

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MARCH 1, 1929.

I.—GENERAL; PLANT; MACHINERY.

Rapid method for approximate determination of sorption isothermals of vapours on charcoal. I. Principle of the method. A. J. ALLMAND and J. E. MANNING. II. **Simple retentivity test.** A. J. ALL-

MAND and L. J. BURRAGE (J.S.C.I., 1928, 47, 369—372 T, 372—376 T).—I. In order to evaluate the efficiency of a charcoal for sorbing vapours, a complete knowledge of its sorption isothermal is necessary. In practice, owing to experimental difficulties, this isotherm is never determined, but is substituted by a constant-pressure "breakdown" test, by tests made on adsorption from solution, by measurements of heat of sorption, etc. The authors have worked out a simple method of obtaining the sorption isotherm or, rather, a close approximation to it. The method depends on charging a column of charcoal with the vapour concerned at a certain pressure, and then following the rate at which this sorbed vapour is removed by passage of a stream of dry air. The tangents to these "rate" or "retentivity" curves give a measure of the vapour pressure above the charcoal at any instant in the desorption process, and from them the form of the isothermal can readily be deduced.

II. The apparatus and method used for obtaining the retentivity curves are fully described. Examples of the derived isothermals are given, together with a comparison between derived and directly measured true isothermals. The agreement in the latter case, though not perfect, is good.

Method for simultaneous measurement of plasticity and elasticity. E. C. BINGHAM and J. W. ROBERTSON (Kolloid-Z., 1929, 47, 1—5).—A method for measuring plasticity is described in which the substance is contained in a capillary tube, the ends of which are subject to a definite alternating pressure. The method has been applied to a 43% ammonium oxalate solution, and it is shown that the elastic deformation is directly proportional to the shearing power. E. S. HEDGES.

Drying of solids. I. T. K. SHERWOOD (Ind. Eng. Chem., 1929, 21, 12—16).—The drying of a solid may proceed either by evaporation at the surface or within the solid, and the resistance to internal liquid diffusion may be great or small. Very wet solids dry at a constant rate, the mechanism being similar to the evaporation of a liquid, but as drying proceeds conditions change. The case where evaporation is at the surface and internal diffusion-resistance is the limiting factor is considered in detail. The equations are similar to those for heat conduction in solids, and are plotted as a series of curves for different ratios of free liquid content to critical free liquid content. A graphical method is

described for obtaining the diffusion constant of Newton's equation from experimental results. Wood and clay are examples of this mechanism in drying if the slabs used are not too thin and if air is circulated mechanically over the surface. The drying of soap, on the other hand, shows a decrease in the diffusion constant as drying proceeds, connected with the shrinkage which occurs. This result is confirmed by determinations of moisture gradient in a slab. C. IRWIN.

Theory of fine grinding. IX. Connexion between the statistical diameter and statistical volume of irregularly-shaped particles of crushed sand. X. Connexion between the statistical diameter of crushed sand particles and their statistical surface. G. MARTIN [with E. A. BOWES]. XI. **Calculations relating to diameters, surfaces, and weights of homogeneous grades of crushed quartz sand.** G. MARTIN (Trans. Ceram. Soc., 1928, 27, 247—258, 259—284, 285—289; cf. B., 1927, 623).—IX. Experimental evidence is presented to show that, with irregularly-shaped particles of crushed sand, a statistical volume constant is given by V/d^3 , d being the statistical diameter of the particles, and V the corresponding statistical volume. The mean value for this constant, obtained with five samples of carefully graded, air-elutriated sand, was 0.277. The value was sensibly the same for particles varying in size from 116,520 to 2188 per g. Hence the average shape of sand particles is the same, whether they be large or small.

X. Similarly, the statistical surface constant is given by S/d^2 , where S is the statistical surface of the particles and d the diameter. For sand particles ranging from 0.00333 to 0.01089 cm. in diam., this constant is about 2.1. It is sensibly the same for large and small particles.

XI. The chief numerical data relating to 20 homogeneous grades of quartz particles ranging in size from 31,000,000 to 1240 per g. are given, and methods are presented for calculating the volumes, surfaces, and weights of powders of different size. F. SALT.

Optical method for measuring size of particles in suspensions. G. I. POKROVSKI (Kolloid-Z., 1929, 47, 55—58).—Sources of error in the gravimetric method of measuring the size of particles in suspensions are indicated, and an apparatus is described by means of which the determination can be made by a purely optical method. This is based on the measurement of the polarisation of the light scattered by the suspension. E. S. HEDGES.

Crystal growth in aqueous solutions. I. Theory. W. L. MCCABE (Ind. Eng. Chem., 1929, 21, 30—33).—If a known weight of seed crystals of known screen

analysis is suspended in a saturated solution and the solution slowly cooled without formation of fresh nuclei, a theoretical method can be developed for the calculation of the final weight and screen analysis of the crystals produced. It is assumed that no considerable degree of supersaturation occurs and that each crystal as it grows retains its geometrical form. The weight is calculated from solubility data and the result interpolated in an equation relating final and initial weights and particle sizes.

C. IRWIN.

Chemistry of mists and dusts. H. REMY (Chem.-Ztg., 1928, 52, 677—679, 698—699).—The difference in behaviour of sulphur trioxide made in a contact plant, which is absorbed only to a slight extent in water, but completely in sulphuric acid of 98.5% concentration, and of that obtained by passing air through fuming acid, which is less completely absorbed by sulphuric acid than by water, being merely rendered invisible by passing through the acid, is attributed to the fact that the trioxide obtained by the latter method is moist, whereas that made by the contact plant is dry. The former is a mist, of particle size about 10^{-4} cm.; the latter a colloidal dust, of particle size 10^{-5} cm. The extent to which a dry colloidal dust is absorbed by aqueous fluids is proportional to the vapour pressures of the fluids, and is influenced by their viscosities. In no case were the particles found to be electrically charged. The influence of particle size is discussed.

S. I. LEVY.

Simplified methods of potentiometric and conductometric analysis and their industrial application. T. CALLAN and S. HORROBIN (J.S.C.I., 1928, 47, 329—343 r).—The general principles of potentiometric and conductometric analysis are briefly explained. Simplified methods of potentiometric analysis involving the use of end-point cells are discussed and the preparation and use of such cells without the use of expensive electrical apparatus is described. A number of applications of such end-point cells are described, including their use in the determination of acidity, alkalinity, and halides in dyestuffs and coloured solutions, analysis by means of titanous chloride, and the determination of amines and phenolic compounds such as aniline, phenol, etc. by means of potassium bromate.

The various forms of apparatus for conductometric analysis are reviewed and an investigation into the use of the thermionic valve, as a rectifier to enable direct reading to be obtained on a D.C. instrument, is described, in the course of which the most suitable type of valve, the best conditions of anode voltage, grid bias, etc. were determined. A new simple apparatus in which alternating current from the lighting mains is transformed down, applied to the titration cell, rectified by a crystal "detector," and measured on a D.C. millivoltmeter is described. The conductometric method is particularly applied to titrations of sulphates with barium salts, of weak acids and bases, etc., for which the potentiometric method is unsuitable.

PATENTS.

Mechanical furnace. O. H. BUSE, Assr. to GRASSELLI CHEM. CO. (U.S.P. 1,693,366, 27.11.28. Appl., 13.10.25).—A two-stage mechanical furnace comprises

a furnace chamber having a charging pot above it provided with a stirrer and with an opening for gravity-discharge into the furnace, the opening being closed by a removable plug provided with a sealing groove situated between the plug and the wall of the opening when in the closed position.

L. A. COLES.

Rotary-hearth furnace. O. A. COLBY, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,695,883, 18.12.28. Appl., 14.7.27).—A rotary-hearth furnace comprises material-carrying trays pivotally mounted upon a hearth, and means for normally maintaining material being heated on the trays and for tilting the trays to discharge the material. J. S. G. THOMAS.

Tiltable furnace. G. SCHMITT, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,695,863, 18.12.28. Appl., 16.2.27. Ger., 20.4.26).—A cylindrical furnace casing is arranged between spaced, vertical members forming a base and having each a horizontal bearing surface and a vertical guiding surface. The casing is supported by means extending through its centre of gravity, and means are provided for engagement of the furnace with the guiding surfaces and for tilting the furnace.

J. S. G. THOMAS.

Pulverulent-fuel furnace. C. DUQUENNE (U.S.P. 1,693,597, 4.12.28. Appl., 10.10.25. Fr., 27.10.24).—An air-grate is placed between the combustion chamber and ash pit of the furnace; the grate is formed of a number of interfitting iron sections forming conduits for passage of air to a hollow wall and thence to the combustion chamber. The iron sections are covered with refractory material and supported by beams within the air conduits, the beams thus being exposed everywhere except on the top to the air currents and kept cool thereby.

B. M. VENABLES.

Furnace regulation. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of A. R. SMITH (B.P. 283,903, 19.1.28. U.S., 19.1.27).—A complicated system of electrical relays is provided by which the ratio of fuel to air is kept constant under varying load. In a boiler the fuel supply is regulated by the steam pressure, and if a number of boilers are working together the steam pressure in the common main regulates, through a master relay, the fuel supply to all the boilers, while individual relays keep the fuel-air ratio constant.

B. M. VENABLES.

Grinding, crushing, or like mills. A. STEINBRÜCKNER (B.P. 301,667, 10.1.28).—In a mill of the type where a grinding ring runs round rollers or where a beater member is rotated inside a stationary grinding ring, an annular space is left between the outside of the ring and the casing in which is a ring-shaped bucket conveyor driven either separately from or by the ring for the purpose of lifting coarse material which has settled in the lower part of the casing up to the top, whence it falls through curved side passages back into the grinding zone. A stream of air is also injected tangentially into the lower part of the annular space and exhausted from the upper part, carrying with it the finely-ground material. A classification device is inserted in the air-exhaust pipe.

B. M. VENABLES.

Crushing, grinding, and refining machine. S. BRAMLEY-MOORE (U.S.P. 1,692,884, 27.11.28. Appl.,

6.4.28).—A cylindrical casing is provided with an interior, fixed grinding surface, and rotating grinding elements are carried by levers which are driven by a wheel within the apparatus. A disc on another co-axial shaft rotates with, and has angular adjustment relative to, the wheel. The disc operates on the levers to vary the pressure between the grinding elements and surface. B. M. VENABLES.

Rotary dryer. F. A. JONES (U.S.P. 1,694,708, 11.12.28. Appl., 24.3.28).—An inclined rotary kiln is surrounded by longitudinal pockets, the material under treatment being charged into the upper ends, and discharged from the lower ends of these. L. A. COLES.

Liquid filter. A. WUEST (U.S.P. 1,693,741, 4.12.28. Appl., 23.5.27).—A cylindrical filtering unit of which the wall comprises, in order outwards, the filter medium, the support for the medium or inner screen, and an outer screen formed from two troughs, of semi-circular section, interlocked at their edges. B. M. VENABLES.

Refrigerating apparatus. P. W. TAYLOR (B.P. 303,569, 8.10.27).

Refrigerating plants. C. H. COOK, and SERVEL, LTD. (B.P. 303,425, 3.9.27).

Refrigeration apparatus. SILICA GEL CORP., Assees. of E. B. MILLER (B.P. 292,938, 20.12.27. U.S., 27.6.27).

Freezing mixture (B.P. 298,623).—See VII. **Centrifugal apparatus for recovery of vapours** (B.P. 298,226).—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Constitution and classification of coal. A. C. FIELDNER (Fuel, 1929, 8, 36–45).—Systems of coal classification are discussed in the light of our present knowledge of its constitution, and the programme of the American Engineering Standards Sectional Committee is outlined. It is concluded that coal should be classed primarily on the basis of its intrinsic chemical and physical properties, and that use classification should be secondary to and as far as possible correlated with scientific classification. Scientific classification depends on two primary factors, the composition and type of the original coal-forming vegetation and the degree of metamorphism or coalification of the vegetable residue. The criteria to be considered for classifying under these two general factors are proximate and ultimate analyses, calorific values, microscopical examination, extraction with solvents, reaction with reagents, and destructive distillation. A. B. MANNING.

Vitrain. M. M. EVANS, L. SLATER, and R. V. WHEELER (Fuel, 1929, 8, 30–36).—Various types of plant structure, in particular parenchyma, periderm, and xylem, have been observed in British vitrains by microscopical examination of polished and etched surfaces in incident light. From a study of its micro-structure, vitrain appears to be a simple substance (as distinct from the aggregates, clarain and durain) consisting of ulmin compounds which have infilled plant cells and partly, if not entirely, replaced the materials of the cell walls. Although the evidence regarding the

species of plants to which the structures relate is indirect, it appears probable that certain vitrains at least represent the residues of *Sigillaria* and of *Lepidophlois*. A close similarity between the structures of vitrain and fusain has sometimes been observed when the two are in close association. Some details are given of the method used in preparing specimens for microscopical examination. A. B. MANNING.

Relationship between the sp. gr. and ash contents of the coals of Korea and Bokaro : coals as colloid systems. L. L. FERMOR (Rec. Geol. Survey of India, June, 1928; Fuel, 1929, 8, 16–29).—A study of coals from Korea and from Bokaro has established the empirical relationship, $a = 100(g - k)$, between a , the ash content, g , the sp. gr., and k , the sp. gr. of the pure coal substance, which is constant for each field (1.28 and 1.26, respectively). The relationship holds up to coals containing 50% of ash. The higher sp. gr. of a few coals which form exceptions to the rule is due to the high iron content of the ash. The sp. gr. of the coals which conform to the linear law, however, are higher than values calculated on the assumption of simple admixture of pure coal and pure shale. This is explained on the supposition that the coals are colloidal systems, in which the ash forms the disperse phase, and the vitrain the dispersion medium. Evidence is adduced also of the colloidal dispersion of the moisture in the vitrain. The bearing of this point of view on the classification and the treatment of coal is discussed. A. B. MANNING.

Reactions and gas-flow in [gas] generators as a basis for their constructional design. E. TERRES (Gas- u. Wasserfach, 1928, 71, 1205–1213).—The percentage of hydrogen in the gas forms a better criterion of the efficiency of the generator operation than the percentage of carbon dioxide. Determinations of the gas composition at points within the fuel bed show that the various reactions occurring therein take place in zones, not disposed horizontally, but extending to the surface of the fuel bed. The gas of the central zone is of good composition, but that in the outer zones contains successively greater percentages of carbon dioxide. The position and breadth of the zones contributing carbon dioxide to the gas depend on the cooling of the fuel bed near the outer surface of the generator. For each size of fuel there is an optimum depth of fuel bed, further improvement of the gas being then possible only by increasing the diameter of the generator, assuming, at the same time, that it is possible to maintain a uniform distribution of air over the cross-section. The reactivity and the sieve analysis of the fuel are of importance in judging its suitability for use in the generator. The amount of dust in the gas and the resistance of the fuel bed to the air are determined principally by the proportion of fuel less than 5 mm. size. A short description of the generator used in the experiments is appended to the paper. A. B. MANNING.

A source of error in the carbon balance [of gas-producer tests]. WEHRMANN (Gas- u. Wasserfach, 1928, 71, 1253).—Errors may arise in drawing up a carbon balance for a gas producer from the absorption

of carbon dioxide in the gas scrubber, the aspirator, and in the gas burette. Analyses of the gas taken directly from the producer outlet have been compared with those of corresponding samples taken after the gas has passed the scrubber, the former determinations being made gravimetrically and the latter volumetrically. The decrease in the percentage of carbon dioxide due to absorption varied from 0.4 to 0.9, the average being 0.65.

A. B. MANNING.

Chemical processes in the gasification of brown coal. H. KNOPF (Gas- u. Wasserfach, 1928, 71, 1218—1221, 1242—1245, 1259—1263).—Measurements of temperature and of gas composition have been made at points in the axis of the fuel bed of a Heller gas generator charged with brown coal. Although the proportions of hydrogen, carbon monoxide and dioxide, and steam in the gas correspond in general with the water-gas equilibrium ($\text{CO} + \text{H}_2\text{O} \rightleftharpoons \text{CO}_2 + \text{H}_2$) a detailed study of the results leads to the conclusion that the water-gas reaction itself plays little part in the generator reactions. The measurements of temperature correspond with that of the solid fuel; that of the gas must always be higher except at the base of the generator. The gas temperatures have been calculated by drawing up heat balances for different zones of the fuel bed. The differences between the gas and fuel temperatures are greater the drier is the fuel. The relation between these temperature differences, the gas composition, the depth of the fuel bed, and other factors is discussed. Except when wet fuel is used, an increase in the depth of the fuel bed beyond a certain limit has no influence on the gas composition.

A. B. MANNING.

Water-gas from bituminous coal. J. GWOSDZ (Gas- u. Wasserfach, 1928, 71, 1233—1239, 1253—1259).—Recent practice in the operation of water-gas producers with bituminous coal, particularly in America, and its probable influence on the development of the German gas industry are critically discussed.

