galvanising process, steel articles are immersed in lead or a lead alloy maintained at 600—800° and are then immersed in molten zinc without coming into contact with the atmosphere. In practice, a layer of molten zinc may float on the surface of the lead bath. C. A. KING.

Manufacture of chromium or similar refractory metals.

GEN. ELECTRIC Co., LTD., and C. J. SMITHELLS (B.P. 285,571, 18.11.26).—Coherent articles of chromium are formed by agglomerating the pure powdered metal into shapes and sintering it at 1500—1550° in hydrogen freed from oxidising impurities. Hydrogen for this purpose may be prepared by passing the gas through a vessel containing incandescent tungsten and then through a tube cooled to the temperature of liquid oxygen. C. A. KING.

Mechanical ore-roasting furnace.

J. HARRIS (B.P. 287,356, 2.6.27).—An upper hearth of a multiple-hearth furnace is provided at its discharge end with a conduit depending from the hearth above the rabble arm of the hearth below. Ore falling into the conduit may be retained there by a sealing-plate carried by the rabble shaft until, by the rotary motion, registration is effected with a second conduit which allows the ore to be guided to the lower hearth out of contact with the furnace draught and so without the production and loss of dust. C. A. KING.

Melting of metals.

F. W. CORSALLI (B.P. 267,972, 19.3.27. Ger., 19.3.26).—Refining metals for addition to the charge of a cupola or shaft furnace are melted separately in a furnace adjoining, and are introduced directly into the melting zone of the cupola. C. A. KING.

Ore- or like mixing machines.

H. ERIKSSON (B.P. 287,016, 2.11.27).—A machine for mixing ore concentrate with coal or coke breeze consists of a vertical, open-ended, rotary drum supported above a stationary bottom plate having an adjustable aperture for the discharge of materials. The drum is provided with a series of cross-bars which are spaced in relation to beaters on a central shaft. C. A. KING.

Sifting or screening apparatus for the classification of ores etc. A. FRANCE (B.P. 276,378, 17.1.27. Belg., 18.1.26).—A screening channel having an increasing inclination towards the discharge end is so mounted that a rotary movement is imparted to the receiving end, *e.g.*, by mounting it on a crank of a driving shaft, and a vertically-oscillating movement is given to the discharge end. C. A. KING.

Flotation processes. D., M., S. R., and S. GUGGENHEIM, J. K. MACGOWAN, and E. A. C. SMITH (GUGGENHEIM BROS.), Assees. of A. H. FISCHER (B.P. 284,198, 29.4.27. U.S., 24.1.27).—Flotation agents comprising the products obtained by the interaction of a metal xanthate or metal alkylxanthate with an acid chloride, such as carbonyl, acetyl, or sulphuryl chloride, or with an alkyl chloroformate are claimed. This class of flotation agents is particularly suitable for working in acid media. A. R. POWELL.

Extraction of metallic values [copper, lead, and zinc] from ores. F. DIETZSCH (B.P. 287,207, 8.12.26 and 16.2.27).—Oxidised or roasted copper ores are leached with a cold solution of sodium or calcium thio-sulphate saturated with sulphur dioxide, whereby the cuprous sulphite first formed is dissolved by the thio-sulphate solution. Addition of lime to the leach liquors precipitates copper hydroxide with the regeneration of the thio-sulphate solution. For the treatment of ores containing lead sulphate the use of sulphur dioxide is unnecessary, otherwise the same process is applicable to oxidised lead ores and zinc ores. Oxidised lead-zinc ores are first leached with sulphur dioxide solution alone to dissolve the zinc and the residue is then leached with thio-sulphate solution. A. R. POWELL.

Reduction of ores. H. WITTEK (B.P. 275,144, 20.11.26. Ger., 27.7.26. Addn. to B.P. 274,803; B., 1928, 269).—Ores are reduced in combination with the nitrogenation of carbides of metals other than calcium, the carbides of which are capable of nitrogenation. C. A. KING.

Manufacture of tinned plates. H. WILLIAMS (B.P. 287,288, 9.2.27).—Plates proceeding from a tinning bath are passed through water at 90–100° so that the plates leave the water-bath hot and any water adhering to the plates is evaporated rapidly. C. A. KING.

Application of thin, reflecting, metal coatings to the surface of flexible or elastic materials. LAMPEN- U. METALLWAREN-FABR. R. DITMAR GEBR. BRÜNNER A.-G., Assees. of A. DOMAN (B.P. 262,827, 14.12.26. Hung., 14.12.25).—The material to be coated is impregnated with a binding material, *e.g.*, resin or gum, laid on the thin reflecting film of metal, and gently heated to cause the two surfaces to adhere. Alternatively, a layer of an emulsion or binding material is applied to the back of the metal film and the fabric is pressed on to this layer before it dries completely. The metal film is deposited on to a reflecting surface by a galvanoplastic process and peeled therefrom after fixing the elastic material to its upper side. A. R. POWELL.

Joining together of metal surfaces. W. SMITH (B.P. 285,995, 25.1.26 and 1.9.27).—Articles of similar or dissimilar metals are united by pouring molten metal to come into contact with the amalgamated surfaces of the

articles; *e.g.*, articles of aluminium are joined by placing a strip of amalgamated zinc against each surface to be joined and pouring molten aluminium between the strips. C. A. KING.

Recovery of light metals from scrap. I. G. FARBENIND A.-G., and A. BECK (B.P. 287,360, 13.6.27. Addn. to B.P. 182,948; B., 1922, 715 A).—Anhydrous calcium chloride containing additions of calcium fluoride may be used as a flux, the eutectic composition of the melt (82% CaCl₂, 18% CaF₂) being avoided. C. A. KING.

Alloys for bearings. W. MATHESIUS and M. NEUFELD (F.P. 622,237, 28.9.26. Ger., 1.10.25).—Further to B.P. 259,211, alloys containing 0.2–0.25% Mg together with 0.1% Bi, Cu, Sn, or of mixtures of two or all of these metals are claimed. C. HOLLINS.

Non-ferrous casting [alloy]. R. L. BINNEY, Assr. to BINNEY CASTINGS Co. (U.S.P. 1,665,435, 10.4.28. Appl., 6.7.25).—A refractory alloy containing, approximately, 1 pt. of manganese, 2 pts. of zinc, 18 pts. of copper, and $\frac{1}{2}$ pt. of aluminium is claimed. F. G. CROSSE.

High-resistance alloys. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (B.P. 285,565, 17.11.26).—The alloys, resistant also to oxidation, contain 70–80% (75%) Ni, 15–25% (20%) Cr, 2–7% (5%) Co. Iron may be present up to 10%, about half of the iron replacing chromium in the alloy. C. A. KING.

[Non-magnetic] alloys. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (B.P. 287,236, 17.12.26).—The alloy of high electrical resistance contains 23–27 (23.7%) Ni, 71–67 (70.8%) Fe, 1.5–4 (3%) Cr, and 1.5–4 (2.5%) Al or Si. C. A. KING.

Enamelled wires. N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 274,079, 4.7.27. Holl., 12.7.26).—Wires, resistant to oxidation and suitable for wireless transformers, have a core of a highly refractory metal, *e.g.*, molybdenum or alloy. C. A. KING.

Thermostatic material. P. H. BRACE, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,660,911, 28.2.28. Appl., 12.3.25).—A thermostatic strip comprises elements of molybdenum and of an alloy of electrolytic iron and chromium. T. S. WHEELER.

Briquetting [of iron ores etc.]. A. CARLSON (U.S.P. 1,666,618, 17.4.28. Appl., 8.4.25. Swed., 10.5.24).—See B.P. 233,731; B., 1925, 962.

Production of rustproofing material [for iron]. H. H. WILLARD and M. GREEN, Assrs. to PARKER RUST-PROOF Co. (U.S.P. 1,660,661, 28.2.28. Appl., 10.5.26).—See B.P. 273,168; B., 1927, 658.

Volatilisation and oxidation of minerals. B. G. COBB, Assr. to COBB ELECTRO REDUCTION CORP. OF CANADA, LTD. (U.S.P. 1,660,150, 21.2.28. Appl., 6.4.21. Renewed 1.3.27).—See B.P. 223,271; B., 1925, 13.

Apparatus for the manufacture of tin plates. G. B. ELLIS. From AMER. SHEET & TIN PLATE Co. (B.P. 287,436–7, 13.9.26).

Crucibles (B.P. 278,367).—See VIII. Emulsions for tin-plating machines (B.P. 287,438).—See XII.

XI.—ELECTROTECHNICS.

Setting time of cement. SHIMIZU.—See IX. **Concentration of ores.** LEE and others. **Conductivity of metals and alloys.** BOHNER, also GRIFFITHS and SCHOFIELD.—See X.

PATENTS.

Electric furnace. J. L. McFARLAND, ASST. to GEN. ELECTRIC Co. (U.S.P. 1,661,842, 6.3.28. Appl., 16.6.27).—Insulating supports, from which a ribbon-shaped heating resistor is hung in the form of a series of depending hoops, are secured to the refractory walls of the heating chamber. Headed spacing members are secured to the furnace wall in alternate spaces between the lower ends of the loops, the heads overhanging the lengths of the adjacent resistor. Parallel-sided, wedge-shaped spacing members for the loops project from the wall in the remaining spaces between the lower ends of the loops, the edges of which members extend horizontally.

J. S. G. THOMAS.

Electric [induction] furnace. C. B. FOLEY, ASST. to C. B. FOLEY, INC. (U.S.P. 1,661,173, 6.3.28. Appl., 23.3.22. Renewed 27.7.26).—A tube passing through a crucible at a substantial distance below the axis of the periphery of the crucible, co-operates with the sides and bottom of the crucible to form a closed channel opening into the crucible on opposite sides.

J. S. G. THOMAS.

Electrolytic rectifier. C. E. OGDEN, ASST. to KODEL RADIO CORP. (U.S.P. 1,661,703, 6.3.28. Appl., 6.8.26).—An electrode containing silicon is used in conjunction with an acid electrolyte containing sulphuric acid (d 1.3—1.7).

J. S. G. THOMAS.

Electric incandescence lamp and manufacture of its illuminating body. F. SKAUPY, ASST. to GEN. ELECTRIC Co. (U.S.P. 1,659,749, 21.2.28. Appl., 2.12.21. Ger., 4.12.20).—Light radiators of the type described in B.P. 187,613 (B., 1923, 542 A) are used and heated by passage of current through a metallic conductor.

T. S. WHEELER.

Helium space-discharge tube. F. S. McCULLOUGH (U.S.P. 1,661,436, 6.3.28. Appl., 3.4.24).—A cathode, on which there is a substance from which helium is liberated when heated, and an anode are assembled in a tube and degassed by baking. After the tube is sealed a current is passed through the cathode whereby helium is evolved in the tube.

J. S. G. THOMAS.

Production of galvanic coatings of varying thicknesses. A. L. MOND. FROM BERNDORFER METALLWARENFABR. A. KRUPP A.-G. (B.P. 287,402, 14.9.27).—A jet of electrolyte from which metal is to be deposited electrolytically is directed on to the non-submerged surface of the article to be coated, and the thickness of metal deposited at any part is controlled by adjustment of the strength, speed, and size of the jet of electrolyte and the duration of its impingement upon the part.

J. S. G. THOMAS.

Brush for generators etc. A. A. SCHUPP, ASST. to FREDERICKSEN Co. (U.S.P. 1,664,708, 3.4.28. Appl., 2.4.24).—An alloy of lead and copper cast in equal proportions is claimed.

H. ROYAL-DAWSON.

Production of galvanic metal coatings. F.

KIRSCHNER (U.S.P. 1,664,750, 3.4.28. Appl., 12.10.23. Austr., 10.6.23).—See B.P. 217,876; B., 1925, 176.

Non-magnetic alloys (B.P. 287,236). **Enamelled wires** (B.P. 274,079). **High-resistance alloys** (B.P. 285,565).—See X. **Purification of water** (B.P. 287,196).—See XXIII.

XII.—FATS; OILS; WAXES.

Relation between constants of butter fat. O. LAXA (Ann. Falsif., 1928, 21, 97—98).—The Reichert-Meissl (*RM*), Wauters-Polenske (*WP*), and iodine values (*i*) of butter fat added together and divided by the refractive index (*n*) gave, for Czecho-Slovakian butter fats, a nearly constant value. Nine February samples gave an average value of 1.36 (1.29—1.43); 18 March samples, 1.4 (1.36—1.51); 19 April samples 1.45 (1.33—1.56); 15 May samples 1.4 (1.3—1.5); and 15 June samples 1.46 (1.34—1.6). The average mean for 82 samples was 1.4, and the equation $RM + WP + i/n = 1.4$ can be used for finding any one of the values.

D. G. HEWER.

Determination of the degree of rancidity in butter. G. S. INKHOV and A. F. SHOSHIN (Trans. Vologda Dairy Inst., 1926, Bull. 64, 161—174).—The butter (1 g.) is mixed with light petroleum (10 c.c.) and Schiff's reagent (1 c.c.); after 10 min. the intensity of the colour is compared with that of standards prepared from formaldehyde.

CHEMICAL ABSTRACTS.

Butter-fat of colostrum. K. A. KUROCHKIN (Jaroslav Zootech. Exp. Stat. (1926), 1927, 142—144).—Colostrum fat obtained (by mixing 200 c.c. of top milk with 600—800 c.c. of hydrochloric acid, d 1.12, keeping at 90—95° for 1.5 hrs., cooling, and washing the fat with water not above 40°) 3.5 and 48 hrs., respectively, after delivery has m.p. 38.6, 28—29°, Reichert-Meissl value 19.65, 28.4, saponif. value 213.3, 226.5. The properties of colostrum fat approach those of lard; during the first 24 hrs. the colour is deep orange.

CHEMICAL ABSTRACTS.