A. B. MANNING.

Cyclic process for ammonia recovery from coke-oven gases. V. I. DENISOV (J. Chem. Ind. Moscow, 1928, 5, 13—18).—The cyclic process, and losses of ammonia occasioned therein, are discussed. Treatment of the stock water with lime is unremunerative. Corrosion of iron apparatus by the ammonia solution does not take place.

CHEMICAL ABSTRACTS.

Cyclic ammonia process. M. D. LIEPIATSKIK (J. Chem. Ind. Moscow, 1928, 5, 480—483).—In the lime water process the ammonia recovered represents 0.22% of the coal used; in the cyclic process the yield is 0.13—0.22%. The losses of ammonia are discussed, and desirable modifications (e.g., distillation of crude ammonia water by external heat) are suggested. The ammonia obtained by the cyclic process is contaminated with carbon dioxide and hydrogen sulphide.

CHEMICAL ABSTRACTS.

Equilibrium combustion of a mixture of carbon monoxide and hydrogen. C. C. MINTER (J.S.C.I., 1929, 48, 35—38 T).—Examination of a set of analytical data obtained by U.S. Bureau of Mines shows that when carbon monoxide and hydrogen are burnt simultaneously in the same mixture at high temperatures (1200—1500°),

the two gases are not oxidised independently. Graphs connecting the concentration of carbon monoxide and hydrogen with time show that the reactions are both unimolecular, with the same velocity coefficient. The ratio of the fractional changes in concentration of the two gases in the same interval is found to be equal to unity. The two gases thus disappear at the same rate, the rate being proportional to the first power of the concentration of the reacting gases. This means that the rate of oxidation is in both cases independent of the oxygen concentration. This anomalous result is explained by assuming that the water-gas reaction is operating, and, under such conditions, the rate of oxidation would be dependent on the rate at which the water-gas equilibrium is adjusted to changes in concentration of the carbon monoxide and hydrogen.

Production of gaseous, liquid, and solid hydrocarbons from methane. I. Thermal decomposition of methane. H. M. STANLEY and A. W. NASH (J.S.C.I., 1929, 48, 1—8 T).—The thermal decomposition of methane has been examined from the point of view of the production of higher hydrocarbons. By passing a stream of methane through silica tubes heated to 1000—1200°, liquid, solid, and higher gaseous hydrocarbons were produced. The liquid and solid products were chiefly of aromatic nature. A special study was made of the gaseous products using low-temperature condensation methods of analysis, and among these were identified, besides hydrogen, acetylene, ethylene, and small quantities of ethane, higher olefines, and aromatic hydrocarbons. For each temperature there is a heating period which gives optimum yields of higher products the duration of which is decreased by rise in temperature. The best yields of higher hydrocarbons were obtained at a temperature of 1150° and a heating duration of 0.6 sec. Under these conditions the yield of a light oil and tar was 11.0% calculated on the methane used, while the quantities of acetylene and ethylene in the reaction gas corresponded to a total conversion of methane into these gases of 8.8%. Thus the total conversion of methane into higher hydrocarbons was nearly 20% of the theoretical, while only about 6% of methane had been converted into its elements. The yield of light oil was 4.8%, or 0.2 gal. per 1000 cub. ft. of methane. The use of comparatively long heating periods tended to cause methane to decompose into its elements almost exclusively, this tendency being greatly increased by the use of large heating surfaces and of catalytically active materials such as iron and nickel. The mechanism of the process is discussed in the light of the theory of nascent residues.

Chemical study of low-temperature tar. G. T. MORGAN, D. D. PRATT, and J. ROSS (J.S.C.I., 1929, 48, 29—34 T).—A sample of low-temperature tar from coal of the 6 ft. seam of Kinneil (Linlithgow) has been submitted to a solvent treatment at temperatures not exceeding 120°. After successive extractions with aqueous alkali and dilute acid, the neutral oils were distilled up to this temperature and the residual oil was treated successively with acetone and petroleum (b.p. 40—60°) in order to remove respectively waxes

and solid aromatic hydrocarbons. During this fractional separation neutral materials were isolated having the properties of resins. When rendered slightly acid the alkaline extract of the tar yielded crystallisable phenols, a resinous variety of phenols termed resinols, and, in addition, a further quantity of the neutral resins. Acidification with mineral acid brought about a separation of carboxylic acids mixed with acidic materials of a resinous character, to which the term resinic acids is applied. The true phenols (crystallisable phenols and resinols) were separated from phenolate-soluble materials by one or other of two alternative extractions, aqueous alkalis with organic solvents or caustic soda-brine reagent. The true phenols were then divided into crystallisable phenols, tar acids, and resinols by systematic employment of ether and light petroleum. When rendered alkaline the acidic extract of the tar furnished crystallisable and resinous bases, the latter being termed resinamines. Comparative experiments were then made on distilled and undistilled Kinnell tar. Distillation which decreased the amounts of alkali-extractable material, neutral oils, waxes, and higher aromatic hydrocarbons, led, moreover, to the formation of 31% of pitch as contrasted with about 5–6% of insoluble residue left after treatment by solvents. By the foregoing method of extraction four groups of resinous materials have been separated from low-temperature tar: neutral resins, phenolic resins or resinols, acidic resins or resinic acids, and basic resins or resinamines. In suitable organic media these resinous materials can be employed as stains and varnishes. By avoidance of high temperature, other decomposable constituents of the tar such as waxes and higher aromatics have been obtained in appreciably higher yields.

Causes of and means of reducing the water content of tar. K. BUNTE (Gas- u. Wasserfach, 1929, 72, 1–3).—The separation of water from tar depends primarily on the sp. gr. and viscosity of the latter. With viscous tars a better separation is usually obtained by warming the mixture and allowing it to settle. Increase in temperature, however, diminishes the difference in sp. gr. between water and tar, and may therefore be disadvantageous with a light tar. The sp. gr. of the tar determines the relative positions of the tar and liquor overflows from the settling tanks. For good separation these tanks should be deep and roomy, and the tar condensing at the various points of the cooling plant should be well mixed before arriving at them. A high water content and emulsion formation may be further due to surface tension effects, the presence of free carbon, the partial miscibility of water and phenols in the tar, the presence of ammonium phenoxides, and colloidal phenomena.

W. T. K. BRAUNHOLTZ.

Carboxylic acids in peat tar. G. STADNIKOV and W. SABAWIN (Brennstoff-Chem., 1929, 10, 1).—The oil which passes over when peat tar is distilled with steam contains saturated and unsaturated fatty acids (about 0.2–0.3% of the tar) having six to nine carbon atoms. It appears probable that these acids are present in the original peat.

W. T. K. BRAUNHOLTZ.

Biochemical studies on pityrol. I. Introduction. S. KOMATSU. **II. Distillation of rice bran.** S. FUJITA. **III. Neutral constituents of pityrol.** S. SAKAMI. **IV. Acidic constituents.** B. MASUMOTO. **V. Basic constituents.** S. SUZUKI (Mem. Coll. Sci. Kyoto, 1928, 11, 481–495, 497–503, 505–515, 517–519, 521–532).—I. Bran from “Shinrinki” rice, when dry-distilled from a Fischer aluminium retort at 200–450°, yields 28% of coke, 36% of aqueous liquor, 12% of gas, and 24% of the tar known as pityrol. The volatile (13%) and non-volatile (87%) portions of pityrol may be separated into neutral, acidic, and basic constituents. Neutral substances form the bulk of the tar, those from the volatile portion consisting mainly of the unsaturated hydrocarbons C_nH_{2n} (C_9 – C_{14}) and C_nH_{2n-2} (C_8 – C_{14}). The non-volatile neutral portion contains polymethylene hydrocarbons of high mol. wt. and some aromatic hydrocarbons. A fraction, b.p. above 250°/10 mm., produced cancer in a rabbit's ear by repeated application. As has been previously noted with other tars, pityrol itself has a pathological effect differing from that of its constituents. The basic portion of pityrol, though small in quantity, is the cause of the offensive odour of the tar; it contains methyl derivatives of pyridine, also quinoline and aniline. The volatile acidic portion of the tar probably contains $C_{10}H_{15} \cdot OMe$, and has a highly irritating effect on the rabbit ear. Rice bran yields more tar than peat or wood under similar conditions, owing to a greater content of protein and fats. De-fatted bran yields less tar. The unsaturated and saturated hydrocarbons in pityrol are derived from the fats in the bran (chiefly palmitic and oleic acids) by thermal decomposition and polymerisation. The saturated hydrocarbons found are those most thermally stable: C_9H_{20} , $C_{10}H_{22}$, $C_{15}H_{32}$, $C_{30}H_{62}$. The acidic compounds in pityrol, mainly furan derivatives, are formed from the pentosans, cellulose, and soluble carbohydrates of the bran, and the proteins in the bran yield the basic constituents of the tar. Pyrrole, though absent from the tar, is probably an intermediate compound in the formation of the pyridine bases.

II. Two samples of rice bran, A and B, the rice oil having been removed from the latter, had the following percentage compositions, respectively: water 14.7, 14.1; ash 9.3, 12.0; crude fat 22.4, 0.7; protein 12.9, 16.5; cellulose 11.4, 14.6; pentosans 8.7, 11.1; reducing sugars 1.3, 1.5; sucrose 10.6, 13.6. Dry samples of A and B were distilled from a Fischer retort. The composition of the coke and aqueous liquor was similar in both cases, but the gas from A contained more olefines and less carbon monoxide than that from B, whilst the preponderance of neutral constituents in the tar from A was less marked in the tar from B.

III. Successive treatment of the volatile neutral portion of pityrol with concentrated and fuming sulphuric acid separated the saturated hydrocarbons (each member of the series from octane to pentadecane being later isolated) from the unsaturated hydrocarbons forming the bulk of the sample. The treatment with sulphuric acid caused polymerisation of these latter, and also formation of acid esters and alcohols. By

identification of these compounds it was deduced that C_9H_{16} , $C_{10}H_{18}$, $C_{12}H_{20}$, and $C_{14}H_{24}$ are the chief constituents of this part of pityrol. The non-volatile neutral portion of pityrol contains polymethylene hydrocarbons similar to those found in Russian and Japanese petroleum and low-temperature coal tars.

IV. The acidic portion of pityrol, a dark brown oil having a phenolic odour, was fractionated, and the middle fraction methylated and separated into 18 fractions under reduced pressure. The presence of at least one methoxy-compound, $C_{10}H_{15} \cdot OMe$, b.p. $70-74.5^\circ/7$ mm., d_4^{25} 0.6922, n_D^{25} 1.4698, was deduced from the analytical and physical data.

V. Both the soluble and insoluble portions of the basic constituents of pityrol were found to contain primary, secondary, and tertiary bases, but no trace of pyrrole. By fractional distillation, and crystallisation of their picrates, hydrochlorides, or double salts with mercuric chloride, the following bases were identified: 2-methyl-, 3-methyl-, 2:6-dimethyl-, 2:4-dimethyl-, 2:4:6-trimethyl-pyridine, aniline, and quinoline; also 3-dimethylpyridines (?), the double salts of which, m.p. $163-164^\circ$, 110° , and 112° , respectively, gave analyses agreeing with $C_7H_9N \cdot HCl \cdot 2HgCl_2$; a pyridine derivative (?) (double salt, $C_5H_5N \cdot HCl \cdot 2HgCl_2$, m.p. 116°); and a tetramethylpyridine (?) (picrate, m.p. 106°). From the mother-liquor after the separation of quinoline picrate, crystals, m.p. 180° , were obtained, which await further investigation. B. W. ANDERSON.

ψ -Cumene derivatives in crude wood spirit. B. HOLMBERG (Svensk Kem. Tidskr., 1928, 40, 304-315).—The substance of unknown composition previously described (A., 1923, i, 226) as obtained during the purification of wood spirit distilled without previous neutralisation from pyroligneous acid derived from a mixture of pine wood (80%) and spruce wood (20%) has now been identified as 5- ψ -cumylacetone. On reduction with zinc and hydrochloric acid it yielded 5-propyl- ψ -cumene, b.p. $226-228^\circ$, d_4^{20} 0.887, n_D^{20} 1.5095. With sodium and alcohol 5- ψ -cumyl-sec.-propyl alcohol, m.p. $74-75^\circ$, was formed. Bromination in acetic acid gave 3:6-dibromo-5- ψ -cumyl- α -dibromoacetone, m.p. $116-117^\circ$. An ethereal solution yielded with magnesium methyl iodide 5- ψ -cumyl-tert.-butyl alcohol, m.p. $45-47^\circ$. On oxidation with sodium hypobromite durylic acid (ψ -cumene-5-carboxylic acid) resulted. Alkalis decomposed it slowly giving resinous products, together with small amounts of durylic acid. In addition to the first-named compound a small amount of 6- ψ -cumenol has now been identified among the products of the above-mentioned distillation; this substance has not previously been reported as occurring in wood spirit. 5- ψ -Cumylacetic acid, m.p. $117-120^\circ$ (ethyl ester, b.p. $141-142^\circ/11$ mm., d_4^{20} 1.006, n_D^{20} 1.507), is also described. H. F. HARWOOD.

Transformation of the fatty acids during geological epochs. I. G. L. STADNIKOV and A. E. WEIZMANN (J. Russ. Phys. Chem. Soc., 1928, 60, 1123-1131). II. G. L. STADNIKOV and Z. S. VOZSCHINSKAJA (Ibid., 1133-1136).—I. In order to elucidate the mechanism of formation of hydrocarbon oils in the earth's crust from organic matter, the deeper layers of

peat-bog deposits, which represent transition stages between peat and young bituminous coals, were investigated. The primary tars from these deposits were found to contain large quantities of highly polymerised and dehydrated acids of the fatty series. The solids themselves (from the Sucho-Kujatsk district, Siberia) were powdered, and extracted successively with (a) benzene, (b) chloroform, (c) ethyl-alcoholic potassium hydroxide, (d) amyl-alcoholic potassium hydroxide, (e) ethyl alcohol, (f) water. Analysis of the extracts showed them to contain the following: (a) (1.6% of total) a brown solid, similar in appearance to peat bitumen, which on esterification and fractionation *in vacuo* was found to be a mixture of fatty and unsaturated acids of the $C_{12}-C_{22}$ series; (b) (0.46%) a gummy tar (not analysed); (c) only salts of acids, both fatty and unsaturated, ranging from octoic to C_{16} acids; (d) mostly fatty acids, including octoic, boiling range $100-215^\circ$, and also some unsaturated acids; (e) a mixture of acids, boiling range $120-280^\circ$, including the butyric, valeric, and hexoic, and higher fatty acids up to C_{11} ; (f) mostly valeric acid, together with an unsaturated acid of high mol. wt. The results thus obtained confirmed the analysis of the primary tars.

II. The samples from peat-bogs of the Matagausk and Sucho-Kujatsk districts were similarly examined. Extract (b) was not analysed, as it contained only 0.22% of black, viscous tar. Extract (a) (1.8%) was a brown solid, which, on hydrolysis, yielded a mixture of acids m.p. 30° (approx.), containing 72.79% C and 10.38% H, and having saponif. value 186.3, iodine value 11.6, approx. formula $C_{13}H_{26}O_2$. Extract (c) gave also a mixture of acids, both fatty and unsaturated, of the series $C_{10}-C_{18}$. Extract (d) on careful fractionation *in vacuo* gave butyric, valeric, and hexoic acids. Extract (e) yielded a solid which approximated to an unsaturated C_8 acid, whilst (f) contained acids, both fatty and unsaturated, of the C_6-C_8 series. The acids, apparently, were all monobasic, and included, in all probability, hydroxy-acids, which formed lactones.

M. ZVEGINTZOV.

Oxidation of motor fuels. E. BERL, K. HEISE, and K. WINNACKER (Z. physikal. Chem., 1928, 139, 453-481).—The self-ignition temperatures of mixtures of the inflammable vapours of motor fuels with air at normal pressures and at pressures up to 20 atm. have been determined together with the influence of the addition of anti-knocking substances on these temperatures. In the case of mixtures of air with pentane, *n*-hexane, and cyclohexane increased pressure has only a slight depressing effect on the temperature of self-ignition for a mixture of constant composition. With ether vapour and air the effect is somewhat greater, but with benzene-air and alcohol vapour-air mixtures ignition does not occur at 800° under the pressures employed. cycloHexene at 60° and 20 atm. is polymerised by the oxygen in the admixed air. In general, substances which cause knocking most readily are those which are the most easily oxidised. The compression strength increases according to the series, hexane, cyclohexane, cyclohexene, and benzene. The addition of lead tetraethyl or iron carbonyl raises the ignition temperatures to approximately the same

extent. The oxidation of the hydrocarbons in the presence of insufficient oxygen has also been investigated together with the effect of the addition of lead tetraethyl, iron pentacarbonyl, and finely-divided lead and iron. In contradistinction to the far-reaching changes in hexane brought about by oxidation, the addition of lead tetraethyl or iron carbonyl is accompanied by only a small alteration in the b.p. curve of pure hexane; the anti-knocks prove to be powerful negative catalysts for the conveyance of oxygen, and act by decomposition into the corresponding finely-divided metal. The effect is simulated to a smaller extent by lead and iron pulverised in a luminous arc. Hydrocarbons rich in hydrogen decompose into hydrogen and an unsaturated residue both of which are oxidised forming water, hydrogen peroxide, and organic peroxides. The finely-divided metal inhibits this formation of peroxides before the succeeding intramolecular oxidation can take place.