Determination of the iodine value [of fats] by means of thiocyanate. H. STADLINGER (Pharm. Ztg., 1928, 73, 340—342; cf. Kaufmann, B., 1926, 165, 447).—The employment of solutions containing free thiocyanogen for the determination of unsaturation values of fats is described and discussed. The determination is carried out by addition of excess of the reagent solution to a weighed quantity of the fat, keeping it in the dark for 24 hrs., and determining the excess of reagent by addition of potassium iodide and titration of the liberated iodine. The results are expressed in terms of iodine, and are thus comparable with the ordinary iodine values. Since thiocyanogen adds directly to one double linking, does not react with a triple linking, and adds only to one double linking when two or more are present in the molecule, comparison of the ordinary iodine value with the "thiocyanate iodine value" gives a method of determining the nature and quantities of the various unsaturated compounds present. Applications of the method are discussed.

S. I. LEVY.

Fluorescence under the analytical quartz lamp of lard containing paraffin. E. FEDAR and L. RATH

(Z. Unters. Lebensm., 1927, 54, 321—330).—The behaviour of lard in the ultra-violet light of the quartz lamp is a valuable indication of its purity. Pure lard obtained by rendering shows no special fluorescence, but the commercial product exhibits white, green, or blue luminescence. This phenomenon may be produced in pure lard by treatment with steam, alkali carbonate, alkaline-earth hydroxides, animal charcoal, or fuller's earth. Some commercial lards showed a peculiar and intense blue-violet iridescence on the surface, and this was found to be associated with a relatively high unsaponifiable fraction. The active substance was isolated by heating the unsaponifiable residue with concentrated sulphuric acid at 105° for 1 hr. and extracting the undestroyed substance with light petroleum. The paraffin obtained on evaporation of the solvent showed a strong blue luminescence in ultra-violet light, and a trace added to a non-fluorescing lard imparted to it the fluorescence characteristic of the above-mentioned samples. The origin of the paraffin in the lard could not be regarded as accidental, and was undoubtedly due to adulteration with "white grease," which had been denatured with petroleum distillate. W. J. BOYD.

Utilisation of acid sludge from oil distillates treated with fuming sulphuric acid. K. LIKHUSHIN (Azerbejdjanskoye Neft. Khoz., 1927, [8—9], 84—88).—A study of the use of mixed acid sludge from the treatment of "vaseline" oil with fuming sulphuric acid for the splitting of animal and vegetable fats.

CHEMICAL ABSTRACTS.

Limited splitting of natural drying oils and the so-called non-drying linseed oil. A. EIBNER and R. HELD (Chem. Umschau, 1928, 35, 65—77).—Earlier work demonstrated that the rise of acid value of films with time varies for the different drying oils. Thus, in 11 days the acid value of a sample of poppy-seed oil rose from 7.7 to 100, linseed oil from 3.04 to over 57, whilst China-wood oil changed more slowly and from 3.7 rose to 7—13 in the same period. After 127 days the poppy-seed oil had an acid value of 221 and the wood oil 62. Determination of this value affords a method of tracing the course of the reactions in film formation. Experiments are described on the artificial partial hydrolysis of fatty drying oils by the action of water under pressure. Linseed oil (25 g.) was heated with 1 g. of water in bomb tubes at 110°, 150°, 200°, and 250° for 70, 55, 36, and 24 hrs., respectively. The resulting oils had acid values 7.07, 64.3, 72.5, and 73.5, whilst the iodine values and mol. wt. had fallen but slightly, 176.5 to 147.5 and 701 to 524 for the oils undergoing the first and fourth treatments respectively. The natural and artificial processes are compared. From the crude products were isolated, purified, and determined: α - and β -linolenic, α - and β -linoleic, oleic, and some solid acids. No selective splitting of the solid or liquid acids was apparent. Linseed oil prepared in various ways from partially fermented seeds was similarly examined, but it was found that the fission products resulting in the drying process contained, under these conditions, as much as 14% of oxy-acids. The mechanism of the drying of fatty-oil films, and their uses in technology, are discussed on the basis of these results.

E. HOLMES.

Cottonseed. M. T. HARRINGTON (Texas Agric. Exp. Stat. Bull., 1928, [374], 19 pp.).—A comparative study of 73 varieties. The oil content is not related to the weight or size of the seed. The characteristics of the oil do not vary. The protein content of the oil- and water-free meats is highest in the seed having the highest oil content.

CHEMICAL ABSTRACTS.

Extractor for the preparation of oat and other cereal oils. L. A. MUNRO and D. S. BINNINGTON (Ind. Eng. Chem., 1928, 20, 425—427).—The extractor described is useful for extraction of large quantities of material of low oil content, and oat oil prepared in it was found to be entirely unoxidised, although of high acid value, probably owing to hydrolysis which could be eliminated by immediate extraction after cracking the oats. The apparatus consists of a 20-gal. drum, containing solvent and extracted oil, provided with a gauge and heated by 2-in. steam-pipes; an extraction chamber 2 × 3 × 3 ft. with false bottom, with a false top which has $\frac{1}{2}$ -in. perforations, flanged upwards, to ensure even distribution of the solvent; and a condenser fitted with $\frac{3}{8}$ -in. copper radiator pipes. The three chambers are placed one above the other, and by suitable manipulation of valves the solvent may be first prepared, the extraction made until a concentration of about 60% of oil is obtained, and, after clarification, the oil concentrated by vacuum distillation.

D. G. HEWER.

Fat emulsions for leather. SCHINDLER.—See XV.
Detection of cacao butter. COLOMBIER and CHAIZE.—See XIX.

PATENTS.

Production of commercial stearic acid. J. STARRELS (U.S.P. 1,659,790, 21.2.28. Appl., 15.10.23).—Crude fats containing free fatty acids are heated with ethyl alcohol and an esterification catalyst, and the ethyl esters obtained are washed with water, distilled *in vacuo*, hydrogenated, and hydrolysed.

T. S. WHEELER.

Manufacture of soaps. P. FRIESENAHN (B.P. 285,174, 19.11.26).—Ordinary soap is compounded with a mixture of higher alcohols, e.g., cyclohexanol, and aromatic or hydroaromatic sulphonic acids, e.g., naphthalenesulphonic acid or tetrahydronaphthalenesulphonic acid, or their sodium salts.

F. R. ENNOS.

Treatment of solid material for extraction of water, oil, and fatty matter therefrom. J. R. STERLING (B.P. 286,752, 8.11.26).—Animal or vegetable material to be extracted is subjected to lixiviation with a suitable solvent on the contra-flow principle in described apparatus.

S. S. WOOLF.

Apparatus for separating and recovering oil from an emulsion of oil and water. G. B. ELLIS. From AMER. SHEET & TIN PLATE CO. (B.P. 287,438, 13.9.26).—Alkaline emulsions of palm oil and water obtained from the tin-plating process are separated, and the oil is recovered by treatment in a specially-designed apparatus with a small quantity of sulphuric acid. The mixture is agitated and heated by means of jets of live steam, and, after separation, the oil is finally dehydrated by means of a centrifuge.

C. O. HARVEY.

Reaction products of cashew nut-shell oil. M. T. HARVEY (B.P. 259,959, 11.10.26. U.S., 16.10.25).—The iodine value of cashew nut-shell oil is reduced by acid oxidation or other method and the resulting gum, dissolved in suitable solvents, *e.g.*, amyl acetate, light petroleum, etc., is used in paints, varnishes, moulded compositions, waterproofing and insulating materials, etc. S. S. WOOLF.

Aralkylated fats and fatty acids (B.P. 286,796).—See III.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Influence of adsorption on the oil absorption of pigments. H. GROHN (Farben-Ztg., 1928, 33, 1660—1664).—The work of Gardner, Wagner, Klumpp, and others on oil absorption of pigments is summarised. A series of experiments on the absorption of oil, water, petroleum spirit with and without the addition of linseed oil fatty acids, stored and freshly-distilled turpentine, and 10% aqueous saponin solution by a range of pigments is described, and the results are considered in the light of Gurwitsch's views on adsorption. It is shown that the "vehicle-absorption" of a pigment depends on the nature of the vehicle, the nature and particle size of the pigment, and on their mutual adsorption capacities. S. S. WOOLF.

Testing of lithopone. ANON. (Farben-Ztg., 1928, 33, 1657).—Standard methods for the examination of lithopone are detailed under the headings:—moisture, total zinc, zinc oxide, sieving, oil absorption, fastness to light, colour, opacity, hiding power, water-soluble matter, behaviour in spirit varnish, and method of extraction from oil pastes. S. S. WOOLF.

Shellac. W. NAGEL and (Frl.) M. KÖRNCHEN (Wiss. Veröff. Siemens-Konz., 1927, 6, 235—275).—A monograph embodying the most recent contributions to knowledge of the composition and properties of shellac. The sources, commercial varieties, and industrial treatment of the material are dealt with in detail. S. I. LEVY.

Albertol resins. H. WOLFF [with W. TOELDTE and G. ZEIDLER] (Farben-Ztg., 1928, 33, 1724—1727).—Albertols compare favourably with kauri and Congo copals, and are decidedly superior to ester gum when each is made up into a standard oil-varnish. Properties studied were elasticity, tensile strength, drying, yellowing, and water-resistance. S. S. WOOLF.

Drying and non-drying oils. EIBNER and HELD.—See XII.

PATENTS.

Manufacture of lacquers, impregnating materials, etc. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 286,789, 10.12.26).—Cellulose esters or ethers are dissolved in strongly aqueous cyclic acetals derived from polyhydric alcohols, with or without other solvents and the usual additions. Examples are cellulose acetate (10 pts.), with water (30 pts.) and monoformaldehyde-glycerol (60 pts.), or with water (20 pts.) and formaldehyde-glycol (70 pts.). These lacquers may be applied with an unusually large proportion of added water. C. HOLLINS.

Manufacture of lacquers. K. SCHMIDINGER (U.S.P. 1,663,733, 27.3.28. Appl., 1.7.25. Ger., 13.5.25).—A benzol solution of resin is saponified with alcoholic potash, the resulting soap separated from the solution of unsaponifiable material, and the latter isolated by evaporating the solvent. F. G. CLARKE.

Nitrocellulose composition. Dibutyl mesotartrate. C. BOGIN, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,659,906—7, 21.2.28. Appl., [A] 14.8.25, [B] 26.1.27).—(A) *n*-Butyl mesotartrate is claimed as a plasticiser for nitrocellulose lacquers. (B) The compound has m.p. 49°, b.p. 205°/20 mm. T. S. WHEELER.

Production of a binding material for colours, varnishes, and cements. J. TENGLER (U.S.P. 1,660,851, 28.2.28. Appl., 14.5.27. Switz., 11.5.26).—Waste rubber is heated under pressure with tetralin to remove sulphur as hydrogen sulphide, the product is dissolved in benzene, and the solution cooled to separate naphthalene and treated with sulphur to revulcanise the rubber. T. S. WHEELER.

Thinning of varnish. A. L. BROWN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,660,914, 28.2.28. Appl., 4.10.24).—The hot varnish is treated with turpentine at 175°, and when cold, with a turpentine substitute which need not be free from sulphur. T. S. WHEELER.

Manufacture of coating compositions containing rubber and drying oils. [A] C. M. STINE, [B] C. M. A. STINE and C. COOLIDGE, [C] C. COOLIDGE and H. E. EASTLACK, Assrs. [A—C] to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,663,100—1 and 1,663,119, 20.3.28. Appl., [A] 19.12.23, [B] 5.9.24, [C] 3.7.25).—(A) A mixture of a solution of rubber (10 pts.) in a rubber solvent, with a drying oil (4—31 pts.), a drier (0.4—5% of the oil), sulphur (not exceeding 0.2 pt.), and an accelerator of vulcanisation is used. Thin films are heated at between 70° and 125° to evaporate the solvent, oxidise the oil, and vulcanise the rubber. (B) The solution of rubber (10 pts.) mixed with a drying oil (30—80 pts.) is thinned with a volatile solvent to a suitable consistency, and a cobalt drier added equivalent to 0.001—0.01% Co on the oil used; the viscosity of the composition on exposure to air remains satisfactory for more than 2 days. (C) The viscosity of a coating composition containing a rubber solution and a drying oil is regulated by the addition of 0.05—10% of oleic acid. D. F. TWISS.

Manufacture of condensation products of urea and formaldehyde. I. G. FARBENIND A. G. (B.P. 258,289, 13.9.26. Ger., 11.9.25).—Carbamide and formaldehyde are condensed in an aqueous solution the pH value of which is maintained at 4—7 by the use of suitable buffer compounds, *e.g.*, potassium oxalate, mono- or di-sodium phosphate, etc. Water is subsequently removed from the condensation mass by evaporation below 50°, preferably *in vacuo*, and the product is hardened at 50—100°. S. S. WOOLF.

Continuous preparation of phenolic resins. V. H. TURKINGTON, Assr. to BAKELITE CORP. (U.S.P. 1,660,403, 28.2.28. Appl., 20.2.24).—A mixture of phenol and aqueous formaldehyde solution is passed under pressure through a coil immersed in an oil-bath maintained by

heating or cooling at a suitable elevated temperature, and the product is discharged into a vacuum chamber in which the resin is dried. T. S. WHEELER.

Grinding or mixing of paints, inks, etc. J. BAXENDALE, and HUGHES & TRELEAVEN, LTD. (B.P. 287,658, 11.1.27).

Cashew nut-shell oil (B.P. 259,959).—See XII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Vulcanised "racking" of masticated rubber. Anisotropic vulcanisation. H. FEUCHTER (Kautschuk, 1928, 4, 48—51; cf. Wiegand and Braendle, B., 1923, 412 A).—Vulcanised rubber is easily distinguished from unvulcanised sheet by the "racking" phenomena possible with the latter. A sheet of rubber showing "calender grain," however, still exhibited this feature after vulcanisation with sulphur chloride vapour and warming to 100°; swelling with benzene occurs anisotropically, but, after drying, the anisotropic character persists in the vulcanised sheet. D. F. TWISS.

Measurement of resistance of vulcanised rubber to penetration of benzene and other combustible substances. F. C. SCHMELKES (Ind. Eng. Chem., 1928, 20, 430—431).—A piece of the hose to be tested is surrounded by the liquid in question and air is passed through at a standard rate. The issuing air is subjected to continuous analysis for hydrocarbon vapours by a calorimetric device described earlier (Lamb and Larson, B., 1920, 132 A), combined with a sensitive recorder. For calibration of the instrument, carbon monoxide is advantageous. D. F. TWISS.

Diffusion researches with rubber solutions. D. KRÜGER (Gummi-Ztg., 1928, 42, 1471—1474).—The diffusion coefficient for various grades of rubber was investigated by allowing a layer of solution to diffuse upwards into a superposed column of solvent for a number of days, then dividing the whole column into four layers, and determining the rubber content of each. The value of the diffusion coefficient for any one sample is greater the higher the concentration and the shorter the period, thus indicating either that rubber is a mixture of substances of different mol. wt. or that the rubber substance is present in various degrees of aggregation. Two components indeed may be present, one easily diffusible and the other sparingly so. Crêpe, smoked sheet, and Para rubbers give substantially equal diffusion coefficients. The diffusion coefficient is much smaller in hexane and ether than in benzene; calculation of the molecular magnitude by the formulæ of Einstein and Euler gives somewhat discordant results, but indicates a much lower mol. wt. in benzene. Mastication also reduces the molecular magnitude of rubber. D. F. TWISS.

Prevention of scorching during the mixing of rubber "compounds." W. ESCH (Kautschuk, 1928, 4, 51—55).—Although generally the tendency to scorching is greater the more powerful the accelerator of vulcanisation used, mixtures containing diphenylguanidine are more prone to scorching than mixtures with the more active di-*o*-tolylguanidine. Softening agents such as stearic acid or lead oleate reduce the tendency to scorching. Lead dioxide is particularly

effective in checking scorching, the quantity necessary (ranging from 1% downwards on the rubber) varying for different accelerators. D. F. TWISS.

Direct determination of rubber in soft vulcanised rubber. A. R. KEMP, W. S. BISHOP, and T. J. LACKNER (Ind. Eng. Chem., 1928, 20, 427—429; cf. Kemp, B., 1927, 372).—After successive extraction with acetone and chloroform, a sample (0.07—0.10 g.) is dissolved by refluxing with 50 c.c. of tetrachloroethane in an oil-bath at 160—170° for 3—5 hrs., a blank experiment being made in parallel. The cooled solution is diluted with 25 c.c. of purified carbon disulphide and 25 c.c. of 0.2*N*-Wijs solution are added. After 2 hrs. in diffused light, 25 c.c. of 15% potassium iodide solution are introduced and 50 c.c. of water, the liberated iodine being then titrated with 0.1*N*-thiosulphate. Sulphur combined with rubber is determined independently by dissolving 0.5 g. of the acetone- and chloroform-extracted rubber in tetrachloroethane, diluting to 250 c.c. with carbon tetrachloride, and allowing to settle; 100 c.c. of the clear liquid are evaporated to dryness and the sulphur is determined in the residue. The rubber content is calculated on the assumption that pure caoutchouc has an iodine value 372.8, and that 1 g. of combined sulphur corresponds with 2.13 g. of caoutchouc. The influence of carbon black, glue, cellulose, common inorganic compounding materials, and, in most cases, mineral rubber is negligible; the presence of factice in quantities exceeding 1% calls for a correction. The process is more accurate and more rapid than the customary indirect method. D. F. TWISS.

PATENTS.

Vulcanisation of rubber. GOODYEAR TIRE & RUBBER Co., Asses. of L. B. SEBRELL (B.P. 265,920, 16.11.26. U.S., 12.2.6).—Substitution or additive derivatives of 1-mercaptobenzthiazoles (excluding halogen derivatives) are claimed as vulcanisation accelerators. Examples are 5-nitro-1-mercaptobenzthiazole, its zinc and lead salts, the corresponding amine, and its benzaldehyde condensation products. C. HOLLINS.

Accelerators for the vulcanisation of rubber. E. C. R. MARKS. FROM RUBBER SERVICE LABORATORIES Co. (B.P. 286,749 and 287,001, 6.10.26).—(A) The accelerative effect of thiol accelerators, *e.g.*, mercaptobenzthiazole, is improved if these are combined chemically with an organic base, *e.g.*, hexamethylenetetramine, phenyldiguanide, triphenyldiguanide, or guanilylurea. The two components may be heated together. (B) The reaction product of a thiol accelerator and an organic base, such as a disubstituted guanidine, is combined with an aldehyde, *e.g.*, crotonaldehyde or heptaldehyde; the resulting resinous compounds are easily dispersed in rubber. D. F. TWISS.

Rubber vulcanisation accelerator. C. O. NORTH and W. SCOTT, Assrs. to RUBBER SERVICE LABORATORIES Co. (U.S.P. 1,664,481, 3.4.28. Appl., 17.12.24).—The compound produced by the interaction of hydrogen sulphide and the ammonia addition product of a straight-chain aldehyde is claimed. F. G. CLARKE.

Manufacture of coloured vulcanised rubber. E. R. BRIDGWATER, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,663,436, 20.3.28. Appl., 29.1.26).—Rubber

or rubber-like material is mixed with a vulcanising agent and with the sodium salt of the dye obtained by coupling diazotised 2-chloro-5-toluidine-4-sulphonic acid with β -naphthol.

D. F. TWISS.

Treatment of rubber emulsion to produce an adhesive. JOHNSON & JOHNSON (GT. BRITAIN), LTD. From JOHNSON & JOHNSON (B.P. 286,527, 15.6.27).—Rubber latex is diluted with a weak solution of a gum, e.g., gum tragacanth. The more concentrated upper layer which forms as a cream is further purified by shaking with an aqueous solution of an alkaline substance, e.g., sodium silicate, and of a germicide, e.g., formaldehyde, and again caused to separate by the addition of gum tragacanth. It is then mixed at 70° with one tenth of its volume of a solution containing 5% of agar and 1% of sodium silicate; the resulting emulsion, which is fluid while hot and gelatinous when cold, is squeezed in the latter state through fine gauze so as to impart a pasty consistency. The product is especially of use as an adhesive for attaching bandages and surgical dressings.

D. F. TWISS.

Manufacture of articles from ebonite and like material. A. FRASER, and RISSIK, FRASER, & Co., LTD. (B.P. 286,915, 2.11.26 and 26.5.27. Cf. B.P. 273,355; B., 1927, 789).—Ebonite dust is subjected to heat and pressure inside moulds, e.g., of iron or steel, the inner surfaces of which are plated with a metal, e.g., tin or chromium, or an alloy which resists adhesion or chemical reaction with the dust.

D. F. TWISS.

Manufacture of [rubber] stuffing. G. J. MILLER (U.S.P. 1,663,852, 27.3.28. Appl., 14.6.27).—Scrap rubber is softened by soaking it in kerosene until it is easily ground and, after grinding, is permanently softened to the required extent by treating it with kerosene at below 200°. The oil is separated from the mass, which is then dried.

F. G. CLARKE.

Manufacture of tubes of indiarubber or similar material. DUNLOP RUBBER Co., LTD., D. F. TWISS, and E. A. MURPHY (B.P. 287,592, 21.10.26).

Disulphide compounds (B.P. 277,338).—See III. **Binder for varnishes** (U.S.P. 1,660,851). **Coating compositions** (U.S.P. 1,663,100—1 and 1,663,119).—See XIII.

XV.—LEATHER; GLUE.

Avaram bark. I. K. S. CHOUDARY and E. YOGANANDAM (J. Soc. Leather Trades' Chem., 1928, 12, 53—58).—Avaram bark (*Cassia auriculata*), or "tarwad," is not very rich in tannin. It contains tans 17.6%, non-tans 10.8%, moisture 9.1%, and insoluble matter 62.5%. Samples from different districts vary slightly in tannin content. The tannin content increases with age from 15.58% to 19% in five years, but the ratio of tans to non-tans is fairly constant. The amount of tannin extracted at different temperatures increases with the temperature (optimum 95—100°). Of the total extractable tannin, 75% is extracted at ordinary temperatures (35—40°). The ratio of tans to non-tans does not vary much with the temperature of extraction. The colour of the leached liquors is deepest at low temperatures of extraction and becomes brighter at higher temperatures. The p_H value of both avaram and

konnar (*Cassia fistula*) liquors is 4.7. Avaram bark liquors do not penetrate so well as liquors prepared from other South Indian tanning materials. Avaram liquor kept for 125 days lost but little tannin, whereas the non-tans diminished by 6.8%; thus, the ratio tans/non-tans fell from 1 : 1.11 to 1 : 0.53. Avaram liquors lose least tannin of all the South Indian materials on keeping. Avaram-tanned leather is mellow and very light coloured, but darkens quickly on exposure to light. Most of the leather exported from Southern India is lightly tanned. An unadulterated sheep skin tanned with avaram had water-soluble matter 8.0%, fat 9.85%, moisture 12.5%, and leather substance 69.65%. The loss on washing was 5.5%. Spent avaram bark from the first, second, and third liquors, respectively, contained 5.87, 7.02, and 9.35% of tan.

D. WOODROFFE.

Microscopical examination of fat emulsions used in the leather trade. W. SCHINDLER (Collegium, 1928, 12—20).—Several emulsions of oils and fats at different p_H values, respectively, have been examined microscopically. Soap (10 pts.) and mineral oil (1 pt.) creamed at p_H 9—10 but remained partly emulsified at 8.6. A mixture of 20 pts. of Turkey-red oil and 1 pt. of mineral oil gave an opalescent emulsion which at p_H 7.2 showed no globules under the microscope, but at p_H 6.8 tiny globules became apparent; the emulsion was milky but stable. At p_H 6.1 there were much larger globules and the liquor creamed. A number of sulphonated fish oils have been similarly examined. A sulphonated neat's-foot oil was stable for 30 min. at p_H 7.9, but separated quickly below this value, and an addition of mineral oil did not improve the stability of its emulsions. Fresh egg-yolk emulsion was creamy and consisted of very small separate globules. At p_H 7.8 the emulsion with mineral oil was opalescent and creamed after being kept several hours; there were no groups of fat globules. The emulsion was more permanent at p_H 6.1. Soap-degrad emulsions creamed at p_H 9.5, 8.95, and 8.7. The results showed that the formation of groups of globules was indicative of emulsions which creamed. Increase of p_H value improves the colour and diminishes the number of visible globules, but does not always make the large drops smaller. These results confirm those of Stiasny (B., 1925, 965) and Riess. The emulsifying oils did not reduce the mineral oil to globules of definite size. Sulphonated oils had very little effect on the surface tension of the mineral oil to water, whilst they split up the mineral oils into very small globules and gave a fairly stable emulsion, which was less sensitive than the soap emulsions. The emulsifying power of sulphonated oils acting on mineral oil appears to depend on the ratio $(\alpha + \beta) : \gamma$. The amount of mineral oil affects the emulsifying power, and in commercial products is often considerable. Since α may be considered to consist of fatty acids and will disappear at p_H 6.1—7.0, it is evident that β is the important factor. The hydroxy-acids in β are less effective than the sulpho-fatty acids, so that the emulsifying power ultimately depends on the latter.

D. WOODROFFE.

Effect of salts and temperature on the manufacture of enzyme [bating] extracts and the results of enzyme analyses. J. SCHNEIDER, JUN., and A.

ULČEK (Collegium, 1928, 22—26; cf. B., 1927, 662, 758).—Enzyme extracts were prepared from "Oropon-standard" and different amounts of various salts by extracting for 30 min. at 18°. The filtered extract (50 c.c.) was tested on casein solution. Increasing amounts of ammonium sulphate, ammonium chloride, sodium chloride, ammonium and sodium chlorides, and ammonium sulphate and chloride caused increased activity of the enzyme extract. Curves have been obtained for the effect of these salts on the activity of the enzyme extract. Extractions at different temperatures showed maxima at 24° in two experiments, at 32° in a third, and a diminution of activity with rising temperature in two other tests.

D. WOODROFFE.

Effect of hydrogen-ion concentration on tannage.

P. PAVLOVITSCH (Collegium, 1928, 2—12).—A 1% solution of gelatin was mixed with 2—3 pts. of a 1% solution of quebracho, and the amount of the precipitate determined for different p_H values, respectively. Maximum combination took place at p_H 2; the amount of combination is practically uniform from p_H 3 to 6; at p_H 7.0 it was less and at p_H 8 very small. Further experiments with hide powder showed a steep fall in the combination curve at p_H 7.5—8.0, but no maximum as suggested by Thomas and again by Wilson (cf. Thomas and Kelly, B., 1923, 1235 A). Sole-leather pelts are penetrated most quickly at p_H 7—8. The penetration at 35—37° is 2—3 times as great as that at 15—20° using quebracho liquors of d 1.091. Oakwood liquors (d 1.091) penetrated best at p_H 6. Valonia liquors showed a more pronounced maximum fixation of tannin from acid liquors than oakwood. Mimosa, mangrove, and chestnut liquors (d 1.091) gave best penetration at p_H 8, 7, and 3—4, respectively. Tannage must be commenced with liquors of minimum astringency, the p_H values of which are as follows: (at d 1.091) quebracho 7—8, oakwood 6—7, mimosa 8, mangrove (d 1.60) 4, valonia extract ("Valex") 4, and chestnut 3—4. Pelts can be tanned in these strong liquors at these respective p_H values. It is not necessary, as Thomas states, to commence the tannage at the isoelectric point of collagen (p_H 5.0). Alkali should be added to strong tan liquors to increase the p_H value to 6—8, the pelts should be well penetrated by this, and then the p_H value should be lowered gradually to 3—4. The leather can afterwards be retanned in strong extracts. This process will not cause "dead tannage" or drawn grain. These various phenomena can be explained not only by the degree of dispersion of the tannin particles, but also by dehydration and rehydration. By combining the rehydrating medium at the commencement of tannage, the tannins can be forced to penetrate the pelt without tanning, and then, by dehydrating, the tannins can be coagulated and fixed on the hide substance. Colloids which have been dehydrated by tannin can be rehydrated by raising the temperature, adding organic substances such as non-electrolytes, e.g., alcohol, glycerin, glycol, or electrolytes such as acetic acid of moderate concentration, or by adding alkalis (cf. B., 1923, 789 A).