L. S. THEOBALD.

Oils for turbines. M. VAN RYSELBERGE (Bull. Féd. Ind. Chim. Belg., 1928, 7, 445—470).—The characteristics of lubricating oils for steam turbines, their requirements, manufacture, method of use, and value of laboratory tests as a means of selection of the most suitable are reviewed. A consideration of the usual physical tests—density, flash points, and viscosity—shows that these are no aid to the selection, and three further specific characteristics are investigated. Twelve oils have been examined with respect to their lubricating power on a Dittmar machine, in which a shaft is rotated in a bearing by means of an electric motor. The bearing is lubricated with each oil in turn. The motor is started, and stopped when the shaft reaches 1400 r.p.m., and the time is recorded for the shaft to come to rest. The determination is carried out at four temperatures. Curves drawn, however, are almost superposable, and from this and other considerations the test, as a selective one, is rejected as unsatisfactory. The rise in temperature of the oil or of the bearing on the same machine after running for 60 min. seemed to be promising, but was discarded owing to the impossibility of duplicating results. A final study was made of the emulsive properties of lubricating oils. The steam-emulsifying method of the I.P.T. is considered as accurate, but insufficiently selective for turbine oils. The method adopted consisted in agitating for 2 min. 40 c.c. of oil and 20 c.c. of distilled water in a 100 c.c. Pyrex cylinder by means of a current of hydrogen, and recording each minute the volume of water separated. This is done at 20°, 50°, and 80°, and the oil with the best demulsibility at all temperatures is considered to be the one likely to give best results for steam-turbine lubrication. With the oils examined, origin, viscosity, and density (with the exception of two oils, $d > 0.900$) had no relation to the classification of oils by demulsibility. Certain anomalies were noted in which the rates of separation were greater at the lower temperatures than at the higher, and this was attributed to influences of the method and degree of refining.

H. S. GARLICK.

Water-miscible mineral oil preparations. R. HART (Ind. Eng. Chem., 1929, 21, 85—90).—The manufacture of clear water-miscible or "soluble" oils is

primarily a problem of miscibility, and free oleic acid is essential to a uniform product. Miscibility curves for several emulsifiers, mineral oil, and oleic acid are given, by means of which uniform and non-uniform mixtures can be traced. Free oleic acid decreases the stability of the emulsion and may even prevent it. A number of terms are defined which simplify investigation of soluble oils, and make for greater convenience in comparing them. The kind and quantity of mineral oil have practically no effect on the quantity of oleic acid that the mixture will tolerate and still give good emulsions. This factor, however, varies with the kind of emulsifier, and even with the same emulsifier provided it is subject to adjustments. Alcohol has the following effects: (a) acts as liquefier for the soap; (b) at first decreases, then increases, the quantity of free oleic acid required for a homogeneous mixture; and (c) exerts no direct effect on the emulsion. The addition of alkali to a soluble oil containing acid sulphonated oil as the emulsifier yields the following results: (a) in the absence of alcohol, it at first decreases then increases the quantity of free oleic acid required for a homogeneous product; (b) in the presence of sufficient alcohol, the more alkali is present the less oleic acid is required to clear; and (c) the more neutralised is the sulphonated oil the better it functions as emulsifier, the completely neutralised oil being the best in this respect. Methods of testing raw materials and the stability of soluble oils based on the above results are developed.

H. S. GARLICK.

Metallic colloids and knock suppression. H. L. OLIN and W. J. JEBENS (Ind. Eng. Chem., 1929, 21, 43; cf. B., 1927, 66).—Further experiments showed that nickel sols prepared by heating nickel carbonyl in boiling hexane increased the highest useful compression ratio (H.U.C.R.) of gasoline to nearly the same extent as undecomposed nickel carbonyl, but that by increasing the temperature of decomposition to 85° the improvement becomes negligible. Lead tetraethyl behaves similarly, the lead sol produced at 250° giving an increase in H.U.C.R. of 7.5%, but that prepared at 300° shows no increase.

H. S. GARLICK.

The double linkings in vaseline, especially in that used for smokeless powders. R. POGGI (Giorn. Chim. Ind. Appl., 1928, 10, 601—605).—Application to a number of unsaturated compounds, such as amylene, allyl alcohol, oleic acid, cinnamic acid, anethole, dihydrocarvone, and terpineol, of the methods of Hanus (B., 1901, 1246) and Rosenmund and Kuhnhehn (B., 1924, 23; 1925, 214) for determining the iodine or bromine values of fats and oils shows that the latter method gives results in the better accordance with the calculated values. Tests made with vaselines from various sources show, in general, moderately good agreement between the two methods, but for Hellfrisch's vaseline and Kahlbaum's white vaseline the Hanus method gives, respectively, 82.2 and 85.2 mg. of bromine per 1 g., whereas the Rosenmund method gives 0.7 and 8.1. Certain qualities of commercial vaseline are found to contain very small proportions of compounds with double linkings.

T. H. POPE.

Determination of sulphur in benzol. W. B. DAVIDSON (Gas J., 1929, 185, 95–96).—The benzol is diluted with four times its volume of sulphur-free methylated spirit, and 10 c.c. of this mixture are burned in a special burner, during $1\frac{1}{2}$ –2 hrs. Air for combustion is purified by passing through a scrubber moistened with caustic soda solution, and the combustion products are drawn up a chimney of Pyrex glass, from which they pass to a perforated bulb which dips below the surface of a solution of neutral hydrogen peroxide. The neck of the flask, by which the gases escape, is filled with broken glass moistened with the same liquid. The contents of flask and scrubber are titrated with 0.1N-sodium carbonate to determine sulphuric acid. The method can be applied to paraffin or lubricating oils, or to petrol by suitable modification.

R. H. GRIFFITH.

Synthetic ammonia plant. PALLEMAERTS.—See VII. **Protection of underground pipes.** SLATER.—See X. **Colour of lamp blacks.** HOCK.—See XIII.

PATENTS.

Coke ovens. KOPPERS Co., Assees. of J. BECKER and J. VAN ACKEREN (B.P. 278,012, 26.9.27. U.S., 27.9.26).—Steam jets are provided in the offtakes from high-chambered regenerative coke ovens so that a forced draught can be applied therein while charging the ovens. By this means all volatile matter evolved during the charging may be collected, and at the same time a regulated pressure above atmospheric may be maintained in the whole gas-collecting main system. Any of the ovens may then be charged without subjecting the remainder to the danger of damage caused by drawing air through the floors of the coking chambers. In the arrangement preferred, each oven is provided with an offtake on either side of a central charging opening.

A. B. MANNING.

Carbonising and gasifying pulverised coal and treating ores. TRENT PROCESS CORP., Assees. of W. E. TRENT (B.P. 277,660, 12.9.27. U.S., 14.9.26).—Finely-pulverised coal is heated to a temperature just below the fusion point, maintained at that temperature until practically no more volatile matter is evolved, and then heated to a higher temperature in order to complete carbonisation. Under these conditions no agglomeration or fusion of the material occurs. The coal, reduced to a fineness of at least 100-mesh, passes down narrow vertical tubes within a retort, up through the space surrounding the tubes, and down through an annular chamber to the outlet. The volatile products of carbonisation travel with the material until it passes into the annular chamber; the vapours and gases then pass into an upper compartment and thence to suitable outlets. The retort is heated by a fire box at the base, the hot gases from which pass up through tubes arranged concentrically within those down which the coal travels. By controlling the rate of passage of the coal and the temperature of the fire box the rate of heating is arranged to fulfil the desired conditions. The process may be used for heating ores or other materials.

A. B. MANNING.

Gas producers. W. M. CROSS (B.P. 289,491, 28.4.28. U.S., 28.4.27).—Pulverised fuel, together with a mixture

of air and steam in such proportions and at a sufficiently high initial temperature to give on interaction with the fuel producer gas of any desired calorific value, is injected tangentially into the upper part of a vertical producer. The air and steam are preheated by means of hot waste gases or by the producer gas itself, and are then further heated in a superheater, which is preferably of the oil-fired type. The temperature of the air-steam mixture is automatically controlled. A baffle above the gas outlet in the lower part of the producer separates impurities from the gas, which then passes through a dust separator to the holder.

A. B. MANNING.

Gas purifiers, scrubbers, and the like. R. W. BROADHEAD, and BROADHEAD CONSTRUCTIONS, LTD. (B.P. 301,440, 30.8.27).—Apparatus for handling gases which have a corrosive action on steel is constructed of steel which is faced internally with concrete, the latter being preferably reinforced where exposed to corrosive action.

A. B. MANNING.

Separation of benzene and similar hydrocarbons from coke-oven and like gas by compression and cooling. GES. F. LINDE'S EISMASCHINEN A.-G., and SOC. MÉTALL. DE SAMBRE & MOSELLE (B.P. 275,633, 3.8.27. Ger., 4.8.26).—The compressed gas is cooled in a first stage to 40° only, eliminating naphthalene, lubricant oils, and water, which are led away through conduits and valves heated above this temperature, without condensing the benzene. It is then expanded under such conditions that the ratio of the pressure before and after expansion lies between 3 and 6, e.g., 4 atm. before and 1.1 atm. after expansion, so that the main portions of benzene and water are separated out from the gas before it enters the expansion machine. Those parts of the apparatus in which separation of solid benzene takes place are duplicated for use while the other is heated to melt the benzene. Alternatively, the separated benzene may be held in solution in toluene, and the mixture separated utilising the heat of compression.

H. S. GARLICK.

[Treatment of gases from] destructive hydro-generation of coals, tars, mineral oils, etc. I. G. FARBENIND. A.-G. (B.P. 279,072, 6.10.27. Addn. to B.P. 254,713; B., 1927, 644).—The process described in the main patent is modified by effecting the decomposition of the gas with carbon dioxide, either alone or in association with steam or other gases not richer in oxygen than air. The mixed gases are passed over heated refractory masses impregnated, if desired, with catalysts.

A. B. MANNING.

Treatment of the gaseous products liberated in the carbonisation of marine algæ. G. J. B. CHAMAGNE (B.P. 284,583, 11.8.27. Fr., 11.8.26).—The products evolved in the carbonisation of marine algæ in the furnace described in B.P. 275,998 (B., 1928, 325) are subjected to electrical precipitation, e.g., by arranging a high-tension electrical conductor axially in the smoke stack. The condensate is collected and treated for the recovery of its constituents, or is used directly, e.g., as an insecticide.

A. B. MANNING.

Distillation of tar, oil, and other materials. INTERNAT. COMBUSTION ENGINEERING CORP., Assees. of W. RUNGE (B.P. 295,945, 6.9.27. U.S., 21.9.26).—The

tar or oil is vaporised in a still of the pattern described in B.P. 277,952 (B., 1929, 116), and is then fractionally condensed. Tubular condensers are used, their temperatures being controlled by passing the hot waste gases from the still, cooled by the addition of the requisite amount of air, through the spaces about the tubes. The waste gases traverse the condensers in succession, the amount of air to be added thereto before entering each condenser being automatically regulated by means of a suitable thermostatic device. The last condenser, however, is water-cooled. A. B. MANNING.

Cracking of hydrocarbon oil. C. ARNOLD. From STANDARD DEVELOPMENT CO. (B.P. 302,211, 12.9.27).—Using a plant similar to that of B.P. 274,763 (B., 1927, 741), the oil to be cracked is first passed into the heated still, whence the vapours pass into the fractionating zone where light vapours are separated. The cracking coil and digester are used for cracking either a condensate from the fractionating tower or residual feed oil from the still. The contents of the digester may be discharged either into the still or into the fractionating tower, and a fraction from the tower may be returned to the still. H. S. GARLICK.

Conversion of hydrocarbon oils into lighter oils. C. ARNOLD. From STANDARD DEVELOPMENT CO. (B.P. 302,415, 22.9.27).—In the process of cracking oils under pressure by passing them rapidly through a heater coil and then into a large thermally insulated chamber where the oil remains for a substantial time at cracking temperature, the coil is operated at a temperature considerably higher than that desirable in the chamber, but excessive rise of temperature therein is prevented by adding a relatively cool oil to the hot oil at a point immediately before the hot oil enters the digestion chamber, whereby the temperature is controlled and the added oil brought up to cracking temperature. The cooling oil may be a diverted portion of the oil stream passing to the coil, or an oil adapted for cracking in the chamber, but not in the coil, and/or a condensate. H. S. GARLICK.

Manufacture of hydrocarbons. J. A. A. ZACON (B.P. 301,949, 7.9.27).—An aqueous emulsion of a mixture of a powdered and purified carboniferous product is continuously injected into a reaction apparatus in which it is exposed at 150 atm. and 300° to the action of an electric spark or arc, with or without the presence of hydrogenation catalysts. The products are expanded, cooled, and condensed, and the crude hydrocarbon mixture is fractionated. H. S. GARLICK.

Manufacture of valuable hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,253, 9.6.27).—Carbonaceous materials which contain substantial amounts of non-aromatic hydrocarbons are treated with an excess of hydrogen at temperatures above 550° and at pressures of at least 20 atm., preferably in the presence of catalysts, so that hydrocarbon mixtures are formed which boil below 200° and consist mainly of aromatic, unsaturated aliphatic, and hydroaromatic hydrocarbons. The catalysts used, *e.g.*, chromium oxide, alone or admixed with zinc, manganese, or aluminium oxides, etc., are so chosen that the cracking action predominates over the hydrogenating action. A. B. MANNING.

Purification of petroleum hydrocarbons. A. G. BLOXAM. From ALLGEM. GES. F. CHEM. IND. M.B.H. (B.P. 301,955, 9.9.27).—A diminished sulphur content and an improved odour of petroleum distillates are obtained by subjecting them to extraction with liquid sulphur dioxide and treating the remaining insoluble portion (after removal of sulphurous acid) with a dilute alkaline solution of lead oxide, with or without the addition of small quantities of elemental sulphur.

H. S. GARLICK.

Separation of gaseous or low-boiling hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,203, 8.9.27).—Cracked gases, especially olefines, liquefied if necessary, are separated into their constituents by fractional distillation under pressure preferably through several columns maintained at different temperatures and successively lower pressures. The separation of ethylene, propylene, and butadiene is described. C. HOLLINS.

Separation of paraffinous constituents etc. from [mineral] oils. AKTIEBOLAGET SEPARATOR-NOBEL, Assees. of AKTIEBOLAGET SEPARATOR (B.P. 276,658, 13.8.27. Swed., 24.8.26).—To a mixture of oil and paraffinous constituents in which the oil is the lighter constituent a heavier component is added which renders the material to be separated lighter than the oil mixture. The oil is separated in a centrifuge in which the chamber in which the lighter component collects is provided with a mechanical transporting device. By this means the separated material is positively and mechanically transported to the outlet continuously during the running of the centrifuge. H. S. GARLICK.

Decolorising or bleaching montan wax. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,626, 15.9.27).—Finely-divided crude or deresinified montan wax is suspended in a warm solution of a chromic salt. The requisite amount of chromic acid to effect bleaching is then run in and the temperature raised to above 100°, simultaneously evaporating water and separating the bleached wax from the spent solution. The wax is allowed to remain in a warm state until complete separation from emulsified solution has taken place. H. S. GARLICK.

Lubricating or like oils. H. L. CALLENDAR, R. O. KING, and E. W. J. MARDLES (B.P. 295,230, 4.5.27).—Partial decomposition and oxidation of mineral and vegetable lubricating oils may be prevented or hindered by the addition of a completely oil-soluble lead aryl or alkyl, with, if necessary, a small proportion of an organic halogen compound. Suitable proportions are 0.25–2% by vol. of lead tetraethyl. When the halogen compound is used it may be added, *e.g.*, as 2 pts. by vol. of the lead tetraethyl to 3 pts. of ethylene dibromide. E. HOLMES.

Method and apparatus for burning finely-divided fuel. PEABODY ENGINEERING CORP., Assees. of J. P. LEASK and H. T. DYER (B.P. 286,293, 18.2.28. U.S., 4.3.27).

Pulverised fuel burners. INTERNAT. COMBUSTION, LTD., and F. H. ROSENCRANTS (B.P. 302,195, 6.9.27).

Gas burners. SOUTH METROPOLITAN GAS CO., D. CHANDLER, and J. J. COOKE (B.P. 302,532, 11.4.28).

Incandescence gas burners. A. J. FALKNER (B.P. 294,827, 6.12.27).

Process and device for burning heavy oils. W. OSTHOFF (B.P. 303,300, 10.3.28).

Oxidation of organic compounds (B.P. 303,268).—See III. **Nitrogenised metallic compounds** (B.P. 277,714—5). **Hydrogen** (U.S.P. 1,689,858 and B.P. 276,687).—See VII. **Pickling of iron etc.** (B.P. 293,701).—See X. **Mineral oil derivatives** (U.S.P. 1,694,461—2).—See XII. **Medicinal paraffin** (B.P. 302,954).—See XX.

III.—ORGANIC INTERMEDIATES.

Synthesis of methyl alcohol. E. AUDIBERT (Chim. et Ind., 1928, 20, 1015—1022).—A *résumé* of the work carried out by the author on the synthesis of methyl alcohol from carbon monoxide and hydrogen under high pressure and presented at the Second Conference on Bituminous Coal, Pittsburg, 1928. The conditions of experiment throughout were: pressure 150 atm., rate of circulation of gases over catalysts 5000 m.³ (N.T.P.) per 1 m.³ of catalyst volume, and gas mixtures containing hydrogen and carbon monoxide in the ratio 5 : 1 (with catalysts having copper as the chief constituent), and 2 : 1 (with those of which zinc oxide is the basis), yields of methyl alcohol are expressed in g. per litre of catalyst volume per hour. It was found that in addition to the physical changes which occur in a catalyst as a result of prolonged use even with carefully purified gases, in the case of copper catalysts a "fouling" of the solid took place owing to the deposition of carbon compounds of high mol. wt. The rapidity of fouling is a function of the partial pressure of carbon monoxide in the gas, and it is reduced to an unimportant value when the proportion does not exceed one sixth. Zinc oxide catalysts do not foul with gases containing 33% of carbon monoxide; such a proportion, in fact, gives the optimum yields of methyl alcohol. When zinc oxide and copper catalysts are heated in hydrogen at, say, 550° for a number of hours, each suffers a loss of activity, that of the zinc oxide catalyst being the greater. The activity of a copper catalyst is completely removed by the addition of small amounts of the oxides of tin and bismuth; those of boron, titanium, and vanadium have no immediate effect, although they accelerate the rate at which prolonged heating reduces the activity. Aluminium and cerium oxides, on the other hand, increase the activity. The addition of beryllium and chromium oxides to a zinc oxide catalyst increases the activity, the presence of tungsten oxide affects it adversely, whilst uranium oxide is without effect. The yields of methyl alcohol obtained with each catalyst under the conditions specified above at temperatures corresponding to the optimum amounts are compared in the cases of copper and zinc oxide catalysts. With copper at temperatures not exceeding 275° the yield was 1100—1150 g., and with zinc oxide at 375°, 300—325 g. The effect of the pressure (*p* in atm.) on the yield, *R*, for a copper catalyst is represented approximately by $R = 0.050p^2$, and for zinc oxide by $R = 0.014p^2$. When the rate of flow over a zinc oxide

catalyst is increased from 5000 to 10,000 the yield rises by 55%, and from 10,000 to 20,000 by 35%. Small amounts of sulphur present as carbon disulphide or thiophen in an otherwise pure gas mixture poison the copper, but are without effect on the activity of the zinc oxide catalysts. Besides the desired reaction which produces methyl alcohol, a number of side reactions occur which result in the formation of water, carbon dioxide, and methane. From a study of the relative rates of the main and side reactions it is shown that the useful conversion of carbon monoxide is much greater with a copper catalyst than is the case with one of zinc oxide. In deciding the relative value of the two types of catalyst, the poor yields from zinc oxide have to be considered in conjunction with the robustness and indifference to small traces of poisons, whilst the extra yields from copper catalysts have to be off-set against the much greater sensitiveness to poisoning.

H. INGLESON.

Analysis of mixtures containing acetone, ethyl alcohol, and isopropyl alcohol. C. A. ADAMS and J. R. NICHOLLS (Analyst, 1929, 54, 2—9).—Tables are given of the immersion refractometer readings of various concentrations of acetone and some of the lower alcohols, and it was found that in aqueous mixtures of such substances, provided the total proportion does not exceed 10% by vol., the apparent proof strength and the refraction of the mixture are practically the sum of those due to each ingredient, and up to about 17% of apparent proof spirit the sp. gr. and refractions may be regarded as additive factors. A rapid method for the detection and colorimetric determination of acetone is given, based on Penzoldt's method (Z. anal. Chem., 1885, 24, 149), in which indigo is formed as a condensation product when sodium hydroxide is added to a mixture of *o*-nitrobenzaldehyde and acetone. *iso*Propyl alcohol may be determined by oxidising with bromine water to acetone, and proceeding as for acetone, and for a mixture of acetone, ethyl alcohol, and *iso*propyl alcohol, the conditions for complete oxidation of each of these constituents are given, and a method for their determination.

D. G. HEWER.

Sp. gr. and immersion refractometer readings of dilute mixtures of acetone and water. J. R. NICHOLLS (Analyst, 1929, 54, 9—11).—Two samples of acetone were prepared: (a) by carefully fractionating commercial acetone, distilling twice from dichromate and sulphuric acid, shaking the distillate with anhydrous potassium carbonate, again distilling, shaking the constant-boiling fraction with fused calcium chloride for several days, refractionating, and repeating the process until the sp. gr. was constant; (b) by similarly fractionating and drying a sample prepared from the sodium iodide compound. Weighed quantities of each were diluted with water to 100 c.c. and the sp. gr. and refractometer readings taken, and from the plotted results the equivalent proof strength and refractometer readings are tabulated for 0 to 10 g. of acetone per 100 c.c.

D. G. HEWER.

Routine catalytic preparations in an organic course. S. GOLDSCHMIDT and L. ORTHNER (Z. angew.

Chem., 1929, 42, 40—42).—Apparatus and analytical methods suitable for students' use are described and illustrated with reference to acetone from acetic acid over iron turnings, methyl or ethyl acetate or *iso*amyl propionate from acid and alcohol, aniline from nitrobenzene, acetaldehyde from alcohol, keten from acetone, and ethylene from alcohol. C. HOLLINS.

Technical importance of adipic acids and their derivatives. W. SCHRAUTH (Chem.-Ztg., 1929, 53, 41—43).—Attention is drawn to possible technical applications of adipic acid, now rendered readily accessible by new methods for oxidation of *cyclohexanols*. It is completely non-toxic, and is an advantageous substitute for tartaric or phosphoric acid in baking-powder. The more soluble β -methyladipic acid is suitable for lemonade crystals. Various applications of the acids in textile, leather, and dyeing industries are mentioned. The esters, *e.g.*, methyl*cyclohexyl* β -methyladipate ("Sipalin MOM"), are used as plasticisers for cellulose esters, and in printing of textiles, whilst with glycerol etc. adipic acids give resins. The sodium salts of acid adipates of higher alcohols are powerful emulsifying and wetting-out agents, and the neutral higher esters are fixatives for perfumes. C. HOLLINS.

[Preparation of] derivatives of *s-m*-xylenol. E. NOELTING (Sealed Note 2333, 4.1.15. Bull. Soc. Ind. Mulhouse, 1928, 94, 648—649). Report by M. BATEGAY (*Ibid.*, 650—651).—Brief reference is made to the preparation of a number of substances from 5-hydroxy-*m*-4-xylyl methyl ketone and 2-amino-*m*-5-xyleneol. A brown sulphur dye was obtained by treatment of the last-named substance with sodium sulphide and sulphur, and a green dye by condensation with tetramethyldiaminobenzhydrol and subsequent oxidation. Battagay draws attention to a number of other researches relating to similar substances.

A. J. HALL.

Potentiometric and conductometric analysis. CALLAN and HORROBIN.—See I. **Carboxylic acids in peat tar** (STADNIKOV and SABAWIN). **Pityrol.** KOMATSU and others. **ψ -Cumene derivatives in wood spirit.** HOLMBERG.—See II.

PATENTS.

Catalytic preparation of oxygenated carbon compounds. E. I. DU PONT DE NEMOURS & Co., Assees. of W. A. LAZIER (B.P. 272,555, 13.6.27. U.S., 12.6.26).—For the hydrogenation of carbon oxides a chromite catalyst is used, obtained, *e.g.*, by heating a chromate or dichromate of zinc, copper, cadmium, magnesium, manganese, silver, or iron at 800—900°.

C. HOLLINS.

Vaporisation of formamide. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 301,974, 17.9.27).—Formamide is vaporised with only slight decomposition by bringing it, preferably in a finely-divided condition, into contact with surfaces heated above its b.p. so that it is vaporised immediately without accumulating in liquid form in the vaporiser. B. FULLMAN.

Manufacture of solid polymerised formaldehyde. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,258, 2.12.27).—Aqueous formaldehyde, preferably over 30%, is treated with sodium carbonate (0.5 pt.

per 100) below 50° in absence of gelatinising agents such as alkaline soaps. With more dilute formaldehyde solutions cooling below 0° is necessary. A filterable product is obtained.

C. HOLLINS.

Production of acetaldehyde. H. S. HIRST, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 302,515, 25.1.28).—Gases poor in acetylene, *e.g.*, those derived from methane by thermal decomposition, are scrubbed with a countercurrent of dilute sulphuric acid containing mercuric and ferric sulphates at 60—70° and at pressures up to 10 atm. Acetaldehyde and a gas richer in hydrogen are produced. A continuous process is described.

C. HOLLINS.

Catalytic oxidation of organic compounds. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 303,268, 21.12.27).—Organic compounds (*e.g.*, mineral oil, neutral oxidation products of higher paraffins, paraffin wax) are oxidised in the liquid phase by air in presence of manganese acetylacetone or other metal enolates.

C. HOLLINS.

Production of aliphatic ketones. HOLZ-VERKOHLUNGS-IND. A.-G., and K. RÓKA (B.P. 302,759, 18.10.27).—Alcohols having at least two carbon atoms, the corresponding aldehydes, or esters are converted into acetone or other ketones by passage with steam at about 500° over a catalyst made, for example, by evaporating a stirred mixture of calcium acetate solution and rusty iron shavings. Acetaldehyde, ethyl alcohol, or ethyl acetate yields acetone.

C. HOLLINS.

Manufacture of isothiocarbamide ethers. SCHERING-KAHLBAUM A.-G. (B.P. 296,782, 28.10.28. Ger., 9.9.27).—Cyanamides are treated with thiol derivatives in aqueous or alcoholic solution in presence of an acid, or the vaporised thiol may be passed into the cyanamide solution. With disubstituted cyanamides the reaction is effected at 100°, dimethyl- or diethyl-cyanamide and methyl mercaptan yielding, *e.g.*, NN-dimethyl- or -diethyl-S-methylisothiocarbamide. R. BRIGHTMAN.

Manufacture of substituted guanidines. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 303,044, 26.9.27).—Thiocarbanilide, or other substituted thiocarbamide, is treated with a zinc compound and an alkali or alkaline-earth in presence of ammonia or a substituted ammonia. Thiocarbanilide with aniline gives triphenylguanidine, with benzylamine *diphenylbenzylguanidine*, m.p. 102—103°; the methyl- (m.p. 108—109°) and dimethyl-compounds (m.p. 199°) are similarly obtained. With phenylhydrazine *anilinodiphenylguanidine*, m.p. 160°, and its oxidation product, an azo compound, m.p. 111°, are produced, whilst phenylhydroxylamine yields N-hydroxytriphenylguanidine, m.p. 154°.

C. HOLLINS.

Manufacture of higher alkylated guanidine derivatives. SCHERING-KAHLBAUM A.-G. (B.P. 279,884, 27.10.27. Ger., 28.10.26).—Cyanamide, an alkyl cyanamide, or a monoalkali salt of cyanamide in slight excess is treated, *e.g.*, in aqueous or alcoholic solution, with the salt of an alkylamine, the alkyl group or groups of which together contain more than four carbon atoms. *isoAmylguanidine sulphate*, m.p. 266° (decomp.), *hexylguanidine*

sulphate, m.p. 255°, heptylguanidine, pentamethylenediguanidine sulphate, decomp. above 320°, diguanylhexamethylene sulphate, decamethylenediguanidine sulphate and hydrochloride, N-methyl-N'-heptylguanidine picrate, and amylguanidine sulphate, m.p. 130°, are described.

R. BRIGHTMAN.

Recovery of volatile organic solvents. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,297, 12.9.27).—The vapours containing the solvent to be recovered are scrubbed in a countercurrent of products, b.p. above 200°, obtained by the catalytic hydrogenation of oxides of carbon.

C. HOLLINS.

Preparation of sodium nitrogen compounds. DEUTSCHE GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 293,040, 26.6.28. Ger., 1.7.27).—Sodium hydride reacts with arylamines, acid amides or imides, etc. to give N-sodio-derivatives, often at temperatures below 100°. Examples are aniline, methylaniline, *p*-chloroaniline, naphthylamines, diphenylamine, acetamide, carbamide, guanidine, phthalimide, succinimide, diacetamide, phenylthiocarbamide, carbazole, aminotriazole, and aminothiazole. For acid amides a solvent is necessary to moderate the reaction.

C. HOLLINS.

N-Aminoalkylation of amines. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 301,401, 24.5.27).—Amines are heated with the hydrochloric acid salts of aminoalkyl *p*-toluenesulphonates. Aniline and the ester hydrochloride of ϵ -diethylaminopentan- β -ol give ϵ -diethylamino- β -amylaniline, b.p. 150–154°/6 mm. From 8-aminoquinoline and the γ -ester hydrochloride of N- β - γ -dihydroxypropylpiperidine 8-(β -hydroxy-1- γ -piperidylpropylamino)quinoline, b.p. 212–213°/1 mm., is obtained.

C. HOLLINS.

Manufacture of aromatic mercaptans [thiophenols]. I. G. FARBENIND. A.-G. (B.P. 279,136, 18.10.27. Ger., 18.10.26).—Diazo compounds, not containing solubilising groups, are converted smoothly into thiophenols or thionaphthols by treatment with a metal polysulphide and subsequent reduction of the disulphide so formed. The polysulphide must contain more sulphur than is required by the formula M'₂S₂ or M'S₂. The preparation of 5-chloro-*o*-thiocresol is described.

C. HOLLINS.

Manufacture of alkylated phenols and their hydrogenated products. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 274,439, 17.6.27. Ger., 14.7.26. Addn. to B.P. 254,753; B., 1928, 740).—The process of the prior patent is applied to the condensation products of aldehydes (formaldehyde, acetaldehyde, or benzaldehyde) to give phenol and *p*-cresol, phenol and *p*-hydroxyethylbenzene, or phenol and *p*-hydroxydiphenylmethane, or hexahydro-derivatives of these.

C. HOLLINS.

Manufacture of anthraquinone derivatives. I. G. FARBENIND. A.-G. (B.P. 282,004, 9.9.27. Ger., 9.12.26).—2:3-Benzbenzanthrone, m.p. 229°, is oxidised with chromic-acetic acid to 1-*o*-carboxyphenylantraquinone, m.p. 236°; the dichloro-derivative (from 2:6-dichloroanthraquinone) behaves similarly.

C. HOLLINS.

Reduction of nitroanthraquinones. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 302,964, 22.9.27).—

Nitroanthraquinones are smoothly reduced when heated with tetrahydro-quinoline or quinaldine in a solvent (pyridine, quinoline, quinaldine, *o*-dichlorobenzene, acetic acid, etc.). At moderate temperatures (125°) the nitro-groups are reduced to the hydroxylamino-stage, at higher temperatures to the amino-stage.

C. HOLLINS.

Manufacture of aminated sulphurised [i.e., sulphur-containing] benzanthrone derivatives. I. G. FARBENIND. A.-G. (B.P. 275,271, 2.8.27. Ger., 2.8.26).—Benzanthronyl sulphides, disulphides, thiol compounds, thio-ethers, etc. are heated with hydroxylamine and sulphuric acid in presence of ferrous or copper sulphate to give amino-derivatives. Amino- and diamino-3:3'-dibenzanthronyl sulphides, a bromo-derivative of the former, and an aminated oxidation product of dibenzanthronyl sulphide are described.

C. HOLLINS.

Separation of hydrocarbons (B.P. 302,203).—See II. Diazo compound and dyes therefrom (B.P. 288,572). Stable diazo compounds (B.P. 280,945). Azo intermediates (B.P. 302,770 and 302,773).—See IV.

IV.—DYESTUFFS.