D. WOODROFFE.

American procedure in the colouring and finishing of leather. M. C. LAMB (J. Soc. Leather Trades'

Chem., 1928, 12, 58—63).—Most of the dyeing is done in drums, and mechanical devices are used wherever possible. The general lay-out of the works is such that there is no retrograde step. A much larger bulk of goods is handled in the different processes. American dyes are used and basic dyes preponderate. Pigment finishes or "dopes" are applied in the finishing of the leather by means of a machine fitted with a row of rotatable brushes. Spray-dyeing has not been applied to the same extent as in Great Britain. The spraying of pigment finishes is quite common. Every leather works in U.S.A. is equipped with drying machines. The skins are tautly strained by means of special clips on wire netting or perforated metal frames, which are placed in position in a drying chamber resembling a filing cabinet. Two men with a dozen sections of such a machine, capable of drying 2 doz. skins at a time, can be continuously occupied throughout the day. The actual drying requires a comparatively few minutes. The more rapid drying ensures greater fulness of shade than slow drying.

D. WOODROFFE.

Causes of drawn grain on chrome upper leathers.

J. S. MUDD (J. Soc. Leather Trades' Chem., 1928, 12, 49—52).—The waste liquors from chrome tannages which had caused drawn grain showed p_H values less than 3.7. Liquors buffered with sodium acetate to give p_H 4.3—4.9 gave a quicker tannage free from drawn grain. Other tests in which the p_H value decreased during the tannage produced drawn grain. It is suggested that acid swelling at p_H values 4.0 or above 5.0 will cause "pebbling" or drawn grain.

D. WOODROFFE.

Centrifuging as an aid to tannin analysis by the shake method.

V. KUBELKA (Collegium, 1928, 21—22).—To avoid the use of linen cloths, the operations in tannin analysis are performed in a centrifuge tube (200 c.c.) fitted with a rubber stopper. The hide powder can be shaken in the tube with water, chroming solution, wash water, or tannin infusion. Centrifuging at 4000 r.p.m. for 5 min. effectively separates the clear liquor.

D. WOODROFFE.

Cleaning leather. GOLDMAN and HUBBARD.—See V.

PATENTS.

Treatment of the skins of sharks and other fish of the same kind. A. EHRENREICH (B.P. 284,197, 24.3.27. Belg., 24.1.27).—The dermal armour is removed by treating the skins before or after tanning with a solution of sodium chloride and phosphoric or hydrofluoric acid, with or without an alkali salt of the acid, or aluminium salts, and/or small quantities of metallic salts which act as catalysts. An electric current may be passed through the solution, which is circulated by jets of compressed air.

D. WOODROFFE.

Derivatives from ligninsulphonic acid (B.P. 286,808).—See III. **Dyeing of fur** (U.S.P. 1,660,826).—See VI.

XVI.—AGRICULTURE.

Availability of potassium to plants as affected by barnyard manure. R. P. BARTHOLOMEW (J. Amer. Soc. Agron., 1928, 20, 55—81).—Barnyard manure may contain 3—20 (from the same herd 5—12) lb. of potassium per ton; all of it is available for plant growth

before much of the organic matter is decomposed. Since crops remove more potassium than is added as manure, the decomposition of the organic matter of the manure may convert some of the insoluble soil potassium into an available form.

CHEMICAL ABSTRACTS.

Ammoniacal nitrogen of peats and humus soils.

II. J. C. B. ELLIS and C. G. T. MORRISON (*J. Agric. Sci.*, 1928, **18**, 346—349).—The amount of ammoniacal nitrogen obtainable from bog peat by distillation under reduced pressure with magnesia increases with the extent of the previous drying of the sample. This is attributed to a reduction of the colloidal adsorptive power of peat during drying. Subsequent moistening of dried peat allows of a partial recovery of its adsorptive properties. (Cf. B., 1916, 1167.)

A. G. POLLARD.

Time of using nitrogenous fertilisers on winter crops. E. MÖLLER-ARNOLD and E. FEICHTINGER (*Z. Pflanz. Düng.*, 1928, **7B**, 119—127).—On light and medium soils better results were obtained with winter wheat and rye by adding the whole amount of the nitrogen fertiliser in spring than when used part in autumn and part in spring. On stiff clays it was preferable to apply the whole of the nitrogen fertiliser in autumn. With rye on sandy soils, the increased yield obtained by a complete nitrogenous dressing in spring as compared with a divided dressing in spring and autumn, was mainly in the straw. In this respect the efficiency of a unit of nitrogen added in spring was five times that of a unit added in autumn when calculated from a Mitscherlich growth-curve.

A. G. POLLARD.

Influence of calcium cyanamide on the bacterial life of soils. J. KUHN and O. DRECHSEL (*Z. Pflanz. Düng.*, 1928, **7B**, 105—118).—Calcium cyanamide causes considerable increases in the bacterial numbers of soils for a period of 2—3 months from its use. This effect is specific to "nitrolim," and is not brought about by free cyanamide either alone or mixed with lime. It is not the result merely of treatment of soil with lime and nitrogen fertiliser. Sodium cyanamide produces relatively small increases in bacterial numbers. The stimulation of bacterial growth following the use of "nitrolim" is greater in neutral and alkaline soils than in acid ones.

A. G. POLLARD.

Sulphur in relation to the soil solution. W. L. POWERS (*Univ. Calif. Pub. Agric. Sci.*, 1927, **5**, 119—170).—The application of sulphur and sulphates to Madera sand soil increased the bases, especially calcium, in the soil solution. Heavy applications of sulphur increased the acidity, at first increasing the iron and phosphate content of the solution, then decreasing it, and inhibiting nitrification. Normal application (100 lb. per acre) increased growth and the nitrogen content of arid soil. Application of sulphur may produce a more concentrated soil solution and thus decrease transpiration.

CHEMICAL ABSTRACTS.

Chemical changes in dusting mixtures of sulphur, lead arsenate, and lime during storage. H. S. SWINGLE (*J. Agric. Res.*, 1928, **36**, 183—192).—A combined insecticide and fungicide dust (sulphur 80%, lead hydrogen arsenate 5%, calcium hydroxide 15%), which is commonly used in the United States, usually becomes dark grey, and eventually almost black, on storage.

This darkening is shown to be due, firstly, to a reaction between the calcium hydroxide and sulphur to form calcium sulphide and sulphite, and secondly, to the reaction of the calcium sulphide with lead arsenate, giving calcium arsenate and lead sulphide. Hot dry conditions of storage favour this change, which, however, has little or no effect on the efficiency of the mixture, the greatest amount of sulphur involved in the reactions in dusts stored for a year being less than 0.5% of the total amount present. Field experiments indicated that there was some increase in the risk of damage to foliage by the use of old samples.

C. T. GIMMINGHAM.

Root-soluble nutrients in soils and subsoils.

M. KLING and O. ENGELS (*Z. Pflanz. Düng.*, 1928, **7B**, 127—139).—Examination is recorded of a large number of surface soils and subsoils. In general, the subsoils are the poorer in root-soluble potash and phosphate, but the relative proportions in soil and subsoil vary enormously. Decrease of nutrients with depth of soil was more marked with phosphate than with potash. Extraction of soils with 10% hydrochloric acid gives no information as to the amount of root-soluble nutrients present. By the Neubauer method one third of the soils examined was deficient in potash and two thirds were deficient in phosphate. The importance of examining subsoils for nutrients, to depths varying with the nature of the root system of the growing crop, is emphasised.

A. G. POLLARD.

Zeolite formation and base-exchange reactions in soils. P. S. BURGESS and W. T. MCGEORGE (*Arizona Agric. Exp. Stat. Tech. Bull.*, 1927, [15], 359—398).—Soil permeability is little hindered by simple silicates, but always completely retarded by aluminium hydroxide or hydrated aluminium silicate gels (zeolites) when precipitated within the soils. Sodium silicate and aluminate are usually present in the soil solution of strongly alkaline soils. When solutions of these substances are added to soils, and the latter air-dried, their base-exchange capacities are greatly augmented; addition of sodium hydroxide to certain soils has a similar effect. Synthesis of artificial zeolites from different proportions of the constituents affords the same zeolite, apparently scolecite. Zeolites are considered to be chemical compounds capable of ionising and entering into true chemical reactions. Soil organic matter appears not to possess a true base-replacement capacity, but rather the ability merely to adsorb solutes.

CHEMICAL ABSTRACTS.

Dye adsorption by hydrous alumina in soils.

H. H. CROUCHER (*J. Agric. Sci.*, 1928, **18**, 350—362).—Examination of the adsorption of a large number of dyes by hydrous alumina led to the selection of Biebrich-scarlet (for acid adsorption) and iodine-green (for basic adsorption) as suitable for adsorptive measurements of soil colloids. The presence of hydrous alumina in soils was demonstrated, and results of the examination of soils by the adsorptive process are recorded.

A. G. POLLARD.

Influence of fertilisers on the vitamin-B content of wheat. C. H. HUNT (*Ohio Agric. Exp. Stat. Bull.*, 1927, [415], 1—41).—The results suggest that acid phosphate alone or in a complete fertiliser with potassium chloride and sodium nitrate produces wheat with the

highest vitamin-B content. Potassium chloride and sodium nitrate together, in two years out of three, produced wheat of the lowest vitamin content. Climate causes significant variations. There was no evidence that spring wheat contains more vitamin-B than winter wheat, nor was any parallelism observed between the vitamin-B content and that of calcium and phosphorus. The reproductive history of rats is a more sensitive criterion of the vitamin-B content of wheat than are growth curves.

CHEMICAL ABSTRACTS.

Nutritive value of pasture. III. Influence of intensity of grazing on the composition and nutritive value of pasture herbage. I. H. E. WOODMAN, D. B. NORMAN, and J. W. BEE (*J. Agric. Sci.*, 1928, 18, 266—296).—Comparison is made of the feeding value of pasture herbage cut weekly and fortnightly. No important difference in chemical composition or digestibility was observed. Although there was a slight widening in the nutritive ratio in the fortnightly- as against weekly-cut herbage, the value was still definitely narrower than that for milk. The total production of herbage during a season somewhat favoured fortnightly cutting. This difference is to be expected to increase during unfavourable growing weather. (Cf. B., 1926, 506; 1927, 588.)

A. G. POLLARD.

Influence of soil heterogeneity on growth and yield of successive crops. T. EDEN and E. J. MASKELL (*J. Agric. Sci.*, 1928, 18, 163—185).—Results of field trials were examined mathematically with a view of correlating physical conditions of the soil and plant growth. Ploughing draught, as a measure of physical condition, showed a fairly close correlation with the germination of wheat, but not with its later development. With swedes no definite relationships were apparent. In both cases the effect of the spacing of the plants on their growth and development was of primary importance.

A. G. POLLARD.

Maize silage. II. H. E. WOODMAN and A. AMOS (*J. Agric. Sci.*, 1928, 18, 194—199; cf. B., 1924, 885).—The preparation and feeding trials of maize silage are described. Silage made in towers from the French variety of maize, *Jaune Gros du Domaine*, showed a higher digestibility and nutritive value than that from American maize or the "green fruity" oat and tare silage made in this country.

A. G. POLLARD.

Micro-Kjeldahl method for determination of total nitrogen in soils. I. POLLAK and R. DIETZ (*Oesterr. Chem.-Ztg.*, 1928, 31, 57—58).—A modified method is described which gives results in close agreement with the macro-method and for which greater rapidity is claimed. 1 g. of soil passing a 1 mm. sieve is digested with sulphuric acid in a small flask together with a drop of mercury and a mixture of copper and potassium sulphates. After digestion ($\frac{1}{2}$ —1 hr.), the contents of the flask are made up to 200 c.c. and 10 or 20 c.c. are withdrawn for distillation. After the addition of sodium hydroxide and sodium thiosulphate the ammonia is distilled in steam into *N*/70-hydrochloric acid and is titrated in the usual way.

H. J. G. HINES.

Use of the Lundegårdh apparatus for the determination of the carbon dioxide production of forest soil. D. FEHÉR (*Biochem. Z.*, 1928, 193, 350—

355).—The apparatus (B., 1927, 662) is simplified and cheapened without losing accuracy.

P. W. CLUTTERBUCK.

Page and Williams' method for the determination of the saturation capacity of soils. P. E. TURNER (*J. Agric. Sci.*, 1928, 18, 257—265).—In this method (B., 1925, 108) the leaching of the soil treated with calcium carbonate to a total volume of 2 litres is insufficient to remove all exchangeable bases from the soil, and should be continued to 3 litres or more. Alternatively, the size of the sample of soil should be reduced. The difference in the amount of calcium appearing in the first and second litres of extract as a result of dissolution of the calcium carbonate is negligible. The displacing reaction proceeds very slowly when nearing completion, and for this reason Hissink's method for determining saturation capacity is inaccurate. The ratio of displaced calcium appearing in the second litre of extract to the total amount is influenced to some extent by the nature of the colloidal matter present.

A. G. POLLARD.

Specific conductivities of soil extracts. C. H. WRIGHT (*J. Agric. Sci.*, 1928, 18, 186—193).—The specific conductivity of Nigerian cotton soil extracts varied considerably with the season, and tended generally to reach a maximum in March and a minimum in June—July. Comparison of the specific conductivities of different soils can only be made when samples are drawn at the same period. There was a relationship between the conductivity of 1:5 soil: water extracts and the yield of cotton lint, and the former may be taken as a measure of soil fertility. Decreased specific conductivity of soils under continuous cropping is paralleled by the decreased solubility of the soils.