Electrochemical reduction of azo dyes to their respective amino-compounds. L. H. HUBBUCH [with A. LOWY] (Amer. Electrochem. Soc., May, 1929. Advance copy. 13 pp.).—Six azo dyes, Methyl Orange, Metanil Yellow, Acid Scarlet, Crimson, Congo Red, and Fast Brown O, representing six different types, have been reduced electrolytically to the corresponding pairs of amino-compounds in a sodium carbonate solution, using a mercury cathode. The successful reduction of Methyl Orange and Metanil Yellow indicates that the statements of Stohr and Schneider, that only azo compounds having an amino- or a hydroxyl group in the *o*- or *p*-position to the azo group can be electrolytically reduced to amines, are erroneous. The conditions generally most favourable for the reduction are: 95°, 0.885 amp./dm.², and 7.5 g. of dye and 7.5 g. of sodium carbonate in 200 c.c. of solution. Current efficiencies obtained were often above 80% under these conditions.

H. J. T. ELLINGHAM.

Potentiometric and conductometric analysis. CALLAN and HORROBIN.—See I. Derivatives of *s-m*-xyleneol. NOELTING; also BATTEGAY.—See III.

PATENTS.

Manufacture of condensation products of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 275,636, 4.8.27. Ger., 4.8.26).—Aminoanthraquinones are condensed with chlorohydrins to give dyes for acetate silk, oils, fats, fatty acids, etc. Examples are: 1- β -hydroxyethylaminoanthraquinone, m.p. 148–150° (red); 1- β - γ -dihydroxypropylaminoanthraquinone, m.p. 192° (red to orange-red); 1- β -hydroxyethylamino-4-hydroxyanthraquinone (violet); 1:4-di-(β -hydroxyethylamino)anthraquinone (blue); 1:5-isomeride (bluish-red).

C. HOLLINS.

Manufacture of dyes of the anthraquinone series. L. CASSELLA & Co. G.M.B.H. (B.P. 295,600, 13.8.28. Ger., 11.8.27. Addn. to B.P. 260,998; B., 1928, 225).—

Chlorination of anthraquinone proceeds smoothly in 90% sulphuric acid at 45–50° in presence of a chlorine carrier. The process can be conveniently combined with the cyclisation of 1:1'-dinaphthyl-8:8'-dicarboxylic acid in monohydrate, which may be diluted to 90% for chlorination without isolation of the anthranthrene.

C. HOLLINS.

Manufacture of brown vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,489, 5.12.27. Addn. to B.P. 289,980; B., 1928, 517).—The brown vat dye of the prior patent is reduced to leuco-compound and halogenated in presence or absence of catalysts to give, after re-oxidation, fast brown vat dyes. The ·N:N· group is probably converted into ·NH·NH·.

C. HOLLINS.

Manufacture of vat dyes [of the dibenzanthrone series]. SOC. CHEM. IND. IN BASLE (B.P. 279,479, 20.10.27. Switz., 20.10.26).—A dibenzanthrone is sulphonated, fused with alkali, and finally alkylated and/or halogenated, to give bluish-grey vat dyes.

C. HOLLINS.

New vat dyes of the dipyrazolanthrone series. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 302,772, 31.10.27).—Dipyrazolanthrone or one of its derivatives is condensed with aldehydes in nitrobenzene, in presence of a chloride of phosphorus, antimony, or aluminium. Dipyrazolanthrone with formaldehyde, acetaldehyde, benzaldehyde, *o*- or *m*-chlorobenzaldehyde, *m*-nitrobenzaldehyde, or *m*-hydroxybenzaldehyde, dichlorodipyrazolanthrone with benzaldehyde, tetrabromodipyrazolanthrone with benzaldehyde, and ethyldipyrazolanthrone with formaldehyde give yellow to orange vat dyes.

C. HOLLINS.

Manufacture of a new diazo compound and of new dyes therefrom. COMP. NAT. MAT. COL. ET MANUF. DE PROD. CHIM. DU NORD RÉUNIES, ÉTABL. KUHLMANN (B.P. 288,572, 23.3.28. Fr., 12.4.27).—Diazotised 4-chloro-*o*-nitroaniline is treated with an alkali (sodium acetate, carbonate, etc.) to give 4-chlorobenzene-1:2-diazo-oxide, which is coupled with naphthols, aminonaphthols, pyrazolones, sulphonic acids of these, or resorcinol to yield chromable dyes. The latter may be converted into copper or chromium complex salts.

C. HOLLINS.

Manufacture of stable diazo-compounds. SOC. CHEM. IND. IN BASLE (B.P. 280,945, 18.11.27. Switz., 18.11.26. Addn. to B.P. 238,704; B., 1925, 840).—Diazo solutions (other than those from aminoazo compounds) are evaporated with sodium 1:3:6- or 1:3:7-naphthalenetrisulphonate to give stable diazo preparations, to which a little sodium hydrogen carbonate may be added to aid re-dissolution if desired. Diluents such as salt, sodium sulphate, or aluminium sulphate may be added to the diazo solution before evaporation.

C. HOLLINS.

Manufacture of [azo] dyes containing chromium. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,709, 15.9.27).—Sulphoarylpyrazolones are used in place of the sulphonamide derivatives of B.P. 210,669 (B., 1924, 412), and give valuable dyes when coupled, for example, with diazotised 1:2:4-aminonaphthol-sulphonic acid and pre-chromed.

C. HOLLINS.

Manufacture of new *o*-hydroxyazo dyes and an intermediate product. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 302,770, 28.10.27).—2:6-Hydroxynaphthoic acid is sulphonated and the product is fused with alkali to give 2:8-dihydroxy-6-naphthoic acid, which when coupled with diazotised *o*-aminophenols yields brown wool dyes becoming brown to black on chroming and having good milling fastness. Examples of diazo components are 4-chloro-*o*-aminophenol, 4-nitro-*o*-aminophenol, 5-nitro-3-aminosalicylic acid, picramic acid, 5-nitro-3-amino-*p*-cresol, 4-chloro-6-nitro-*o*-aminophenol, *o*-aminonaphtholsulphonic acids, and *o*-amino-hydroxyazo compounds.

C. HOLLINS.

Manufacture of new azo dyes and intermediate products. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 302,773, 31.10.27).—Arylamides of 2-aryl-amino-8-hydroxy-6-naphthoic acids are coupled in substance or on the fibre with a diazo compound. 2-Anilino-8-hydroxy-6-naphthoic acid, obtained from 2:8-dihydroxy-6-naphthoic acid (B.P. 302,770, preceding) by the action of aniline and bisulphite, yields an *anilide*, decomp. 170°, *p*-anisidide, m.p. 160° (decomp.), and β -naphthylamide, m.p. 170–175° (decomp.). The anilide is coupled with diazotised *p*-chloroaniline (dark olive), 5-nitro-*o*-toluidine (dark olive), *o*-aminoazotoluene (brownish-black), *o*-phenetidine \rightarrow α -naphthylamine (black), *p*-nitroaniline-*o*-sulphonic acid (dark brown on wool), *m*-aminobenzoic acid (similar), the *p*-anisidide with 4-chloro-*o*-toluidine (brown-olive), 4-chloro-*o*-nitroaniline (grey-brown), 2:5-dichloroaniline (reddish-brown), 4:4'-diaminodiphenylamine (black); the β -naphthylamide with dianisidine (blue-black, suitable for lakes).

C. HOLLINS.

Manufacture of azo dyes containing chromium and their application. O. Y. IMRAY. From SOC. CHEM. IND. IN BASLE (B.P. 301,772, 4.8.27).—Loss of dye in the form of insoluble complex compounds during pre-chroming is avoided by adding an organic salt and/or an organic acid, or an inorganic salt and an organic acid; if the addendum also serves as an acid-binding agent, excess is added. Examples are: dye, chromium formate, sodium acetate, with or without formic acid; dye, chromium formate, sodium chloride, and formic acid.

C. HOLLINS.

Manufacture of a black tetrakisazo dye. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 300,722, 29.8.27).—Benzidine is tetrazotised and coupled successively with 1 mol. of H-acid in acid solution and 1 mol. of *m*-phenylenediamine in alkaline solution, the resulting disazo dye being then coupled alkaline with 2 mols. of diazotised *p*-aminoacetanilide. On alkaline hydrolysis a tetrakisazo dye is obtained which gives greenish-black shades on chrome leather. Alternatively, *p*-nitroaniline may be used in place of *p*-aminoacetanilide and the tetrakisazo dye reduced.

R. BRIGHTMAN.

Preparation of triarylmethane dyes. BRIT. DYESTUFFS CORP., LTD., F. W. LINTH, and E. H. RODD (B.P. 301,193, 17.11.27).—Condensation of tetraalkyldiaminobenzophenones with aromatic chloro-compounds (B.P. 272,321; B. 1927, 598) is effected at 60°

and in presence of benzene as solvent, giving higher yields and purer products. R. BRIGHTMAN.

Oxidation of leuco-compounds of the triaryl-methane series. I. G. FARBERIND. A.-G. (B.P. 299,473, 26.10.28. Ger., 28.10.27).—Leuco-compounds of triarylmethane dyes are oxidised with air in presence of a copper salt and pyridine or other cyclic tertiary base, with addition of a diluent (glycerol) if desired.

C. HOLLINS.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Tensile strength of abacá fibres in relation to their acidity. P. L. and H. E. SHERMAN (Philippine J. Sci., 1928, 37, 21—40).—An examination of various samples of abacá fibre indicates that the higher is the natural acidity of the fibre the lower are its elasticity and tensile strength; the same relationship holds in the main for Canton fibres, but with less uniformity. The loss of strength of abacá during storage is also affected by the acid content, but no definite relationship was found between the rate of loss of tensile strength and the acid content determined at the end of the storage period.

D. J. NORMAN.

Fermentation as affecting the quality of Philippine abacá. T. BAÑUELOS and P. L. SHERMAN (Philippine J. Sci., 1928, 37, 41—65).—Abacá fibre as commercially produced is heavily infected with bacteria; drying the fibre immediately after stripping causes a partial sterilisation, which remains effective so long as the fibre is kept dry, but should the drying process be omitted, or should the dried fibre be wetted again, fermentation sets in, the immediate results of which are increased acidity, and loss of colour and tensile strength. This deterioration appears to be due to acid fermentation of the soluble constituents of the fibre and to bacterial attack of the fibre itself. D. J. NORMAN.

Heartwood of the pine. I. Adsorption and infiltration experiments with sapwood and heartwood of pine and spruce. II. Extraction by organic solvents and alkalis. C. G. SCHWALBE and A. AF EKENSTAM (Cellulosechem., 1929, 10, 1—11, 11—18).—I. The experiments included measurements of the rate and amount of adsorption of water vapour and of infiltration by water and various solutions, and were designed particularly for comparison of the heartwood and sapwood of the woods examined. It was found that pine heartwood in all circumstances absorbed less liquid than the other samples. In infiltration experiments with both pine and spruce the absorption of water by green sapwood was greater than that by green heartwood, but with increasing dryness the two values converged. Pine heartwood adsorbed more sodium hydroxide than spruce heartwood, but this difference disappeared when the former had been extracted with ether. In pine heartwood the sodium salt of an ether-soluble acid is formed, and partly retained by the wood. In sealed tubes at 110° pine heartwood showed no difference in ability to take up liquid, but it differed particularly in absorbing Mg^{++} ions to a relatively larger extent than HSO_3^- ions from magnesium bisulphite solution, so that the composition of the absorbed salt

corresponded to that of a neutral sulphite. This, which is the chief cause of the failure of acid disintegration processes, is ascribed to comparatively slow diffusion of HSO_3^- ions, whilst the Mg^{++} ions appear to react with an acid of high mol. wt. in the wood.

II. The amounts extracted by ether were in the order: pine heartwood > pine sapwood > spruce heartwood > spruce sapwood. Since pine sapwood can be disintegrated by the usual process, failure in the case of heartwood is considered to be due to a difference in the nature rather than in the amount of resinous substances present. Examination of the ether extracts showed that pine sapwood contained a larger proportion of fats (soluble in light petroleum) than the heartwood. The similarity of acid values indicated the presence of an acid of high mol. wt. in pine heartwood. The amount extracted by ether from pine heartwood decreased with time of storage, the fats decreasing most rapidly. Again, the residues obtained by evaporation of the extracts became insoluble when heated or kept. These effects are explained by the precipitation of colloidal constituents. Benzene extracted smaller amounts from pine heartwood than ether, but alcohol extracted larger amounts, including the fraction which had become insoluble in ether on storage. Pine heartwood yielded the largest amount of extract with 1% sodium hydroxide solution, and this was largely precipitated by hydrochloric acid. The precipitate was extracted with alcohol, and the alkaline solution fractionally precipitated by acid. Similar results were obtained with pine heartwood and sapwood. Large amounts of very weakly dissociated acids were present, and an acid with an insoluble calcium salt was isolated. Spruce, on the other hand, when similarly treated, yielded acids which were more strongly dissociated and precipitated within narrower limits of hydrogen-ion concentration. Comparison of the ether extracts of the substances precipitated from the sodium hydroxide extracts of pine heartwood and sapwood showed that the former contained a quantity of an acid with a soluble calcium salt which was lacking in the latter. Carbohydrates, compounds containing nitrogen or sulphur, or tannins could not be recognised in the extract of pine wood by 1% sodium hydroxide.

R. K. CALLOW.

Lignosulphonic acid obtained from spruce wood by the action of sulphurous acid in presence of ammonia. C. DORÉE and E. C. BARTON-WRIGHT (J.S.C.I., 1929, 48, 9—12 T).—The acid obtained by the above process of resolution due to Cross and Engelstad (*ibid.*, 1925, 44, 267 T), in which 0.1—0.5% of ammonia is present, has been isolated and its properties are compared with those of the acid obtained in the original process, in which sulphurous acid alone was employed. The acid has the formula $C_{40}H_{44}O_9 \cdot 2H_2SO_3$ and is present apparently as a monoammonium salt. It was purified by dialysis, conversion into a β -naphthylamine derivative, and resolution of this compound by means of pyridine. Experimental results are summarised in the dissected formula $C_{35}H_{33}O_2 \cdot CO \cdot CH(OH) \cdot (OH)_2 \cdot (OMe)_2 \cdot (SO_3H)_2$. On oxidation with nitric acid a nitro-compound, containing 2 carbonyl and 2 nitro-groupings, is formed. The nitro-compound is reduced by magnesium, the nitro-groupings being eliminated

and replaced by carbonyl groupings. In its reactions the acid closely resembles the acid obtained by the action of sulphurous acid alone, for which a formula $C_{26}H_{30}O_{12}S$ was found (Dorée and Hall, *ibid.*, 1924, 43, 257 r). The C_{40} -unit formula of the acid now described brings it into line with lignosulphonic acids isolated by previous workers.

Composition of commercial artificial silks. A. WAHL and J. ROLLAND (Rev. Gén. Mat. Col., 1929, 33, 1—4).—The moisture contents of commercial samples of viscose, hollow viscose, nitro- and cuprammonium silks, as determined after exposure of the material over sulphuric acid (at R.H. not stated) lies between 10 and 12%, whilst their ash content is 0.1—0.56%. The sulphur content, determined by ashing the finely-divided material in the presence of magnesium oxide and sodium and potassium carbonates, with subsequent dissolution of the ash and precipitation of the sulphate in the usual way, generally varies from 0.24 to 0.42%. The copper numbers (Braidy) of Tubize and of Charbonnet nitro-silk are approx. 3, those of viscoses, including the hollow variety Celta, 0.7—1.2, and of cuprammonium silk 0.5—0.7. Values for other kinds of cellulose material such as cotton and wood pulp, bleached to different extents, are included for comparison. The high values recorded, particularly for the nitro-silks and for some samples of viscose, are attributed to the presence of oxycellulose in the material, and suggestions are put forward to explain its formation.

B. P. RIDGE.

Viscosity in relation to cellulose acetates. M. DESCHIENS (Chim. et Ind., 1928, 20, 1023—1033).—The different methods of determining viscosity are explained and the range of their suitability is pointed out. In France, the official (Aeronautic) standard practice for cellulose acetate solutions is to employ 6 g. of the ester in 100 c.c. of solvent, and to compare the viscosity, in the same apparatus, with that of glycerin at d 1.26 and at 15° taken as 100. This practice is compared with that given in Brit. Eng. Stand. Assoc., No. 83, 1918. Relative values of viscosity of some cellulose acetates of commerce suitable for varnishes and dopes are given. The effect of variations in the proportions of the constituents of a dope on the viscosity has been examined. Acetates suitable for use as dopes or varnishes should be among the most rapidly soluble, and should have a viscosity when dissolved in acetone of 12—30. Solvents which possess the greatest dissolving power are those which give the most rapid dissolution, and also give solutions of the desired viscosity with the smallest volume of solvent. In general, such solutions are limpid, and have the least tendency to coagulate. The heterogeneity of cellulose esters of commerce can be readily demonstrated.

H. INGLESON.

Determination of the transparency of paper to light. W. HOLWECH (Papier-Fabr., 1929, 27, 37—45).—A method of determining the amount of light transmitted by paper and the absorption coefficient of the latter is described which involves the use of a polarisation photometer. The influence of the wave-length of the incident light, of increasing the number of layers of the same

paper, and of different kinds of paper, on the results obtained has been investigated.

B. P. RIDGE.

Bleaching cellulose. ESCOURROU.—See VI.

PATENTS.

Treating or retting flax fibre. M. WADDELL and H. C. WATSON (B.P. 302,300, 14.9.27).—The flax fibre in the form of a rove is immersed in water containing yeast at 21—27° with or without addition of sulphurous acid; it is finally treated with a tar product to prevent further fermentation.

F. R. ENNOS.

Degreasing of raw wool. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 300,941, 20.8.27).—Raw wool is treated with a mono- or di-chlorinated hydrocarbon, b.p. 175—200°, the solvent being subsequently removed from the wool by washing and aeration and from the wool fat by steaming.

F. R. ENNOS.

Manufacture of powdered plastic material. SOC. CHIM. DES USINES DU RHÔNE (B.P. 285,829, 31.1.28. Fr., 22.2.27. Addn. to B.P. 275,558; B., 1928, 330).—A mixture of cellulose ester or ether with a plasticiser is precipitated from solution by distillation of the solvent in the presence of a liquid which does not dissolve the material and which is miscible with and less volatile than the solvent. The precipitating liquid may be added to the solution of the plastic material before or during distillation of the solvent, or to the solvent itself before forming the solution.

F. R. ENNOS.

Manufacture of copper oxide-ammonia cellulose solutions for artificial silk production. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 302,956, 21.9.27).—Raw cellulose is pressed into thin paper-like sheets, avoiding unnecessary mechanical and chemical treatment, and owing to the large surface area dissolution is effected in 10—12% copper oxide-ammonia solution in about 1 hr.

W. G. CAREY.

Manufacture of ethers of carbohydrates. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 302,191, 9.8.27).—High-molecular carbohydrates, e.g., cellulose and starch, are treated with a gaseous alkylating agent, e.g., ethyl or methyl chloride, in the presence of moisture and less than 20 mols. of an alkali hydroxide per 1 mol. of carbohydrate, calculated as $C_6H_{10}O_5$. The carbohydrate may be kept in motion during the treatment, or may be treated with a current of the gaseous agent, e.g., by circulating it, alone or mixed with a non-reacting gas, successively through a heater, through the treatment chamber, and through a cooler to condense and remove water, whence it is returned to the circuit.

L. A. COLES.

Manufacture of aminocellulose derivatives. I. G. FARBENIND. A.-G. (B.P. 279,801, 14.9.27. Ger., 30.10.26).—Sodium cellulose is esterified with benzene- or toluenesulphonyl chloride, and then acetylated or alkylated and finally treated with a primary, secondary, or tertiary amine; alternatively, the amine treatment may precede the acetylation or alkylation. The "animalised cellulose" so obtained, as also its mixtures with ordinary cellulose esters or ethers, has affinity for acid dyes. Amines used in the examples are aniline, diethylamine, pyridine, and isoamylamine.

C. HOLLINS.

Composition of matter containing a cellulose derivative. H. M. WEBER, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,690,515, 6.11.28. Appl., 13.10.25).—Glycerol, or other polyhydric alcohol, is heated at 235–265° with an organic acid and a vegetable oil, or a blown vegetable oil, or fatty acids. Thus, glycerol, phthalic anhydride, and tung oil give a resin, m.p. 73°, acid value 56, miscible with nitrocellulose, whilst glycerol, phthalic anhydride, and the fatty acids from soya-bean oil yield a similar resin, m.p. 71°, acid value 17.

R. BRIGHTMAN.

Manufacture of artificial silk by the dry-spinning method. SOC. FABR. SOIE "RHODIASETA" (B.P. 288,618, 14.9.27. Fr., 14.4.27. Addn. to B.P. 238,842; B., 1926, 627).—The spinning operation takes place in an evaporating atmosphere richly laden with the solvent vapour, the required concentration of which is maintained by a gaseous exchange between the atmosphere of the heated spinning cell and that of the cooled solvent condenser through a single large aperture connecting one with the other.

F. R. ENNOS.

Manufacture of rayon, artificial horsehair, films, etc. DU PONT RAYON Co., Assees. of W. H. BRADSHAW (B.P. 271,517, 23.5.27. U.S., 21.5.26).—Artificial silk of high wet strength is prepared by using as parent material a cellulose containing 98–100% of α -cellulose and conducting all subsequent operations under such conditions that the degree of hydration of the cellulose is not materially increased. Thus, in the manufacture of cuprammonium silk, purified cotton linters (99% of α -cellulose) is dissolved in cuprammonium hydroxide solution and the solution is beaten in a closed vessel with just sufficient air or oxygen to reduce the viscosity to the required degree without affecting the α -cellulose content. *E.g.*, the partial pressure of air in the mixer may be reduced to less than 4 in. of mercury, or the air may be suitably diluted with inert gases. When ready, the solution, containing, *e.g.*, 6% of cellulose, 5.5% NH_3 , and 2.5% Cu, is stretch-spun into 22–27% caustic soda solution at 0–14° and is preferably collected in a centrifugal spinning box. After keeping it for about 15 min. to complete the coagulation, the thread is passed through 20% sulphuric acid at 20° and wound on a rotating cylinder, the lower part of which is immersed in 2½% sulphuric acid at 20°. The speed of reeling and the length of travel of the thread in the 20% acid bath should be so adjusted that the acid carried over is sufficient to maintain the weak acid bath at a constant concentration. The thread is finally washed with water, drained, and dried.

D. J. NORMAN.

Cyclic process for manufacture of kraft pulp. G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,689,534, 30.10.28. Appl., 4.12.25).—Raw cellulose material is digested in a liquor containing sodium sulphide. After separation of the pulp from the spent liquor, the latter is evaporated and the inorganic constituents are smelted in a reducing atmosphere, the sodium and sulphur present as sublimed solids and sulphur dioxide in the products of combustion being absorbed in more spent liquor, which is also evaporated and smelted. The recovered solids are dissolved in water and used for the digestion of other material, any deficiency of sulphur or sodium being made

up by addition during the smelting and dissolution stages respectively.

F. R. ENNOS.

Manufacture of pulp board. H. T. PRICE (U.S.P. 1,696,896, 25.12.28. Appl., 13.4.27. Appl., 13.4.27. Austral., 14.6.26).—The board consists of finely-ground ti-tree wood.

H. ROYAL-DAWSON.

Saccharification of cellulose (B.P. 273,317).—See XVII. **Treatment of textiles** (B.P. 272,541).—See XXIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Action of ultra-violet rays in bleaching cellulose.

R. ESCOURROU (Chim. et Ind., 1928, 19, 989–997).—The effect of ultra-violet radiation in accelerating the bleaching of wood-pulp pastes (in conditions varying from the raw to bleached) has been examined by exposing the reactants to the radiation from a mercury-vapour lamp. When chlorine bleaching agents (*e.g.*, hypochlorite solutions) are used, the acceleration produced enables the reaction to be carried out at a lower temperature, but more chlorine is used up owing to increased formation of chlorates (which play no part in bleaching) and to the fact that under these conditions the cellulose is attacked as well as the non-cellulose impurities. The material thus obtained is rich in oxy- and hydro-cellulose, as shown by its enhanced copper number (Hägglund), its α -cellulose content falls, and the β - and γ -contents are increased. Whilst this may not be important in papermaking, it is a disadvantage when the material is to be used for making viscose and some kinds of cellulose nitrate. This effect is shown only when chlorine bleaching agents are used; it is not observed when bleaching with ozone or hydrogen peroxide. In this latter case the degraded forms of cellulose are not produced, and normally low copper numbers are found. Under these conditions α -cellulose remains unaffected, and for the viscose industry, which demands high α - and low β - and γ -cellulose contents, the use of oxidising agents such as ozone or hydrogen peroxide, either alone or in conjunction with hypochlorite solutions, is recommended.

B. P. RIDGE.

Behaviour of cellulose acetate towards amino-derivatives of anthraquinone. H. M. BURNS and J. K. WOOD (J. Soc. Dyers and Col., 1929, 45, 12–15; cf. Kartaschov, B., 1926, 49, 188; 1928, 812).—Experiments were carried out in order to test the hypothesis that the dyeing of cellulose acetate silk with amino-anthraquinones is attributable to adsorption. The bases employed were purified samples of α -, β -, and 1:4-diamino-anthraquinone suspended in 0.5% solutions of gelatin. To 100 c.c. each of a number of suspensions of various concentrations was added 0.2 g. of dried cellulose acetate silk, and the materials were kept at 26° for 8 days, being shaken at intervals. Portions of the suspensions were then removed and the amount of base remaining was determined. When the equilibrium values showing the concentration of the base on the fibre and in the liquid, respectively, were plotted against each other, curves approximating in shape to those of an ordinary adsorption isotherm were obtained. Of the three bases, the 1:4-diamino-compound was adsorbed most strongly and the β -compound least, but there did

not appear to be any definite numerical relationship between their degrees of adsorption. As the adsorption was irreversible the authors conclude that after the adsorption of the base by the fibre a secondary action occurs leading to the formation of a complex, possibly a solid solution. L. G. LAWRIE.

Application of Naphthol AS dyes to animal fibres. J. RATH (Textilber., 1928, 9, 585—586).—Priority is claimed for the successful application of Naphthol AS dyes to wool and silk. The fabrics may be satisfactorily "prepared" with a Naphthol AS compound by the usual methods (but using the minimum quantity of caustic alkali) without suffering deterioration. To obtain satisfactory Naphthol AS dyeings on animal fibres, coupling of the developer and naphthol should be effected under acidic conditions. It is suggested that the usual addition of formaldehyde to the "prepare" liquor should be omitted in the case of wool, but not of silk. Naphthol AS dyeings on animal fibres have excellent fastness, their fastness to light on wool being greater than that on cotton; dyeings on silk are resistant to processes of degumming. A. J. HALL.

Reserves under sulphur colours [in printing]. F. WOSNESSENSKY (Sealed Notes Nos. [A] 1920, [B] 1926, [C] 1945, [D] 1951, and [E] 1952, 3.8., 2.9., 30.10., and 19.11.09. Bull. Soc. Ind. Mulhouse, 1928, 94, 657—660). Report by P. BINDER (*Ibid.*, 660—661).—(A) Khaki-coloured reserves under sulphur colours are obtained by means of thickened pastes containing nitroso- β -naphthol, zinc chloride, and a metallic mordant (salts of iron and cobalt for greenish and reddish shades of khaki, respectively). (B) Reserves coloured with Thioindigo Red B or Scarlet R, or brominated indigo dyes are obtained by means of a thickened paste consisting of the indigo dye, glycerin, Rongalite, zinc chloride, and sodium acetate. (C) For indigo reserves a paste is used prepared by adding indigo powder, glycerin, Rongalite solution, and anthraquinone paste to a thickened solution of zinc chloride and caustic soda or sodium acetate. (D) Blue reserves under sulphur colours cannot be obtained satisfactorily by means of Dianisidine Blue because the acid treatment of fabric after printing affects the blue coloured reserves; good reserves result if the β -naphthol usually employed is replaced by 2:3-hydroxynaphthoic acid. (E) A Sulphur Blue reserve may be obtained by means of a paste consisting of the leuco-derivative of Sulphur Blue (B.A.S.F.), glycerin, sodium hyposulphite, and a thickened solution of zinc chloride. The leuco-derivative is prepared by concentrating a mixture of Sulphur Blue, in alkaline sodium hyposulphite solution, to which ice and acetic acid have been added. BINDER reports favourably on the processes. A. J. HALL.

PATENTS.

Dyeing of textile fibres, films, etc. made with or containing cellulose esters or ethers. BRIT. CELANESE, LTD. (B.P. 282,036, 23.11.27. U.S., 11.12.26).—Tightly wound, knitted, knotted, or woven goods of cellulose acetate etc. are evenly dyed by applying suitable dyes in solution in an organic solvent, *e.g.*, benzene, trichloroethylene, alcohols, or mixtures of these.

C. HOLLINS.

Treatment [loading, mordanting, or dyeing] of materials made of or containing cellulose derivatives [esters or ethers]. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 302,775, 2.11.27).—Acetate silk etc. is treated with alkaline tin solutions under conditions which bring about partial hydrolysis, with or without addition of swelling agents. Material so treated may be dyed with direct, mordant, or vat dyes as well as with the usual basic or insoluble colours.

C. HOLLINS.

Printing with vat dyes. E. C. R. MARKS. FROM E. I. DU PONT DE NEMOURS & Co. (B.P. 302,252, 8.6.27).—Textiles are printed with vat dye pastes containing, besides reducing agent, tri-(β -hydroxyethyl)amine or a similar substance.

C. HOLLINS.

Coating, impregnating, dressing, sizing, and printing of fibrous material. L. LILIENTHAL (B.P. 302,115, 20.6.27).—A viscose solution into which air has been injected is applied to the fabric, and the cellulose is regenerated to produce a dressing containing hollow spaces distributed throughout its mass; this gives to the dressing a downy texture. Gas generated within the viscose solution from sulphites or carbonates has a similar effect.

C. HOLLINS.

Treatment [delustring] of artificial silk. H. A. GARDNER (U.S.P. 1,692,372, 20.11.28. Appl., 13.5.27).—The silk is impregnated with 4% of its weight of a soluble titanium salt, which is hydrolysed at 65—100° to give a matt deposit of insoluble titanium compound in the silk.

C. HOLLINS.

Aminocellulose derivatives (B.P. 279,801).—See V.

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

B.p. of electrolytic caustic solutions. C. C. MONRAD and W. L. BADGER (Ind. Eng. Chem., 1929, 21, 40—42).—The b.p. of solutions of sodium hydroxide of varying concentration and saturated with sodium chloride were redetermined to an accuracy of $\pm 0.2^\circ$ for pressures varying from 90 to 760 mm. The boiling flask was of monel metal and the thermometer tube used was nickel-plated, whilst an automatic device kept the pressure constant to within 1—2 mm. The slope of the Dühring line for solutions of increasing concentration shows several discontinuities when plotted, the form of the curve closely resembling the f.p. curve of similar solutions.

C. IRWIN.

Synthetic ammonia plant at Ostend. F. A. F. PALLEMAERTS (Ind. Eng. Chem., 1929, 21, 22—29).—This plant is one of a number recently erected obtaining hydrogen from coke-oven gas treated by the "U.C.B." process and then liquefied. Under conditions in Belgium such hydrogen costs less than half that made from water-gas. The cost of production of ammonium sulphate is given as 9 American cents/kg. of nitrogen. The U.C.B. process consists in treating the gas with successive washes of ammonia solution, water, sulphuric acid, and caustic soda. The purified gas is then compressed in two stages to 9 atm., refrigerated, warmed again, and subjected to a pressure water-wash mainly for the purpose of removing acetylene. The ammonia wash

reduces the carbon dioxide of the gas to below 0.1%. The pressure water-wash also removes any benzene which may not have been recovered previous to the water-wash by the refrigerator. The pure gas is cooled to -45° and fractionated. Ethylene, methane, and carbon monoxide are in turn condensed, and the final gas mixture is further purified by washing with liquid nitrogen. This removes methane entirely and leaves only 0.001% of carbon monoxide. The rich residual gas is returned to the ovens, but the possibility exists of other uses, particularly for ethylene. The nitrogen required comes partly from the gas and partly from the liquid nitrogen produced in a Linde liquid air plant. The Casale plant at 750 atm. produces all its ammonia in a liquid form, and it is therefore available for refrigeration. The Casale catalysis tube, which has a life of 1 year (after which the tube should be tested and the catalyst changed, though the tube may last much longer), is insulated from the catalyst space by the incoming cold gases. The reaction is kept slightly endothermic by allowing some ammonia to remain uncondensed in the gas mixture and regulated by an electrically-heated resistance. The ammonia is at present converted into sulphate with sulphuric acid, but it is intended to replace this by converting into ammonium carbonate with flue gases and then proceeding in a way similar to the gypsum process.

C. IRWIN.

Solid solutions of lime and arsenic acid. A. T. CLIFFORD and F. K. CAMERON (Ind. Eng. Chem., 1929, 21, 69—70).—Varying quantities of arsenic acid solution were added to a saturated solution of calcium hydroxide with stirring. A precipitate formed in every case and the whole was kept for 2 months at 25° with occasional shaking. The ratio of calcium oxide to arsenic acid in the liquid and solid phases was then determined. The ratio in the solid phases showed a continuous variation according to the quantity of arsenic acid added. It is found that for concentrations up to 25 g. per litre of arsenic oxide, solid solutions are formed, and there is no evidence of the real existence of tricalcium arsenate.