A. G. POLLARD.

Apparatus for measuring the hydrogen-ion concentration of the soil. R. H. BRAY (*Ind. Eng. Chem.*, 1928, 20, 421—423).—The electrode vessel is a Gooch funnel with the stem bent up, and a perforated porcelain plate sealed into the bottom. The entering hydrogen bubbles up through the plate at a sufficient rate to keep the soil particles (10 g. of soil in 25 c.c. of distilled water) in suspension, and escapes from a hole in the stopper which also serves for the entrance of the agar-potassium chloride tube. The electrode is a piece of approximately 20-gauge platinum wire 10 cm. long, sealed into a glass tube, bent to a spiral cone, and plated with palladium-black. The agar-potassium chloride tube is connected with saturated potassium chloride solution, connected again with a saturated calomel half-cell. The electrodes must have reached equilibrium before readings are taken, and should be checked when newly plated against a standard phthalate buffer solution.

D. G. HEWER.

Determination of the p_H values of turbid soil and other solutions. C. H. GADD (*J. Agric. Sci.*, 1928, 18, 206—208).—To each of two similar portions of a 1:2 soil: water suspension decanted after being kept for 24 hrs. is added the requisite amount of indicator solution prescribed by Clark's method. To one of the samples a buffer solution of known p_H value is added, and the colour of the mixture compared with the second or control sample. The process is repeated with other

buffer solutions until no colour change (or a minimum) results.

A. G. POLLARD.

An index of soil texture. F. HARDY (J. Agric. Sci., 1928, 18, 252—256).—For soil survey purposes an "index of texture" (*I.T.*) is developed, and is evaluated thus: $I.T. = P - S/5$, where *P* is the moisture content of the soil at its point of stickiness and *S* is the sand content. Although distinguishing only main soil types, and giving no indication of organic content, the index of texture offers a single-valued and rapidly-determined figure for soil characterisation. Values obtained are closely paralleled by those of hygroscopic coefficients, the mean ratio to which for a number of soils is 4—6.

A. G. POLLARD.

Mechanical analysis of heavy ferruginous soils. R. C. GROVES (J. Agric. Sci., 1928, 18, 200—205).—The difficulty of dispersing ferruginous clays prior to mechanical analysis is overcome by a modification of the Robinson method, whereby the soil organic matter is broken up by heating with successive small quantities of ammoniacal hydrogen peroxide. The residual soil may be worked up into a suitable suspension merely by gentle rubbing with a rubber pestle and decanting in portions. Mechanical shaking is unnecessary.

A. G. POLLARD.

Soil stratometer. Examination of deep-lying soil. M. ZAGHLOUL (Nature, 1928, 121, 537).—A simple instrument for measuring variations of the mechanical resistance of soil in a vertical direction is described.

A. A. ELDRIDGE.

Analysis of insecticides. Dry products: naphthalene; naphthalene and camphor; blue powders containing strychnine; red corn. M. FRANÇOIS and (Mlle.) L. SEGUIN (J. Pharm. Chim., 1928, [viii], 7, 331—340).—Methods are given for the identification and determination of naphthalene and camphor in admixture, strychnine, brucine, and other alkaloids in presence of methylene-blue, and arsenic in blue powders, and of strychnine sulphate and colouring matters in impregnated corn.

E. H. SHARPLES.

Analysis of sugar beets. SHERWOOD.—See XVII.

PATENTS.

Production of insecticides. E. C. R. MARKS. From ASSOCIATED OIL CO. (B.P. 287,371, 4.7.27).—The products contain basic compounds extracted with sulphur dioxide from crude shale oil or other hydrocarbon oils and subsequently purified by extraction with sulphuric acid and steam distillation of the extract after neutralisation with sodium hydroxide, a refined oil containing, preferably, at least 90% of unsulphonatable residue, and an ammonium salt of a fatty acid. Inert material, e.g., kieselguhr, and a red dye may also be added, the mixture being worked up to a stable emulsion with water.

L. A. COLES.

Fungicides for seed grain and the like. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 287,246, 20.12.26).—The fungicides contain arsenious or arsenic acid or their salts and mercury compounds of phenol or its homologues, e.g., mercurinitrophenol, with or without

the addition of copper compounds or other fungicides and talc, gypsum, kieselguhr, or other inert material.

L. A. COLES.

Fungicide. H. L. KLEINHANS, Assr. to C. COOPER & Co., INC. (U.S.P. 1,662,462, 13.3.28. Appl., 1.11.24).—A mixture of sulphur and copper carbonate so proportioned as to be miscible with water is used. The copper carbonate neutralises the acids generated by the sulphur in the soil.

F. G. CLARKE.

Insecticide (U.S.P. 1,663,883).—See XXIII.

XVII.—SUGARS; STARCHES; GUMS.

Use of the refractometer in the analysis of individual sugar beets. S. F. SHERWOOD (J. Agric. Res., 1928, 36, 41—51).—Total soluble solids in the juice from very small samples of sugar-beet pulp may be determined satisfactorily and rapidly by measuring the refractive index of a few drops of the juice squeezed from the pulp with the fingers and allowed to fall directly upon the prism of the refractometer. The determination may be delayed with safety up to 24 hrs. if the pulp is stored in air-tight containers in a refrigerator at about 12°. The sucrose present is not, however, proportional to the total solids, and it should be determined by means of the polariscope. The ratio of % sucrose in juice: % solids by refractometer is called "the apparent purity by refractometer," and is regarded as a valuable criterion for judging the comparative quality of individual beets.

J. R. NICHOLLS.

Extracts from a [sugar] laboratory notebook. J. P. OGILVIE (Internat. Sugar J., 1928, 30, 120—126).—Using Spörry's "equal-effect" method (B., 1923, 369 A) "Carboraffin," "Anticromos," "Supra Norit 5 X," "Supra Norit 3 X," "Suchar," and "Radit" were amongst the carbons found to give the best results in the decolorisation of molasses solution (*d* 1.03). Examined by the same process, the amount of decolorising earth (as used in the oil industry) necessary to produce the same effect as 1 pt. of "Superior Norit" was not found to be less than 8—9 pts. by wt.; although of low decolorising power, however, such earths may considerably assist in the production of clear and light-coloured juices or syrups. Sodium hyposulphite was shown to decompose at a rapid rate on exposure to air, and packing in small hermetically-sealed containers is advised. Lime in the form of hydrate is advocated for use in tempering cane juices, on account of its high purity and convenience of packing for shipment in paper-lined bags. A good filtration kieselguhr should have an apparent density of from 12 to 17 lb./cub. ft., should consist mainly of relatively large diatoms (preferably acicular in form), and should be free, or almost so, from amorphous particles of clay or chalk. Arsenic determinations in products used in cane and beet sugar manufacture gave the following results for As₂O₃ in grains/lb.:—limestone, 1/70—1/240; coke, 1/10; unrefined sulphur rolls, 2.0; refined ditto, 1/140; concentrated superphosphate, 4½; "Sumaphos," 1/117; ultramarine-blue, 1/5.

J. P. OGILVIE.

Calculation of purity factors for dry lead defecation at 20°. E. W. RICE (Sugar, 1927, 29, 522).—

Errors amounting to 28% are liable to be made if purity values are calculated from tables meant to be used with Mohr c.c. whilst actually the polarimetric readings are taken at 20°. A table of factors calculated for 20° to be used with the dry lead method is presented.

J. P. OGILVIE.

Control of p_H value in the sugar industry. BARBAUDY (Chem. et Ind., 1927, 18, 984—992).—A review.

J. P. OGILVIE.

Starch in gluten bread. FLEURY and BOYELDIEU.
Honey. FIEHE and KORDATZKI, also FIEHE.—See XIX.

PATENTS.

Batteries for continuous diffusion of sugar and the like. A. RAK (B.P. 286,125, 7.5.27).—The slices of beet move upwards through a succession of towers, and the diffusion liquid flows downwards and counter-current to the slices.

B. M. VENABLES.

Cleaning of chicle and similar gums. D. W. HOWE, E. R. PICKETT, and D. M. McBEAN, Assis. to BEECH-NUT PACKING Co. (U.S.P. 1,664,981, 3.4.28. Appl., 13.7.25. Renewed 2.9.27).—The gum is emulsified in hot water, the emulsion is filtered, and the gum and excess water are separated.

H. ROYAL-DAWSON.

XVIII.—FERMENTATION INDUSTRIES.

Carbonyl value of wine. H. STRACHE and A. BRANDL (Z. Unters. Lebensm., 1928, 55, 50—53).—Aldehydes and ketones play a considerable part in the bouquet of wine, and the kind is more important than the quantity; a determination of the carbonyl group is of value in characterising a special kind of wine or a particular vintage. For this purpose a definite quantity of phenylhydrazine is added to the distillate from 100 c.c. of wine, and the nitrogen evolved from the uncombined phenylhydrazine after boiling with Fehling's solution is measured. The carbonyl oxygen from the wines examined varied from 0.0078 to 0.0239 g./litre, being higher from the wines with stronger aroma. The major part of the carbonyl compounds was found to be readily volatile, and after two thirds of the wine had been distilled no more came over. In the case of Muscatel and Chablis, the residue after distillation on being steam-distilled gave an additional quantity.

J. R. NICHOLLS.

Oenological indices and reversion of malic acid in wines. L. FERRÉ (Ann. Falsif., 1928, 21, 75—84).—Malic acid is an important factor in the relation of alcohol content and acidity in wines, and frequently diminishes in wines that have been kept, whilst the lactic acid increases. If the acidity of a wine and its content of lactic acid are known, the initial acidity may be calculated, and it is considered logical to calculate all the oenological indices to the original acidity. The proportion of tartaric, malic, and lactic acids in a wine forms a useful foundation in giving an opinion on the wine, lactic acid chiefly indicating the presence of malic acid reversion.

D. G. HEWER.

Adulteration of brandy and its detection. (A) H. ZELLNER (Z. Unters. Lebensm., 1927, 54, 389—390). (B) G. GRAFF (*ibid.*, 391—393).—(A) Statements made in

a lecture on the above subject by Graff (B., 1927, 953) are criticised. The fusel oil and ester number recommended by Graff are deprecated on the ground that the content of higher alcohols in wines and therefore in wine products varies too widely. Whilst analytical numbers are not to be despised, taste and odour are the most reliable indications that wine products so-called are genuine.

(B) A reply. It is claimed that tasting must occupy a secondary position seeing that it is subjective. Further, the addition of sugar to wine before fermentation, or of rectified alcohol before distillation, cannot be detected by taste, but can be detected by determining the amount of higher alcohols present, these being in lower proportion in wine products so adulterated.

W. J. BOYD.

Detection of cider in wine by microscopical examination of the sediment. L. MINDER (Z. Unters. Lebensm., 1927, 54, 387—388). A. WIDMER and O. E. KALBERER (*ibid.*, 388; cf. B., 1927, 568).—A question of priority.

W. J. BOYD.

Protomalt. NEUMANN.—See XIX.

XIX.—FOODS.

Glutelins of the cereal grains. R. K. LARMOUR (J. Agric. Res., 1927, 35, 1091—1120).—Alkali-soluble proteins were prepared from wheat, spelt, rice, maize, oats, einkorn, emmer, durum wheat, teosinte, rye, and barley. The general method of preparation was to treat the alcohol-insoluble grain with 0.2% alkali, to super-centrifuge, and to precipitate the soluble proteins with hydrochloric acid. The precipitate was washed with alcohol and ether, dried, and ground. The nitrogen distribution was determined by the Van Slyke method (B., 1911, 1135) whereby values were obtained for ammonia-nitrogen, total humin-nitrogen (including acid-soluble, acid-insoluble, and phosphotungstic acid-insoluble), total basic nitrogen (including arginine, cystine, lysine, and histidine), and total filtrate nitrogen (including amino- and non-amino-). The results showed well-marked relationship between the various preparations, especially in respect of the basic nitrogen fraction. Glutenin of wheat (*Triticum vulgare*) and oryzanin of rice (*Oryza sativa*) occupy positions at the extreme limits of the class in respect to ammonia and total basic nitrogen, and the corresponding values for the other proteins described fall within these limits. This is submitted as evidence that the preparations obtained belong to a definite class of proteins, the glutelins, which is represented in all the cereal grains thus far studied. A nomenclature for these substances is suggested.

J. R. NICHOLLS.

Determination of dextrose in presence of proteins; with particular reference to starch in "gluten" bread. P. FLEURY and G. BOYELDIEU (J. Pharm. Chim., 1928, [viii], 7, 207—214, 249—255).—The dextrose in a standard solution was determined by the method of Fleury and Boutot (A., 1922, ii, 879) in the presence of certain proteins and their degradation products. Albumins and amino-acids had no appreciable effect on the result, but albumoses and peptones caused a diminution in the amount of reduced copper (cf. Pringsheim and Winter, A., 1926, 1274; Neuberger and Simon, A., 1927,

450). Peptones etc. were removed therefore by treating the solution with neutral lead acetate or preferably acid mercuric sulphate solutions. The method of analysis recommended is as follows:—The dried sample of bread is hydrolysed with dilute sulphuric acid at 120° for 20—25 min. An aliquot proportion of the cooled solution is then treated with mercuric sulphate solution (200 g. of yellow mercuric oxide and 175 c.c. of sulphuric acid, *d* 1.83, per litre), neutralised until just acid to litmus, filtered, and the filtrate treated with zinc dust. The dextrose is finally determined polarimetrically or preferably by reduction in the new filtrate. S. COFFEY.