C. IRWIN.

The system ferrous oxide-silica. C. H. HERTY, JUN., and G. R. FITTERER (Ind. Eng. Chem., 1929, 21, 51—57).—When silicon is used for the deoxidation of steel ferrous silicate inclusions are produced. A series of m.p. determinations were made for various compositions of the system ferrous oxide-silica, using slags prepared in the laboratory. For each the softening point was also recorded. The m.p. of ferrous oxide was determined as 1355° . Minimum m.p. were observed with mixtures containing 22% of silica and 34% of silica, but the softening points scarcely follow the m.p. curve. The curve is nearly straight, from the second eutectic up to 60% SiO_2 , at which point it intersects the transition line from cristobalite to tridymite. Micro-analysis confirmed the existence of the first eutectic as a mixture of fayalite ($2\text{FeO} \cdot \text{SiO}_2$) and ferrous oxide. Photomicrographs are given and the system is represented in a three-dimensional diagram including the heat content together with temperature and composition.

C. IRWIN.

Preparation of artificial white earths. M. NEK-

RITSCH (Z. anorg. Chem., 1928, 177, 86—90).—By the interaction of aluminium sulphate and sodium silicate solutions a precipitate of high adsorptive power is produced, which contains much crystalline material and does not yield up all its aluminium on treatment with acids. The composition, although somewhat variable, is approximately that of hydrated aluminium silicate, $\text{Al}_2\text{O}_3 \cdot 7\text{SiO}_2 \cdot n\text{H}_2\text{O}$. The material may be used for the adsorption of gases and for the purification of mineral and vegetable oils, and resembles closely natural bentonite.

H. F. GILLBE.

Sublimation of sulphur between 25° and 50° .

R. P. TUCKER (Ind. Eng. Chem., 1929, 21, 44—47).—The sublimation of sulphur at temperatures of 25° and upwards was observed in flasks with sulphur in the bottom, crystals in the upper part appearing in 2—4 weeks. The rate of sublimation was observed by consideration of the time necessary to produce a black film on silver foil. It increased very rapidly with temperature. An admixture of 10% of lampblack increased the rate of sublimation in sunlight owing to the higher temperature reached. Attempts were made to compare the sublimation rates of different forms of commercial sulphur by the silver-foil method, by weighing, and by microscopical measurement. Whilst results were variable no definite differences could be detected, and a mixture of equal parts of sulphur and calcium carbonate ground together sublimed as readily as pure sulphur. The rate of sublimation at temperatures below 35° decreases with time. The insecticidal value of sulphur is considered to depend on sublimation.

C. IRWIN.

Mists and dusts. REMY. **Potentiometric and conductometric analysis.** CALLAN and HORROBIN.—See I. **Ammonia from coke-oven gas.** LIEPIATSKIK, also DENISOV.—See II. **Phosphates for plants.** UNGERER. **Manuring with iodine.** DOERELL.—See XVI.

PATENTS.

Acid-concentrating chamber. O. C. TRAUTMANN (U.S.P. 1,695,619, 18.12.28. Appl., 15.4.27).—A series of concentrating chambers has horizontally projecting tubular parts joined in stairway fashion so that only these horizontal parts are located in the inclined flue from a firebox, the joints being on the outside of the flue. Means for preheating the acid and connecting the preheater with the uppermost chamber are provided, and an aperture is arranged in each chamber for escape of the vapours.

W. J. BOYD.

Catalytic apparatus for the synthesis of ammonia.

F. C. REED (U.S.P. 1,689,684, 30.10.28. Appl., 2.8.27).—A number of concentric members are connected as a unit to the cover of a pressure vessel forming a catalytic chamber above which is an annular heating chamber having a heat exchanger in the upper portion and a heater in the lower portion. Gases are admitted to the heating chamber and are led into the catalytic chamber at points midway in its wall, being directed in an axial direction by an annular baffle. They then pass from the centre of the catalytic chamber to the heat exchanger. A cylindrical shell is attached to the cover of the vessel and

is spaced therefrom, and a circulating pipe and condenser connect the upper and lower portions of the vessel.

W. C. CAREY.

Manufacture of alkali cyanides. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 301,565, 6.9.27).—A mixture of ammonia and carbon monoxide is caused to react at 400–800° with the hydroxide, oxide, or salt of an alkali metal diluted with an indifferent filler, *e.g.*, magnesium oxide, and with the addition of a suitable catalyst, *e.g.*, an iron compound not reduced during the reaction.

W. G. CAREY.

Production of nitrogenised metallic compounds [cyanides, cyanamides, and nitrides]. R. B. GOLDSCHMIDT and S. COULIER (B.P. 277,714, 20.9.27. Belg., 20.9.26).—Mixtures, containing excess carbon, of raw peat with alkali, alkaline-earth, or earth-metal compounds for the production of cyanides or cyanamides, *e.g.*, sodium carbonate, or with magnesium or aluminium compounds for the production of the nitrides of these metals, together with catalysts for fixing nitrogen, *e.g.*, iron, manganese, nickel, or cobalt, or their oxides, or alkali halides, and catalysts for activating the carbon, *e.g.*, zinc or magnesium chloride, oxide, or phosphate, or titaniferous compounds, are briquetted, dried if desired, carbonised, and nitrogenised. After the product has been extracted, the residual carbon is purified, and part of it may be added to a fresh charge. L. A. COLES.

Production of nitrogenised metallic compounds. R. B. GOLDSCHMIDT and S. COULIER (B.P. 277,715, 20.9.27. Belg., 20.9.26).—The compounds are obtained by the carbonisation and nitrogenisation of mixtures of carbonaceous material, *e.g.*, wood, spent malt, oil cake, etc., with alkali, alkaline-earth, or earth-metal compounds, together with catalysts, if desired. Activating gases, *e.g.*, steam, carbon monoxide, carbon dioxide, hydrogen, nitrogen, or chlorine, are passed over the material during the carbonisation, and excess of active carbon, obtained in a previous operation, is added to the mixture before and/or after the carbonisation, the excess of carbon being separated from the nitrogenised product after the treatment. Both processes may be effected in the same vertical or inclined retort in which a descending mass of the mixture is treated with an ascending stream of the gases.

L. A. COLES.

Production of calcium cyanamide or mixtures containing it. N. CARO and A. R. FRANK (B.P. 279,419, 30.9.27. Ger., 23.10.26).—Calcium carbonate is treated with ammonia or mixtures of it with non-reacting gases, at above 450° and at 3–10 atm., the apparatus being constructed of material which does not cause dissociation of ammonia, *e.g.*, quartz, ceramic material, or copper or its alloys. Means are provided for condensing and removing the steam liberated during the reaction.

L. A. COLES.

Freezing mixtures. L. A. and B. GARCHEY (B.P. 298,623, 4.8.28. Austr., 14.10.27).—Ammonium nitrate and crystallised sodium carbonate are mixed in equal proportions at the time of use and water is added.

W. G. CAREY.

Producing a mixture of calcium nitrate and ammonium nitrate. KUNSTDÜNGER PATENT-VER-

WERTUNGS A.-G., Assees. of F. G. LILJENROTH (B.P. 301,486, 23.10.28. Sweden, 1.12.27).—A mixed fertiliser of calcium and ammonium nitrates is produced by acting on calcium sulphate with ammonia and carbon dioxide, treating the resulting calcium carbonate, together with a suitable amount of ammonium sulphate left therein, with nitric acid, and removing the precipitated calcium sulphate.

W. G. CAREY.

Reduction of [barium] sulphate minerals and briquettes therefor. J. E. BOOGE and J. P. KOLLER, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,685,772, 2.10.28. Appl., 10.11.24).—Barytes (4 pts.) and coal (1 pt.) are mixed with a binder, *e.g.*, starch, molasses, or a crude oil, and the mixture is formed into briquettes having a diameter of 1–4 in. On heating the briquettes at 950–1150° a high yield of barium sulphide is obtained even with ore containing up to 10% of silica, owing to the low porosity of the briquettes preventing ingress of furnace gases.

A. R. POWELL.

Production of anhydrous zinc chloride. METALLBANK U. METALLURGISCHE GES. A.-G. (B.P. 238,253, 1.3.28. Ger., 9.4.27).—Zinc oxide is treated at 400–500° with chlorine in the presence of excess of a mixture of hydrogen and carbon monoxide.

W. G. CAREY.

Production of water-softening silicate. E. W. SHAWEN and A. C. ARNETT (U.S.P. 1,693,873, 4.12.28. Appl., 9.2.25).—Sodium bicarbonate is incorporated with porous vitreous material, the mixture is heated, and while hot is treated with sodium chloride.

W. G. CAREY.

Separation of gases [helium] from [natural] gaseous mixtures. M. H. ROBERTS, Assr. to AIR REDUCTION Co., Inc. (U.S.P. 1,693,052, 27.11.28. Appl., 25.1.19).—Natural gas is compressed, cooled, and expanded to liquefy a portion of it, and the residual gas is subjected to successively lower temperatures, at the pressure to which it was expanded, to condense constituents other than helium, the condensed fractions being used separately and successively for cooling the gas to such a temperature that only the helium remains uncondensed.

L. A. COLES.

Production of hydrogen of great purity. A. W. BURWELL, Assr. to ALOX CHEM. CORP. (U.S.P. 1,689,858, 30.10.28. Appl., 22.8.27).—Hydrocarbon material is heated at 1050–1150° to produce a mixture of hydrogen, carbon monoxide, carbon, and hydrocarbons, from which carbon is removed by settlement and aromatic hydrocarbons by scrubbing with water. Hydrocarbons similar to methane are converted into carbon monoxide by heating the moist mixture at 1230–1280°, and the carbon monoxide is converted into dioxide by adding excess of oxygen to the dried gases and passing them over a manganese dioxide catalyst below 100°. Any oxygen remaining is removed by passing the gases over heated copper, and carbon dioxide by caustic alkali solution.

W. G. CAREY.

Purifying hydrogen and gases containing hydrogen. GEWERKSCHAFT DER STEINKOHLENZECHÉ MONT-CENIS (B.P. 276,687, 26.8.27. Ger., 30.8.26. Cf. B.P. 276,668; B., 1928, 894).—Organic compounds detrimental to contact processes are removed by passing the

gases at high pressure and temperature over catalysts, *e.g.*, iron, cobalt, nickel, chromium, manganese, zinc, or vanadium oxides or metals, with the addition of alkaline-earth oxides, so that inorganic compounds and saturated hydrocarbons are formed, the former being absorbed by basic or oxidising substances added to the catalyst. Copper is excluded from the process.

W. G. CAREY.

Production of sulphur dioxide and hydrogen.

C. H. MACDOWELL and H. H. MEYERS, Assrs. to ARMOUR FERTILIZER WORKS (U.S.P. 1,693,244, 27.11.28. Appl., 21.11.27).—A mixture of sulphur vapour and steam is heated at a suitable temperature in the presence of iron oxide as catalyst for a sufficient time to yield a mixture containing sulphur dioxide and hydrogen, and these gases are recovered separately from the product.

L. A. COLES.

Contact sulphuric acid process. SELDEN Co.,

Assees. of A. O. JAEGER (B.P. 294,975, 23.1.28. U.S., 3.8.27).—See U.S.P. 1,675,308; B., 1928, 783.

Sodium nitrogen compounds (B.P. 293,040).—See III.

VIII.—GLASS; CERAMICS.

New type of tunnel kiln for firing pottery. J. WILLIAMSON (Trans. Ceram. Soc., 1928, 27, 290—296).—A tunnel kiln of the combined direct-flame and muffle type is described. It consists of preheating, equalising, furnace, and cooling zones. In the furnace zone the flames take a transverse course, either by up- or down-draught. Six burners are provided on either side, spaced a truck length apart. The flames from the burners meet a stream of hot air at the top of the bag walls. The equalising zone is a double-vaulted muffle which completely encircles the trucks. The flue systems in the various zones are described. F. SALT.

[Effect of heat on] the crystalline break-up of kaolin. J. F. HYSLOP and H. P. ROOKSBY (Trans. Ceram. Soc., 1928, 27, 299—302; cf. B., 1928, 605).—The β -phase previously reported has been identified as α -alumina. The α -phase formed at 550° has not been identified. This breaks down at 870° with the formation of γ -alumina and sillimanite. There is no evidence of the formation of α -alumina above 870°, though between this temperature and 1060° the γ -alumina decreases and finally disappears. Mullite is formed above 1060°.

F. SALT.

Drying of clay. R. S. TROOP and F. WHEELER (Trans. Ceram. Soc., 1928, 27, 303—319).—The contraction of clay and its relation to rate of drying and shape of piece, and the effect of the physical conditions of the drying medium on the rate of removal of moisture from the clay and on the distribution of moisture in the piece have been studied. Measurement of linear contraction is rejected as a basis for comparing the drying behaviour of clays because it is vitally affected by compression and extension stresses associated with the method of making the test pieces. Rectangular test pieces showed irregular contraction on the different faces, and also at different points along the same face. Volume contraction was measured in a mercury volumeter. Results are given for fireclay, china clay, and ball clay. With china clay an expansion occurred between a moisture

content of 11% and the dry state. The rate of drying appeared to have no direct effect on contraction. A "main contraction" occurs in the early stages, when the contraction is equivalent in volume to the amount of water removed by evaporation; at a "critical point" the rate of contraction decreases suddenly, and "residual contraction" then proceeds at a much slower rate. The "plasticene coating method" of measuring porosity was also applied to the measurement of contraction. Measurement of the rate of drying of clay cylinders by air of definite humidities and temperatures (with apparatus previously described; B., 1928, 569) indicated lower water gradients in ball clay than in fireclay samples. An increase in the drying temperature reduced the water gradients in clays, and thus ensured safer drying. F. SALT.

Crushing strength of unfired fireclay bodies.

W. C. HANCOCK and J. G. COWAN (Trans. Ceram. Soc., 1928, 27, 243—246).—A Stourbridge clay, and four mixtures of this clay with 20% and 40% of coarse and fine grog prepared from another hard-fired Stourbridge clay, were tested in the dry state for crushing strength, and the results were compared with the tensile strength. Three shapes of test-piece were used in determining the crushing strength. The highest figures were obtained with a mixture of 80% of clay and 20% of fine grog. Three types of fracture observed in the crushed pieces are illustrated. F. SALT.

Refractory material used as mortar for laying-up refractories. D. A. MOULTON (Trans. Ceram. Soc., 1928, 27, 329—333).—Bentonite is a good "floating" medium to prevent settling of a refractory cement when mixed to a thin consistency. A satisfactory refractory cement is made from ground fireclay grog, of similar chemical composition to the refractories, mixed with a colloidal material such as bentonite or aquacryptite. A monolithic lining for various types of furnaces may be made from this cement, using coarse grog.

F. SALT.

Artificial white earths. NEKRITSCH.—See VII.

PATENTS.

Manufacture of glass threads. F. POLLAK (CHEM. FABR. STOCKERAU DR. F. POLLAK) (B.P. 288,978, 14.4.28. Austr., 16.4.27).—The glass is fused by an electric resistance heating member suitably distributed in the melting pot, and the fused glass is discharged through nozzles.

W. G. CAREY.

Prevention of ageing in ceramic materials. F. SINGER (B.P. 282,404, 14.12.27. Ger., 18.12.26. Addn. to B.P. 282,403; B., 1928, 712).—The ceramic materials previously described are treated with oxides, carbonates, silicates, or aluminates of cerium, zirconium, chromium, manganese, phosphorus, tungsten, or vanadium in such amount that the product is practically entirely micro-crystalline.

W. G. CAREY.

Bonded refractory. R. E. LOWE, Assr. to DOHERTY RES. Co. (U.S.P. 1,694,924, 11.12.28. Appl., 28.4.25).—A graded refractory aggregate is bonded by less than 1% of a hydroxide precipitated in intimate contact with the particles of aggregate by such means that practically no other substances are formed during the precipitation.

L. A. COLES.

IX.—BUILDING MATERIALS.

Weathering [of the building stone] of the Bremen Town Hall. E. BLANCK and A. RIESER (Chem. Erde, 1928, 4, 137—144).—Analyses are given of the weathered red sandstone of the building erected in 1609—1612.