Addition of protomalt to rye bread. M. P. NEUMANN (*Z. Unters. Lebensm.*, 1927, 54, 341—347).—A new bread improver for accelerating the fermentation process in rye dough has been examined. The extract known as protomalt on analysis was found to contain: water, 29.3%; protein, 6.15%; amino-nitrogen, 0.08%; nitrogen-free extract, 63.35%; reducing sugar, 43.60%; sugar reducing after inversion, 51.60%; starch, nil; ash, 1.20%; it had p_H 4.9, acidity 16.2, diastatic value 17, viscosity (100 c.c. of 1:1 solution) 80 sec. (water 34 sec.). Fermentation trials showed that protomalt added to rye flour greatly promoted the development of acidity. Baking tests were carried out with leaven and with yeast, with and without protomalt. The bulkiness of the bread made with leaven was greater than that of the yeast bread, the pores were more uniform, and the results more certain. Protomalt added to the "sponge" in the yeast process gave a better bread than that obtained without it, but its addition to the dough had less satisfactory results. The addition of protomalt to the complete leaven gave a very good bread of greater bulk without the excessive browning of the crust usually found when malt preparations are used. Added to the dough it had little effect. W. J. BOYD.

Properties and uses of an edible rice cellulose. E. R. HARDING (*Ind. Eng. Chem.*, 1928, 20, 310—311).—A pure edible cellulose may be prepared from the hulls of rice and other cereals by a strong soda boil followed by a hydration treatment, the object of which is to reduce the material to a suitable physical form. The cellulose so obtained (α -cellulose 73%, hydrated cellulose 24%, ash 0.7%) is used for raising the roughage content of many types of food. W. J. POWELL.

Detection and determination of rice flour in other flours and in spices. M. WAGENAAR (*Z. Unters. Lebensm.*, 1927, 54, 357—369).—Microscopical and chemical methods for the detection of rice flour in buckwheat, oat, wheat, rye, and barley flours, and in pepper, mustard, mace, nutmeg, allspice, cloves, cinnamon, and cocoa are described. The microscopical appearance of the starch grains, aleurone layer, germ tissue, and the effect of various reagents on these, provide means of distinguishing particles of rice from those of other products. A quantitative microscopical count method has been devised by the author depending on the adsorption of an acid dye, *e.g.*, fuchsin-S, by the protein granules. The peculiar distribution of these in the rice grain gives a characteristic appearance to the particles of rice flour. The actual proportion of

rice present is calculated from the number and size of the rice particles in a known quantity of flour.

W. J. BOYD.

Correlation of kernel texture, test weight per bushel, and protein content of hard red spring wheat. J. H. SHOLLENBERGER and C. F. KYLE (*J. Agric. Res.*, 1927, 35, 1137—1151).—Data from 1290 representative samples of hard red spring wheat were studied. A fairly strong tendency was noted for protein content to increase with the proportion of dark, hard, and vitreous kernels, and there was a significant correlation between test weight and protein content, the latter increasing with the former up to 54 lb. per bushel and then decreasing. The variation in protein was found to be more influenced by kernel texture than by test weight, and no correlation was observed between the latter. A method is presented for estimating the protein content when the kernel texture and test weight are known. For this purpose a table is given relating the percentage of dark, hard, and vitreous kernels with that of proteins; a second table gives the correction to be applied to the protein for the test weight.

J. R. NICHOLLS.

Solids-not-fat as criterion for watered milk. C. E. KLAMER (*Z. Unters. Lebensm.*, 1928, 55, 45—47).—The limit of 8% for solids-not-fat in milks is stated to have no essential significance with regard to watering. The determination of the f.p. is considered to be much more reliable. For genuine milks the f.p. generally lie between -0.53° and -0.55° , although some milks give values between -0.52° and -0.53° or above -0.55° . Numerous examples are given of unwatered milks with less and watered milks with more than 8% of solids-not-fat. The f.p. of the genuine milks were from -0.530° to -0.550° , and of the watered milks from -0.486° to -0.512° . The f.p. is regarded as the final criterion. J. R. NICHOLLS.

Milk from sick cows. J. KRENN (*Z. Unters. Lebensm.*, 1928, 55, 47—49).—Analyses are given of the milk from sick cows. The values are exceptional and might suggest dilution, but the usual chemical analysis together with the f.p. and the chlorine-sugar value show these milks to be abnormal. J. R. NICHOLLS.

Effect of working of cows on the composition of the milk. F. HAUN (*Z. Unters. Lebensm.*, 1927, 54, 337—341).—The chief alteration in the composition of the milk of cows put to work is in the fat content, which may increase by more than 1% in 24 hrs. The density of the milk and of the serum and the refraction all show lower values on days of rest between days of work. The yield of milk decreases greatly when the animal is put to work. W. J. BOYD.

Preservation of milk samples for fat determination. P. MARSCHEW (*Milch. Zentr.*, 1928, 57, 85—88).—Varying quantities of formalin, potassium dichromate, and sublimate were added to milk, and after definite periods the fat was determined by three butyrometric methods. A saturated solution of potassium dichromate is regarded as the best preservative, and to keep 100 c.c. of milk at 15—19° for 14 days, 0.5—1.0 c.c. is required; for 1 month, 1.5 c.c.; and for milk at 19—20° for

14 days, 1.5 c.c. Using formalin, 100 c.c. of milk at 15—19° require 0.2 c.c. to keep it for 14 days and 0.3 c.c. for 1 month, and the fat must be determined by the Gerber process, using sulphuric acid of d 1.815 or above. To keep a similar quantity of milk for 14 days at 15—19°, 0.6—0.8 c.c. of 5% solution of sublimate is necessary.

J. R. NICHOLLS.

Determination of milk fat in small quantities of fat. J. GROSSFELD and F. WISSEMANN (*Z. Unters. Lebensm.*, 1927, **54**, 352—356).—For the examination of fat extracted from foodstuffs of low fat content, the ordinary methods are unsuitable, owing to the large quantity of material which has to be treated. The semi-microchemical methods of Lührig (*B.*, 1922, 725 A) for the determination of the Reichert-Meißl and saponification values are described. For these, the amounts of sample and reagents used are reduced to one tenth of those required for the ordinary methods. The author describes a similarly reduced semi-microchemical method for the determination of the butyric acid value. The distillate of 11 c.c. is titrated with 0.01*N*-sodium hydroxide solution. The results are in agreement with those obtained by the macro-method. W. J. BOYD.

Determination of chlorine and sodium in mammalian milks. L. BARTRE and E. DUFILHO (*Ann. Falsif.*, 1928, **21**, 98—100).—Mares' milk was found to have less than 0.006 g. of sodium per litre; during the colestrum period it contained over 1 g. of chlorine per litre and later between 0.5 and 0.7 g. per litre. Human milk also contained at first over 1 g. of chlorine per litre and later between 0.6 and 0.7 g., and the sodium content was negligible till after about the 45th day. It is concluded as a result of these and previous investigations that sodium is not necessary to the newly born, and serves later for the combination of other necessary elements: mares' milk may to a certain extent be substituted for human milk, but after the fifth month of lactation the latter more closely resembles cows' milk.

D. G. HEWER.

Detection of adulterants in cacao butter in chocolate. COLOMBIER and CHAIZE (*Ann. Falsif.*, 1928, **21**, 91—97).—The following adulterants of cacao butter were divided into two classes, viz. (a) comprising "Kunérat, Chocotal, and Karamélol," and (b) "Illipé, Pontianak, and Banka," with "Kayao" in a separate class. For a sample of 100—125 g. of chocolate 10 g. are extracted for total fat, 20 g. are used for sugar determination, and the remainder is extracted with carbon tetrachloride and on the residue are determined, after two days, (1) refractive deviation, (2) soluble and (3) insoluble volatile acids, and (4) the H.M. value, derived from a modification of Halphen's method (*B.*, 1909, 841) which consists in dissolving 1.5 g. of fat in 3 c.c. of carbon tetrachloride, adding drop by drop a solution of bromine in an equal volume of carbon tetrachloride until an orange colour is obtained, shaking, filtering, and adding to the clear filtrate an equal volume of light petroleum, when, with fats containing enough Banka or illipé butters, a white band forms at the surface of contact; a turbidity in the light petroleum is visible with 5% of these fats. If the sum of values (2) and (3) is greater than (1), a fat of class (a) is present, and if (4)

is positive a fat of class (b). If (4) is negative, and (2) and (3) less than (1) all fats of classes (a) and (b) are absent, whilst one fat of each group present together may also be detected. To detect Kayao fat the fact that a blue fluorescence forms in Wood's light is utilised. The method is applicable to nut and milk chocolates.

D. G. HEWER.

Determination of coating material in coffee. A. SCHUGOWITSCH (*Z. Unters. Lebensm.*, 1927, **54**, 330—336).—In Austria and other countries the use of coating material such as colophony or sugar for brightening coffee is controlled by defining the permissible proportions of ether, alcohol, and water extracts. It is shown that whilst these values are nearly constant for all kinds of raw coffee, in roasted coffee the alcohol- and ether-soluble portions vary according to the kind of coffee and the degree of roasting. The aqueous extract increases with increased roasting, but is nearly constant for all varieties. In very strongly roasted beans the amount of ether and alcohol extract is a multiple of that in normally roasted beans and exceeds very considerably the limits hitherto permitted. In normally roasted beans (dull brown) the ether and alcohol extract never exceeds 1% nor the aqueous extract 0.50%; if these values are exceeded artificial brightening may be assumed with certainty. During storage the ether extract of roasted coffee almost always increases and the alcohol extract decreases. For the detection of colophony in coffee, 10 g. are shaken for 3 min. with 30 c.c. of 0.5*N*-caustic soda and some kieselguhr. The filtered extract is acidified with dilute sulphuric acid and shaken with ether. The ether extract is washed with water, evaporated, and the residue dissolved in 1—1.5 c.c. of acetic anhydride. To this in an inclined test-tube a drop of sulphuric acid (62.5%) is carefully added, whereupon a blue-violet coloration develops, changing to dirty brown on shaking and finally to yellow. In the absence of colophony no colour or only a faint rose tint develops.

W. J. BOYD.

Acidity of honey and artificial honey. J. FIEHE and W. KORDATZKI (*Z. Unters. Lebensm.*, 1928, **55**, 59—63).—Forty-two samples of honey and 12 of artificial honey have been examined. The p_H values for natural honeys were about 3.9 (limits 3.8 and 4.3); and for artificial honeys 3.2 (limits 3.0 and 4.0). The number of c.c. of *N*-alkali required to neutralise 100 g. to phenolphthalein was 0.6—4.0 for pure honey and 0.38—0.90 for artificial. Mixtures gave p_H values sometimes within and sometimes outside the limits for pure honey.

J. R. NICHOLLS.

Separation of honey. J. FIEHE (*Z. Unters. Lebensm.*, 1928, **55**, 64—65).—A honey which was apparently regularly crystallised had a dazzling white surface layer sharply differentiated from the remainder. The honey was examined in three portions, viz., the surface layer, the upper half of the remainder, and the lower half. The white layer was richer in sucrose, dextrose, salts, and non-sugars. The water contents of the three layers from top to bottom were 6.6%, 15.7%, and 11.25%. It is assumed that shaking had aerated the honey, and that on being kept the air bubbles had risen, carrying sugar particles etc. to the surface. The top layer had

lost water by evaporation, and the bottom layer had less water than the middle on account of crystallisation of sugar.

J. R. NICHOLLS.

Keeping qualities of frozen meat, especially frozen sausage meat. O. ACKLIN (Z. Unters. Lebensm., 1928, 55, 31—44).—Employing appearance, smell, and bacteria ratio as criteria, no fundamental difference with regard to deterioration was observed between frozen and native meat after storage at 4°, 20°, and 37° in raw and in cooked states. No difference was observed between first-quality native sausage meat and 10—16 months' old frozen meat after storage at an average temperature of 15° and at an average humidity of 60%, with systematic control of character, oxygen consumption, methylene-blue reduction, and acidity and rancidity of the fat.

J. R. NICHOLLS.

Treatment of meats via the blood stream. A. GAUDUCHEAU (Ann. Falsif., 1928, 21, 84—90).—Flesh of chickens, rabbits, pigeons, etc. may be flavoured as required by injecting suitable substances directly into the blood stream. The muscles readily take up the aroma, and the changes taking place are being studied with a view to the commercial application of the technique.

D. G. HEWER.

Rapid test of pepper powder for excessive chalk content. W. PLAHL (Z. Unters. Lebensm., 1927, 54, 369—371).—A suitable quantity of pepper powder is placed on a microscope slide glass, quickly mixed with 2 or 3 drops of chloral hydrate solution containing about 1% of dilute hydrochloric acid, and covered with a cover glass. The evolution of bubbles of carbon dioxide is noted. Some trials with unadulterated pepper, and with pepper of known chalk content, give the experience necessary for the recognition of adulterated samples. With ash content up to 7% in black pepper, and 4% in white pepper, only very slight evolution of gas occurs, in bubbles visible under the microscope or with a strong lens. In samples with 13% or 14% of ash, the bubbles are readily seen with the unaided eye.

W. J. BOYD.

Chemical analysis of nutritive preparations. E. KOMM and R. MÜLLER (Z. Unters. Lebensm., 1928, 55, 53—59).—The analysis of such preparations must be sufficient to indicate their compositions so that conclusions can be drawn as to their biological values. An example is given of the examination of "Provita" in which the following were determined:—water, total and water-soluble sugar substances, total lipoids, cholesterol, ether-soluble and alcohol-soluble phosphatides, fat, nitrogen (total, lipid, and water-soluble), tyrosine, tryptophan, fibre, total and water-soluble ash (and its analysis).

J. R. NICHOLLS.

Technique of biological protein differentiation. A. BRÜNING and B. KRAFT (Z. Unters. Lebensm., 1927, 54, 347—352).—A semi-microchemical method for the differentiation of proteins by the precipitin reaction is described. The tubes used are 63—65 mm. long, with internal diameter 4.5 mm. The pipettes are 12 cm. long and 3.5 mm. wide with drawn-out points 12 mm. long, so adjusted in aperture that the liquid flows out in a series of drops. The tubes are placed in a stand and held firmly by their middles by metal clips

so that the liquid can be freely observed. For greater quantities of material tubes 7.5 mm. wide and 65 mm. long can be used. A simplified technique for the Hauser capillary method is also described.

W. J. BOYD.

Detection and determination of methyl *p*-hydroxybenzoate in foodstuffs. F. WEISS (Z. Unters. Lebensm., 1928, 55, 24—31).—Methyl *p*-hydroxybenzoate, recommended as a preservative for foodstuffs etc. (cf. U.S.P. 1,627,342; B., 1927, 457), appears in commerce under the names of "Solbrol" and "Nipagin." It can be extracted quantitatively from weakly acid solutions by means of ether or a mixture of light petroleum and ether, and the solvent can be evaporated without loss if the temperature is not allowed to exceed 40°. Potassium ferrocyanide and zinc acetate may be used as cleaning agents to remove proteins without carrying down any appreciable quantity of the ester. From fats and oils it can be partially extracted with hot water or by steam distillation, but in neither case is the extraction complete. With Millon's reagent the ester gives a gradually increasing red colour, the non-appearance of which within 15 min. indicates its absence. Salicylic acid under the same conditions gives a brownish-red colour which is, however, usually distinguishable. The ester does not interfere with the tests for salicylic and benzoic acids and only large quantities interfere with the test for mikrobin (B., 1927, 795). The colour produced with Millon's reagent can be compared with that produced under the same conditions from known quantities, or the methyl alcohol can be determined after hydrolysis with potash or by Zeisel's method.

J. R. NICHOLLS.

Report of the Government Laboratory at Amsterdam for 1927. J. STRAUB (Chem. Weekblad, 1928, 25, 161—165).—A brief account of the lines along which original work in connexion with the examination of milk, edible fats, cheese, eggs, flour, and bread has been conducted.

S. I. LEVY.

Composition of Camembert cheese at various stages of ripening. M. P. BABKIN (Trans. Vologda Dairy Inst., 1926, Bull. 65, 205—206).

Rancidity in butter. INIKHOV and SHOSKIN. **Cereal oils.** MUNRO and BINNINGTON.—See XII. **Vitamin-B in wheat.** HUNT.—See XVI. **Lecithins.** MATTHES and BRAUSE.—See XX.

PATENTS.

Manufacture of stable dry substances from colloidal liquids, especially liquids containing fat and albumin. C. KNOCH and F. GROSS (B.P. 285,313, 21.7.27).—After concentration of the colloidal liquid, e.g., milk, by evaporation below 60°, a part of its soluble salts and sugar content is removed by osmosis and evaporated either to dryness or to a crystallisable syrup. The product is then rapidly dried in the form of separate drops on glass or china rollers heated by steam below 60°. The granules are scraped off the rollers, compressed into shapes to remove occluded air, and coated with an air-tight layer by "glazing" with the syrup or by "draging" with the dried powder of salts and sugar.

F. R. ENNOS.

Treatment of vegetable produce. J. B. BUTLER

and J. J. DRUMM (U.S.P. 1,666,551, 17.4.28. Appl., 7.10.26. U.K., 30.11.25).—See B.P. 266,069; B., 1927, 314.

Apparatus for producing refined cocoa liquor from cacao nibs and the like. W. E. PRESCOTT, BAKER PERKINS, LTD., and ANC. ÉTABL. A. SAVY, JEANJEAN & CIE., SOC. ANON. (B.P. 287,628, 22.12.26).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Evaluation of saponin drugs. L. KOFLER and P. A. ADAM (Arch. Pharm., 1927, 265, 624—652).—Physical and chemical methods of determining the strength of saponin drugs are faulty, and reliance has therefore necessarily been placed on determinations of hæmolytic potency. It is now shown that this method is subject to hitherto unsuspected errors. Thus the hæmolytic index of isolated saponins is influenced considerably by the hydrogen-ion concentration of the medium, and in such a way that they can be divided into two classes (cf. Kofler and Lázár, A., 1928, 318). In extracting the powdered drugs, the "reaction" of the medium therefore needs careful control. Further, in any series of tests, only one sample of blood may be used, and from that the corpuscles must be preferably unwashed, as the presence of serum has the same effect on the hæmolytic index of drug extracts as on that of a pure saponin (cf. Kofler and Lázár, *loc. cit.*). Earlier published tables of the resistances of different kinds of blood corpuscles (cow, man, rat, etc.) to saponins are quite untrustworthy, as the order of resistances depends on the particular saponin, or drug, under investigation. Differences are seen in the resistances of blood samples from different individuals of the same species. Values for the hæmolytic index (the authors adhere to Kobert's method of indicating strengths) are thus only comparable among themselves for one series of tests, unless a standard saponin is used as a control. For this purpose "saponin pur. albius." (Merck) is used; this has a hæmolytic index, towards unwashed cow's blood corpuscles, at p_H 7.4, of about 25,000. It should be used in freshly prepared solution. The extraction of the drugs is facilitated by using an alkaline medium, but the alkalinity must be controlled. A phosphate buffer of p_H 7.4, $M/30$, in 0.9% sodium chloride solution is therefore used. This gives directly a solution suitable for hæmolytic tests. The extraction is carried out at water-bath temperature for 30 min. (0.25 g. of drug to 50 c.c. of solution). When the mixture is boiled irregular results are obtained, the saponins apparently undergoing change. When extraction is carried out at room temperature it is incomplete even after 6 hrs. Sarsaparilla, however, behaves abnormally when heated and must be extracted in the cold. The drugs are not satisfactorily extracted in the form of chips, and must be ground, but not powdered, to pass a "No. 5" sieve. Methyl or ethyl alcohol as an extracting medium is unsatisfactory and less efficient than a mildly alkaline saline solution, and itself interferes with the hæmolytic. The suspension of blood corpuscles is conveniently 2—5%, and is made up from defibrinated blood with the phosphate buffer saline solution specified. It is found that the lethal strength of extracts of saponin drugs

towards tadpoles runs parallel with the hæmolytic index, and this property may also be used in determining the strength of such extracts. Many samples of various drugs were examined. Radix senegæ has a hæmolytic index of about 4000 and is fairly constant in strength; radix saponariæ albæ is similar, with an index of about 10,000; cortex quillaia, radix primula, and radix saponariæ rubræ show lower and less constant values, as also does herba herniariæ; radix sarsaparillæ varies over a wide range (hæmolytic index from below 100 to above 3000) and to this variation is attributed the discrepant statements regarding the value of the drug in pharmacy.

W. A. SILVESTER.

Preparation of decoctions of saponin drugs. L. KOFLER and P. A. ADAM (Arch. Pharm., 1927, 265, 652—653).—That the saponins are more thoroughly extracted from the drugs when these are ground rather than in the form of chips has been demonstrated in the preparation of solutions for analysis (cf. preceding abstract). It is now shown that commercial decoctions are also preferably made from ground drugs. The powder should pass a "No. 5" sieve. Frequently, *e.g.*, with water alone, more than twice the usual amount of saponin is extracted. The addition of a small amount of sodium carbonate is beneficial. Extraction may be carried out at the b.p. except with sarsaparilla, which must be extracted cold.

W. A. SILVESTER.

Action of picric acid on tropine silicotungstate. R. HAZARD (J. Pharm. Chim., 1928, [viii], 7, 255—262). Tropine solutions which are too dilute or too acid to give a precipitate with 10% silicotungstic acid solution do so when treated with aqueous picric acid solution, the amount and speed of precipitation being proportional to the amount of tropine in solution. The yellow precipitate is a silicotungstate, and it may be freed completely from both the yellow colour and picric acid by washing with hydrochloric acid. Atropine silicotungstate is very sparingly soluble, and does not give this reaction, which affords a method of separating and identifying the two bases, in which case it is advantageous to work in 2% hydrochloric acid. The hydrolysis of atropine may be followed in this way. ψ -Pelletterine gives a similar precipitate after treatment with silicotungstic acid followed by picric acid in 2% hydrochloric acid.

S. COFFEY.

Determination of morphine in opium. V. MACRI (Boll. Chim. Farm., 1928, 67, 129—131).—Eder and Märki's method (Pharm. Acta Helv., 1927, [2—4]) is discussed. The use of benzene in place of ether for the extraction of the opium does not seem to be of any great advantage, and the author prefers to employ a saturated aqueous solution of morphine for this purpose, and to effect only the final washing with ether or benzene. The introduction of extraneous substances such as sodium carbonate or borax involves possible error and is to be avoided.

T. H. POPE.

Cocaine and Maclagan's test. P. BRETEAU (J. Pharm. Chim., 1928, [viii], 7, 329—331).—The variations of Maclagan's test for isotropylcocaine (truxilline) in different pharmacopœias are discussed and the following procedure is recommended: 0.1 g. of the cocaine salt is dissolved in 80 c.c. of water, and 2 c.c. of 1% ammonia

solution ($d 0.995$) are added, stirring with a wooden or rubber-covered rod. After settling for 15 min. the liquid should be clear. The sides of the vessel are then rubbed from time to time, but not too vigorously, with a glass rod. A crystalline deposit of cocaine should separate and the supernatant liquid should be clear. The presence of isotropylcocaine is indicated by the formation of an opalescent liquid which is cleared by the addition of hydrochloric acid.

E. H. SHARPLES.

Fructus papaveris and sirupus papaveris as possible poisons. L. VAN ITALLIE and A. J. STEENHAUER (Arch. Pharm., 1927, 265, 698—705).—Poisoning has been known to result from the administration of sirupus papaveris, which is a popular soporific for infants. There are considerable discrepancies in published figures for the percentage of morphine in both the fruit and the extract. It is now found that much more morphine may be present than is commonly supposed. Thus *Fructus papaveris* contains 0.4—0.5% of morphine, all of which passes into an aqueous extract; the syrup may contain 0.01—0.06%. In detecting morphine, Pellagri's reaction is modified: an excess of iodine is used, and the excess is removed by addition of sodium thiosulphate; 0.05 mg. of morphine can then be detected. In determinations the procedures of Georges and Gascard (B., 1906, 779) and Mai and Rath (B., 1908, 828) give almost identical results. In toxicological examinations the extract obtained by the Stas-Otto method should be purified. Other methods are compared.

W. A. SILVESTER.

Composition of commercial plant lecithin. H. MATTHES and G. BRAUSE (Arch. Pharm., 1927, 265, 711—712).—Plant lecithins have a higher iodine value (90—100) than egg lecithin. The phosphorus : nitrogen ratio in egg lecithin is always less than 2.21 (% : %), whereas in plant lecithin it is greater than 2.21, *i.e.*, there is more, instead of less, phosphorus than corresponds with the theoretical ratio of 1 atom of each element. Five commercial plant lecithins showed moisture varying from 1.13 to 4.92%; phosphorus from 2.30 to 3.45%; nitrogen from 0.72 to 1.51%; phytosterol from 0.65 to 1.71%; iodine value from 90.0 to 100.2; acid value from 26 to 123.

W. A. SILVESTER.

Differentiation of animal and plant lecithins. H. MATTHES and G. BRAUSE (Arch. Pharm., 1927, 265, 708—710).—Commercial animal lecithins have been found (Brause, Diss., Königsberg i. Pr., 1926) to contain 0.22—3.38% of cholesterol, although Cohn (Z. öffentl. Chem., 1913, 59) has stated that this compound should be absent. Even material prepared in the laboratory contains 0.44%, and so it is evident that all lecithins of animal origin can be presumed to contain cholesterol. Plant lecithin is likewise found to be impure, and to contain, according to its commercial source, from 0.70 to 1.75% of phytosterol. This provides a means of determining whether any particular lecithin is of plant or animal origin, or is a mixture. The cholesterol, or phytosterol, is converted, through the digitonide, into the acetate, using the methods of Windaus and Fahrion (cf. Grün, "Analyse der Fette und Wachse," 1925, 1, 206, 265). Cholesteryl acetate from egg lecithin has, in the

crude state, m.p. 112.5—113.8°, and, after recrystallisation from absolute alcohol, m.p. 115°. Phytosteryl acetate similarly has m.p. 129—130° (crude) or 131.5—133.5° (recrystallised). A mixture of lecithins gives a steryl acetate the m.p. of which lies between these limits and is not constant on repeated crystallisation.

W. A. SILVESTER.

State of dispersion of [commercial] preparations of colloidal silver. H. BECHHOLD and E. HEYMANN (Arch. Pharm., 1927, 265, 669—675).—"Collargol" (the original preparation), four other German preparations, and three other samples, of French, Swiss, and Polish origin, respectively, have now been examined. All, except the Polish preparation, which contains 75%, have the normal proportions (about 25%) of protective colloid. Wide differences are, however, shown in the degree of dispersion of the silver. The samples were analysed before and after being centrifuged, to give the proportion of relatively large particles, filtered through an ultra-filter, to give the proportion of very small particles and ionic silver, and examined ultramicroscopically to give the mean size of the particles. They were also kept for 8 months and the precipitate, if any, was examined. "Collargol" contains 74% Ag; it gives a very small precipitate after 8 months; the mean size of the particles is 19 $\mu\mu$ (they are assumed to be cubes, and the length given is that of the edge); no separation takes place when a 1% solution is centrifuged, but a fair proportion passes the ultra-filter. The other samples contain particles the mean size of which varies from 19 $\mu\mu$ to 32 $\mu\mu$ but in some samples, although the mean size of the particles may be low, yet they are far from being uniform. The worst sample, *viz.*, the Polish preparation, contained particles of mean size 35 $\mu\mu$, and gave separations both on centrifuging and keeping, although so heavily loaded with protective colloid.

W. A. SILVESTER.

Rate of dissolution of essential principles during percolation. I. Press cake from bitter almonds. M. BRIDEL and (Mlle.) M. DESMAREST (J. Pharm. Chim., 1928, [viii], 7, 153—162, 201—207; cf. A., 1928, 201).—The press cake from bitter almonds containing both invertase and β -glucosidase furnished 1.73% of reducing sugar, 3.47% of sucrose, and 6.84% of amygdaloside on repeated extraction with boiling 70% alcohol, whilst percolation in the cold with one quarter of the amount of alcohol afforded 1.76% of reducing sugar, 3.65% of sucrose, and 7.35% of amygdaloside. The sucrose is very rapidly hydrolysed during maceration, but amygdaloside is more resistant, being decomposed to the extent of 10% in 10 days. A study of the rates of dissolution proved that all the above constituents are completely extracted from press cake by percolation with five times its weight of 70% alcohol, the sucrose being removed most rapidly and amygdaloside least rapidly. The sucrose is extracted in 5 hrs., and the amygdaloside in 12 hrs., this rapid extraction being the reason why the sugar is not hydrolysed by the invertase present. The sugars and glucoside together account for rather more than 50% of the total extracted solids. Pure amygdaloside is obtained in 3% yield by percolation with 70% alcohol, and the residual cake on maceration with water furnishes 1.4% of emulsin as a reddish

powder. The yield of emulsin from the press cake is only one half that from the corresponding quantity of almonds. A comparison of the enzyme with that from almonds shows that the latter is the more active with concentrated aqueous solutions of dextrose, but no difference is observed in methyl alcoholic solution. Emulsin from the press cake hydrolyses salicoside and lactose more rapidly than that from almonds, but no difference is found with galactose and sucrose. S. COFFEY.

Terpineol from terpin. A. ROBERT (Bull. Inst. Pin, 1927, 153—156, 177—182; Chem. Zentr., 1927, ii, 2295).—Dehydration of terpin hydrate by boiling with a little phosphoric or sulphuric acid yields, in the first runnings, dipentene, terpineols, α - and γ -terpinene, and cineole, but chiefly α -terpineol. Details are given for the detection of these substances.

A. A. ELDRIDGE.

Insecticides. FRANÇOIS and SEGUIN.—See XVI.

PATENTS.

Manufacture of ammonium sulphoichthyolate. D. H. ROWLAND, Assr. to AMER. ICHTHYOL OIL CO. (U.S.P. 1,664,376, 27.3.28. Appl., 4.2.27).—Petroleum is sulphonated and heated while being agitated. The heating is stopped, the agitation continued, and the mixture diluted with kerosene. A salting-out reagent is added, the whole agitated until frothing ceases, and the ammonium sulphoichthyolate isolated by adding ammonia solution until the mixture is alkaline and again diluting with kerosene. F. G. CLARKE.

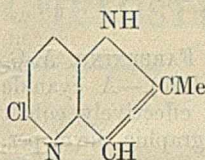
Diallylcyanamide. E. B. VLIET, Assr. to ABBOTT LABORATORIES (U.S.P. 1,659,793, 21.2.28. Appl., 12.4.26).—An allyl halide is heated with disodium cyanamide in aqueous-alcoholic solution to yield *diallylcyanamide*, b.p. 105—110°/18 mm. and 140—145°/90 mm., which is readily hydrolysed to diallylamine.

T. S. WHEELER.

Manufacture of salts of diethylaminoethyl *p*-aminobenzoate. F. HOFFMANN-LA ROCHE & Co. A.-G. (Swiss P. 118,336, 17.10.25, and addn. Swiss P. 120,447, 17.10.25).—An equimolecular mixture of β -diethylaminoethyl *p*-aminobenzoate and glutamic acid in aqueous solution is evaporated to dryness in a vacuum, or the ester hydrochloride and sodium glutamate may be used, the dry residue being extracted with alcohol and the alcohol removed in a vacuum. An alcoholic solution of *p*-aminobenzoic acid and β -diethylaminoethyl *p*-aminobenzoate deposits non-hygroscopic crystals of the salt, m.p. 104°. These salts have stronger anæsthetic power than the ester hydrochloride. C. HOLLINS.

Production of new pyridine derivatives. DEUTS. GOLD- & SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 259,982, 14.10.26. Austr., 17.10.25).—3-Pyridylhydrazine, or a derivative of 2- or 3-pyridylhydrazine, is condensed with aldehydes or ketones, keto-esters, etc., and the products in suitable cases are converted into pyrazolones or indoles. *Benzaldehyde-3-pyridylhydrazone*, m.p. 165—166°, *ethyl β -6-chloro-3-pyridylhydrazinocrotonate* (from ethyl acetoacetate), m.p. 123—124°, *6-iodo-3-pyridylhydrazine* (from 6-chloro-3-iodopyridine and hydrazine hydrate), m.p. 124°, *ethyl β -5-nitro-2-pyridylhydrazinocrotonate*, m.p. 93—95°, *5-nitro-2-pyridylhydrazones of acetone*, m.p. 168°, *propaldehyde*, m.p. 151°, and

benzaldehyde, m.p. 226—228°, are described. By heating the compound from 5-nitro-2-pyridylhydrazine and ethyl acetoacetate at 100° there is formed *1-(5-nitro-2-pyridyl)-3-methyl-5-pyrazolone*, m.p. 240—260°. Acetone 5-nitro-2-pyridylhydrazone, heated with zinc chloride at 200°, gives *5-chloro-2-methyl- β -*pyrindole** (an-extended formula), m.p. 208—210°; *5-chloro-3-methyl- β -*pyrindole**, m.p. 199—202°, is similarly obtained from the propaldehyde compound.



C. HOLLINS.

Manufacture of derivatives of 2-aminopyridine. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 265,167, 6.1.27. Ger., 27.1.26).—Alkylated 2-aminopyridines having more than two carbon atoms in the *N*-substituent group or groups are obtained either from 2-chloropyridine and an aliphatic amine, or from sodio-2-aminopyridine and an aliphatic halide. *2-Diethylaminopyridine*, b.p. 208—214°, *2-isopropylaminopyridine*, b.p. 105°/16 mm., *2-allylaminopyridine*, b.p. 124—129°/18 mm., *2-isoamylaminopyridine*, b.p. 135—140°/12 mm., and *2-cetylaminopyridine*, m.p. 65—66°, 215—225°/2 mm., are described. C. HOLLINS.

Manufacture of barbituric acid soporifics. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 285,598, 7.12.26).—Barbituric acid derivatives containing an acetylenic linking in one or both of the 5-substituent groups are good hypnotics producing no bad after-effects. *5-Propargyl-5-isopropylbarbituric acid*, m.p. 165°, is obtained from *5-isopropylbarbituric acid*, alcoholic sodium hydroxide, and propargyl bromide. Butinen- γ -ol (Hess and Munderloh, A., 1918, i, 291) gives with hydrogen bromide *γ -bromobutene*, from which *5- γ -methylpropargyl-5-isopropylbarbituric acid* is similarly obtained. By the action of the magnesium compound of *isopropenylacetylene* (*γ -methylenebutene*), the dehydration product from γ -methylbutinen- γ -ol (*loc. cit.*), on formaldehyde there is produced *α -isopropenylpropargyl alcohol*, $\text{CH}_2 : \text{CMe} : \text{C} : \text{C} \cdot \text{CH}_2 \cdot \text{OH}$, b.p. 68—69°/11 mm., which is converted by phosphorus tribromide into *α -isopropenylpropargyl bromide*, b.p. 65—67°/20 mm.; from this is prepared *5- α -isopropenylpropargyl-5-isopropylbarbituric acid*, m.p. 157°. *5-Propargyl-5-sym.-sec.-amylbarbituric acid*, m.p. 175—178°, is obtained from propargyl bromide and the barbituric acid prepared from *ethyl sym.-sec.-amylmalonate*, b.p. 114—116°/9 mm.

C. HOLLINS.

Manufacture of complex gold-nucleic acid compounds. SOC. CHEM. IND. IN BASLE (GES. F. CHEM. IND. IN BASEL) (B.P. 276,677, 25.8.27. Switz., 25.8.26).—Complex compounds (insoluble in acid), in which the gold cannot be detected by ordinary reagents, are formed by the interaction of nucleic acids or their soluble salts and gold compounds. B. FULLMAN.

Production of liquids of medicinal value. A. T. PORTER (U.S.P. 1,660,655, 28.2.28. Appl., 10.2.21. Renewed 25.5.27).—Hydrocarbon oils are atomised and partially burned with a limited supply of air, and the vapours obtained are condensed. T. S. WHEELER.

Vanillin and isovanillin (B.P. 285,156 and 285,551). **isoEugenol** (B.P. 271,819).—See III. **Rubber emulsions** (B.P. 286,527).—See XIV.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

PATENTS.

Photographic developers. I. G. FARBENIND. A.-G. (B.P. 280,525, 31.10.27. Ger., 13.11.26).—A cyanide of a heavy metal (mercury, or less effectively cobalt or cadmium) is added to any photographic developer. Mercuric cyanide (0.3 g. per litre) acts as a desensitiser, enabling development to be completed in bright yellow light; it also prevents fogging in prolonged development with weak developers. The cyanides give no precipitate with any of the usual developers. C. HOLLINS.

Mercurial printing surface. A. R. TRIST (U.S.P. 1,665,000, 3.4.28. Appl., 5.10.26. U.K., 5.11.25).—See B.P. 265,294; B., 1927, 429.

XXII.—EXPLOSIVES; MATCHES.

Season-cracking of cartridge cases. GRIMSTON.—See X.

PATENTS.

Nitration process. J. MARSHALL and F. H. BERGEIM, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,660,651, 28.2.28. Appl., 31.12.26).—Solid polyhydric alcohols dissolved in sulphuric acid are added to nitric acid to form the corresponding nitrates.

T. S. WHEELER.

XXIII.—SANITATION; WATER PURIFICATION.

Fumigation with hydrogen cyanide. P. BUTTENBERG and G. GAHRTZ (Z. Unters. Lebensm., 1927, 54, 376—387).—An account is given of the progress in the use of hydrogen cyanide for the destruction of pests. The advantages of using this agent in powder form (adsorbed on kieselguhr) over the tank process (in which sodium cyanide is thrown into vessels charged with dilute sulphuric acid) are discussed. Between 1923 and 1927 the former entirely displaced the tank process in the fumigation of ships in Hamburg harbour. A pungent-smelling substance is incorporated in the powder, viz., methyl bromoacetate in Zyklon B, and chloropicrin in Zyklon C. In the gaseous concentration used (0.10—3 vol.-%) hydrocyanic acid is neither inflammable nor explosive. These risks begin at a content of 15 vol.-%. Other methods of application are described. The uses mentioned include the destruction of rats and mice in ships, storehouses, and refrigerators, of insect pests in greenhouses, and of mites and insects in foodstuffs, the fumigation of furniture, cushions, fabrics, straw, asbestos, and books. Information is given concerning the concentration of the gas and time of exposure required for the destruction of pests in various materials. The retention of hydrogen cyanide by the articles exposed to it depends on such factors as the nature of the material, concentration of the gas, time of exposure, and temperature and humidity conditions during treatment. Vacuum treatment is usually effective in removing most of the retained hydrogen cyanide. For some products, such as coffee, the objection has been raised that the treatment spoils the flavour. Other commodities such as eggs for

hatching, photographic plates, etc. are rendered useless thereby. W. J. BOYD.

Determination of dissolved oxygen in water. V. G. ANDERSON and J. R. DICKSON (Chem. Eng. Min. Rev., 1927, 19, 467—468).—A modification of the Miller-Linossier method for determining dissolved oxygen in water is described. CHEMICAL ABSTRACTS.

Colorimetric determination of iron in water. P. LEHMANN and A. REUSS (Z. Unters. Lebensm., 1927, 54, 374—376).—100 c.c. of water are evaporated to dryness in a platinum basin, 5 c.c. of a 10% potassium sodium carbonate solution are added to the residue, and the contents of the basin again brought to dryness. After drying at 170—200°, the residue is heated to dull redness. When cool it is dissolved in 5 c.c. of 15% hydrochloric acid, and the solution transferred to a colourless glass cylinder with flat bottom. To the cooled clear solution, 1 c.c. of a bromide-bromate solution is added, the mixture kept for 5—10 min., and treated with 5 c.c. of a 7.5% ammonium thiocyanate solution. Simultaneously a blank test is carried out, to which a solution of iron ammonium alum of known strength is added from a burette until the colours of the two solutions are equal, both being brought to the same volume by addition of water. The amount of iron in the sample is calculated from the volume of standard iron solution added to the blank.

W. J. BOYD.

Boiler scale. WALDE.—See I.

PATENTS.

Production of disinfecting agents. A. BOEHRINGER (C. J. BOEHRINGER SOHN) (B.P. 272,543, 16.6.26. Ger., 28.2.26. Addn. to B.P. 253,918; B., 1927, 894).—Tartaric, citric, oxalic, or malic acid is used as disinfecting agent instead of, or in addition to, lactic acid, alkali being added until the p_H value of the solution is 3.0 or below. Heavy-metal salts, phenols, or colouring matters which are disinfectant or antiseptic may be added, and the material may be converted into solid or semi-solid form by the addition of gelatin, with or without glycerol. W. G. CAREY.

Disinfecting and insecticidal agent. H. GÜNZLER and O. NEUBERT, Assrs. to WINTHROP CHEM. CO., INC. (U.S.P. 1,663,883, 27.3.28. Appl., 31.3.27. Ger., 13.4.26).—A composition comprises a water-soluble salt of an aromatic aminosulphonic acid, e.g., sodium sulphanilate, and a water-soluble mercury salt, e.g., mercuric chloride. An inert wetting agent and an agglomerant miscible with water may also be present.

F. G. CLARKE.

Purification of water and removal and prevention of scale incrustations in boilers etc. G. S. NEELEY and G. WATKINS (B.P. 287,196, 12.11.26).—Feed water is subjected to the influence of an externally applied alternating electric current before being discharged from the inlet pipe, whilst an alternating current differing in voltage and phase from the former is passed through the shell and water content of the boiler.

J. S. G. THOMAS.

Treatment of sludge (B.P. 286,855).—See I.