L. J. SPENCER.

Reaction of water on calcium aluminates. L. S. WELLS (U.S. Bur. Stand. Res. Paper No. 34, 1928, 59 pp.).—A study has been made of the mechanism of the reaction of water on a commercial high-alumina cement and on four anhydrous calcium aluminates ($\text{CaO}:\text{Al}_2\text{O}_3$ ratios of 1:1, 3:5, 5:3, 3:1, respectively). The reaction of water with the last-named compound is very vigorous, and the changes in composition of the resulting aqueous solutions could not be followed. The rapid setting is probably due to the formation of a hydrate occurring directly or by the reaction of the excess of lime on a calcium aluminate produced in the very early stages of setting. The activity of the other aluminates and of the high-alumina cement towards water is distinctly less than that shown by tricalcium aluminate. During the early periods of the reaction a portion of the lime and alumina in each case dissolves as the calcium salt of monobasic aluminic acid, the total concentration depending on the composition of the anhydrous aluminate or cement from which it is formed as well as on the time of contact. These solutions are metastable and soon decompose with precipitation of a portion of the lime and alumina. The molar ratio of the former to the latter remaining in solution after precipitation shows an increase, attended in each case by an increase in the p_H from approx. 11.1 to 11.75. Identical changes occur in the metastable solutions after filtration from the reaction mixture. The "equilibrium" solutions which result when the filtered, metastable solutions are kept are similar to those obtained after prolonged contact of the reaction mixture with water, and contained 0.35—0.50 g. of lime and 0.15—0.30 g. of alumina per litre. The establishment of equilibrium requires several weeks. As equilibrium is approached hydrated alumina and hydrated tricalcium aluminate are precipitated. Calculations based on electrometric measurements and chemical analyses indicate that the alumina exists in solution as the calcium salt of monobasic aluminic acid, and that the excess of calcium hydroxide determines the p_H of the solution. The conclusion of Slade (A., 1913, ii, 54), Blum (A., 1913, ii, 963), and Heyrovsky (A., 1922, ii, 771) that aluminic acid is a relatively strong monobasic acid is confirmed by these calculations, which, however, are only approximate. As the concentration of calcium hydroxide is raised, increasing precipitation of the alumina occurs until at $p_H \geq 12$ it is complete and *tetracalcium aluminate*, $4\text{CaO}.\text{Al}_2\text{O}_3.12\text{H}_2\text{O}$, appears to be formed. Analogous changes occur in aluminate solutions when mixed with tricalcium silicate. The electrometric titration of aluminium chloride with calcium hydroxide shows that precipitation of aluminium is complete between p_H 6.0 and 7.5, but that the aluminium hydroxide begins to redissolve at p_H 9.0, dissolution being complete at p_H 10.9. At still higher values of p_H calcium chloroaluminate is precipitated. It may also be prepared by acting on an aluminate

solution with calcium chloride, and when the concentration of the latter exceeds 3% the precipitate approximates in composition to $3\text{CaO}.\text{Al}_2\text{O}_3.\text{CaCl}_2.10\text{H}_2\text{O}$. The corresponding barium compound appears to be more soluble than the calcium chloroaluminate. Ammonia and magnesium chlorides decompose calcium aluminate solutions, precipitating, respectively, aluminium hydroxide and a mixture of aluminium and magnesium hydroxides.

H. INGLESON.

Hardening of Portland cement and the liberation of lime. S. YAMANE (Bull. Inst. Phys. Chem. Res., Tokyo, 1928, 7, 1177—1190).—The lime content of Portland cement attains a value of over 5% within 7 days of mixing with water, and increases with the water content of the mixture. The strengthening process taking place in Portland cement is due to the reaction: $3\text{CaO}.\text{SiO}_2 + \text{H}_2\text{O} \rightarrow \text{CaO}.\text{SiO}_2.m\text{H}_2\text{O} + \text{Ca}(\text{OH})_2$, and the concentration of hydrated silicate might be taken as a measure of the strength of a given sample of cement. Sand has, in Portland cement mixtures, amongst other functions that of conserving water, whereby a more complete utilisation of $3\text{CaO}.\text{SiO}_2$ is attained. If hardened cement be powdered and extracted with warm glycerol-ethyl alcohol solution, an opalescent filtrate is obtained, the particles of which are coagulated by the addition of water. It follows that hardened Portland cement contains substances in a state of colloidal division.

R. TRUSZKOWSKI.

Setting of cement. II. H. GESSNER (Kolloid-Z., 1929, 47, 65—76; cf. B., 1929, 56).—An apparatus is described for the measurement of the viscosity of cement pastes. Measurements of viscosity were made with pastes containing 30—33 pts. of water to 100 pts. of cement. Immediately after mixing, such a paste has a viscosity comparable with that of glycerol, but the viscosity slowly increases with time and the change becomes rapid after 0.5—1.5 hrs. The change is considered to be due to the formation of a gel through hydration of the particles. The measurements were made on different test portions of the cement; if the same portion of paste is passed repeatedly through the viscosity apparatus, little change in the viscosity is indicated, since the gel structure is destroyed. The electrical conductivity of cement was also measured during setting, and was found to reach a maximum after about 40 min. The subsequent fall in conductivity is rapid at first and gradually slows down, taking some months for completion. With hydraulic mortars, the reduction in volume after mixing with water takes place rapidly during the first five days, then more slowly for about 20 days, and the change is not completed until after about 275 days. With Portland cement the decrease in volume amounts to 3% after 1 day, 4—7% after 3 days, 5—8% after 7 days, 5.5—9% after 28 days, and 6—9% after 3 months. A hydraulic lime under the same conditions showed a smaller decrease in volume, viz., 1% after 1 day, 2% after 10 days, and 4% after 250 days. An alumina cement showed a greater volume decrease than Portland cement, viz., 11% after 1 day, 13% after 5 days, 16% after 28 days, and 17% after 150 days.

E. S. HEDGES.

Refractory cement. MOULTON.—See VIII. **Dental materials.** COLEMAN.—See X.

PATENTS.

Manufacture of artificial stone of crystalline structure. G. CONOD and F. C. F. LECOULTRE (B.P. 288,202, 26.3.28. Belg., 4.4.27).—The stone (artificial marble) is obtained by mixing a binding agent, *e.g.*, calcium sulphate, aqueous colloidal silica solution, and powdered calcium carbonate obtained synthetically in a crystalline form by heating limestone at about 1290° under 100 atm. pressure and dropping it into water while still hot, or by adding sodium carbonate to calcium bicarbonate solution, colouring matter being added if desired.

L. A. COLES.

Wet process for manufacturing cement. W. P. ECKDAHL (U.S.P. 1,693,644, 4.12.28. Appl., 24.5.26).—The setting of a slurry of finely-divided silicious material having a basic reaction is prevented by bringing gases with a high content of carbon dioxide into contact with it.

A. R. POWELL.

Hardened plaster product. Plaster board. W. H. KOBÉ, Assr. to TEXAS GULF SULPHUR Co. (U.S.P. 1,693,715—6, 4.12.28. Appl., [A, B] 14.3.27).—(A) Dehydrated, set gypsum is impregnated either wholly or in part with sulphur. (B) The board comprises an interior of hardened plaster covered in part at least with fibrous material which is bonded to the interior by sulphur.

W. G. CAREY.

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

Protection of underground pipe from corrosion. Method used in Southern California. E. O. SLATER (Ind. Eng. Chem., 1929, 21, 19—21).—The deterioration of underground pipe lines in relation to the capital value involved is discussed. The rate of corrosion is determined to a major extent by the character of the soil and soil water in contact with the pipe. Any protection against corrosion apart from corrosion-resistant metal must therefore prevent effective contact between the moist soil and the pipe line. A coating successfully used in Southern California consists of a paint produced from native bitumens. The bitumen coating on previously sand-blasted metal usually proves a satisfactory protection, but where the soil conditions are known to be severe a bitumenised cotton wrapping is applied with a subsequent "paint" coating.

C. A. KING.

Effect of additions of lime and soda ash to brackish water on the corrosion of iron and steel. H. O. FORREST, J. K. ROBERTS, and B. E. ROETHELI (Ind. Eng. Chem., 1929, 21, 33—35).—As a protection against the corrosion of iron and steel particularly in cooling systems situated along sea coasts and using brackish water, the formation of a calcium carbonate scale was tried with success. Small quantities of calcium bicarbonate in distilled water greatly accelerate corrosion, but higher concentrations assist in building up a carbonate film. In waters containing 50% or more of sea-water the protection afforded by treatment with calcium hydroxide is relatively poor, being about

30—40% as a maximum. This is due largely to the presence of magnesium salts which probably lower the hydrogen-ion concentration of the water, and which also produce a non-adherent precipitation of magnesium hydroxide. The further addition of sodium carbonate corrects this tendency, and when the p_H of the solution was 8.5 or above a satisfactory protective coating was obtained.

C. A. KING.

Influence of nitrogen on the solubility of ferrous materials in hydrochloric acid. II. Effect on the carbon content. III. Further experiments. H. H. GRAY and M. B. THOMPSON (J.S.C.I., 1929, 48, 21—25 T, 25—28 T; cf. B., 1926, 919).—II.—The ferrous material was heated for a period (usually 6 hrs.) in pure dry nitrogen at temperatures up to 1100°. Nitrogen is found to decarburise ferrous alloys with the formation of a volatile compound, probably cyanogen. This reaction is found to be reversible. The loss of carbon depends on temperature and on the initial carbon content of the alloy. The importance of the results in connexion with case-hardening and nitrogen fixation is indicated. The literature of the subject is reviewed.

III. The solubility of the nitrogenated material is found to be affected by the initial carbon content and by the temperature of nitrogenation. Microscopical examination shows the nitrogenated metal to be covered with a transparent, crystalline layer which is practically insoluble in dilute hydrochloric acid. This is the chief obstacle to dissolution in acid, but the underlying mass of the ferrous material is also rendered less soluble by the nitrogenation. The "resistivity" shown to hydrochloric acid is confirmed by *E.M.F.* experiments. The appearance of the nitrogenated material is illustrated by means of a photomicrograph showing a well-developed crystalline structure without the use of etching reagents.

Illustration of the transformations of brasses containing 57.5—63.5% Cu by the study of their mechanical properties. P. DEJEAN (Compt. rend., 1929, 188, 169—172).—The temperature-plasticity curves of these alloys show two points of inflexion corresponding with the transformation temperatures observed by other workers. The first change occurs at 480° for each alloy, and is considered to be the conclusion of a progressive transformation starting at 250—300°. The second represents the transformation of the ($\alpha + \beta$) into the β -constituent, and occurs at a temperature (685—800°) which increases with the copper content of the brass.

J. GRANT.

Internal transformations of a cupro-aluminium. A. ROUX and J. COURNOT (Compt. rend., 1929, 188, 172—173).—The structural transformations of a 90:10 cupro-aluminium have been studied by means of X-rays before and after annealing for 20 min. at 900° and slow cooling, quenching in cold water after 5 min. at 900°, and reheating of the quenched metal for 20 min. at 650° or 200°. Only the spectrum of the α -solution appears in that of the annealed metal; in the case of the quenched metal this spectrum shows with additional lines, but without the lines corresponding with the [100] plane. A solid solution of Cu_3Al in the α -solution is indicated.

J. GRANT.

Determination of traces of antimony in copper and its alloys. S. G. CLARKE and B. S. EVANS (Analyst, 1929, 54, 23—28).—5 g. of sample are dissolved in 30 c.c. of 1:3 sulphuric acid and 15 c.c. of concentrated nitric acid, and evaporated to fuming point. After boiling, the residue is dissolved in 150 c.c. of water, 150 c.c. of hydrochloric acid (*d* 1.18) and 10 g. of sodium hypophosphite are added, and the mixture is boiled for 10 min. If a brownish-black precipitate due to arsenic appears, the liquid is boiled for another 20 min., and the precipitate coagulated by 20 c.c. of benzene and filtered off. If a tin bronze is being tested, 10 g. of oxalic acid are dissolved in the hydrochloric acid solution before adding the hypophosphite. In the absence of any appreciable amount of arsenic, the Reinsch test is at once proceeded with. A flat spiral of electrolytic copper foil is cleaned and then boiled in the solution for 2 hrs., quickly rinsed in water, and placed without delay in a small beaker, covered with distilled water, and about 1 g. of sodium peroxide added, and after 5 min. the beaker is warmed until the coil becomes darkened with oxide. The antimony should now have been completely removed from solution together with any bismuth and arsenic and a little copper. The coil, after rinsing, is immersed in dilute sulphuric acid, which removes the oxide and shows up any antimony which may have escaped stripping. The antimony solution is treated with a rapid current of hydrogen sulphide for 15 sec., and after a short while the precipitated copper and bismuth sulphides are washed with 1% ammonium nitrate solution, and 5—6 c.c. of concentrated sulphuric acid are added to the filtrate, which is evaporated to fuming point after addition of a few drops of nitric acid. The solution of antimony is diluted with 15 c.c. of water, heated to b.p., cooled, and the antimony determined colorimetrically. 10 c.c. of 1% gum arabic, 5 c.c. of 20% potassium iodide, 1 c.c. of 10% aqueous pyridine, 1 c.c. of dilute solution of sulphur dioxide, and 60 c.c. of cold 1:3 sulphuric acid are consecutively added to a 10 c.c. Nessler tube, followed by the antimony solution. Standard antimony solution (0.0001 g. Sb per c.c., 0.2764 g. of tartar emetic in 1 litre of 10% sulphuric acid) is run into another glass containing the same reagents, except that 80 c.c. of sulphuric acid instead of 60 c.c. are used, until the colours match. The concentration of the acid used in the quantitative separation of the antimony on copper in the presence of cuprous chloride is of great importance.

D. G. HEWER.

Lead-tin-cadmium as a substitute for lead-tin wiping solder. E. E. SCHUMACHER and E. J. BASCH (Ind. Eng. Chem., 1929, 21, 16—19).—The replacement of part of the tin in solder by cadmium would lower costs, not by a difference in the intrinsic value of the two metals, but because it allows the use of a higher proportion of lead. As judged by the working qualities, porosity of joints, and tensile strength, certain of the ternary alloys, though within narrow limits of composition, produced joints as satisfactory as those from the standard alloy (62% Pb, 38% Sn). The most satisfactory composition was 68% Pb, 23% Sn, and 9% Cd, and increase to the extent of even 1% in lead or cadmium showed markedly in an adverse direction. C. A. KING.

Physical properties of dental materials (gold alloys and accessory materials). R. L. COLEMAN (Bur. Stand. J. Res., 1928, 1, 867—938).—The physical properties of dental gold alloys, in the wrought and cast states, investments, and pattern waxes were investigated. 18 wrought alloys of commercial manufacture for use as wire, clasps, and plate were selected. They vary widely in composition, but the chief constituents are gold, silver, copper, and palladium or platinum, with small amounts of zinc, tin, nickel, manganese, and iron. The fusion temperature for wires varies from 895° to 1000°, and for plate from 950° to 1400°. The alloys are softened by heating to 700° and quenching in water, or hardened by cooling from 450° to 250° during 30 min., and are more resistant to corrosion in the hardened state. Static flexure, Brinell hardness, tensile, and alternating stress test results are given for all the alloys. The best physical properties were shown by an alloy containing Ag 5.1%, Au 64.5%, Pt 17.6%, Pd 2.1%, Cu 10.4%, and Zn 0.8%, which had an ultimate strength of 153,000 lb./in.² and elongation of 7.5% on 8 in. The fatigue-resistance is greatly increased by hardening. 35 commercial alloys representative of casting golds and solders were investigated, and showed the same variation in composition as the wrought alloys. The casting alloys melt at 900—1000°, and the solders at 750—800°. The alloys show no serious change in composition on remelting 3 or 4 times. Castings showed porosity due to occluded gas and localised shrinkage. The former can be eliminated by using vacuum-melted alloys, and the latter by using a short, wide sprue. Gold foil fillings were harder than an inlay cast of 24-carat gold, and nearly as hard as 22-carat gold. The net casting shrinkage is unaffected by the pouring temperature, the average value being 1.25%. The thermal expansion of 5 inlay waxes was determined. The shrinkage on cooling from 40° to 25° was 0.75%. 11 investment materials were analysed and dimensional changes caused by setting and heating measured. They contained calcined gypsum 20—55%, silica 80—45%, and expanded on setting up to 0.5%. In general, an expansion of 0.2—0.3% is obtained by heating to 300°. Data on compressive strength and porosity of investments are given. Accurate casting to dimensions can be achieved by suitable selection and manipulation of alloy, wax, and investment.

C. J. SMITHELLS.

System ferrous oxide-silica. HERTY and FITTERER.—See VII. **Electrodeposition of copper.** JULIARD and LEDRUT.—See XI.

PATENTS.

Metallurgical furnace. D. CUSHING (B.P. 303,042, 26.9.27).—The base of a cupola furnace is hinged and movable at will, the body having an annular sealing rim. The refractory lining is tongued and grooved or otherwise ribbed to retain its position in the flanged base, and is inclined down to the tapping spout. C. A. KING.

Operation of blast furnaces. A. E. WHITE. From S. G. ALLEN (B.P. 303,206, 30.9.26).—The blast to a furnace is oxygenated to a degree that would cause excessive smelting capacity as compared with the shaft

reduction capacity, and the ratio of ore to coke is increased, additional heat and reducing gases being supplied above the mantle in the form of oil, gas, or pulverised fuel. C. A. KING.

Treatment of ores with gases in rotary furnaces. C. P. DEBUCH (B.P. 303,072 and 303,096, 27.7.27).—(A) In a rotary tubular furnace heated by a number of gas ports distributed along the length of the furnace, the wall is increased gradually in thickness from the main reaction zone. Particular zones may be defined by internal rings, and the speed of movement of the charge is regulated by the number and disposition of prongs which also act as protectors against ore falling into the gas ports. (B) When roasting weak ores, fuel is introduced at various points along the furnace; the fuel may consist of the finely-divided ore. C. A. KING.

Furnace for treating ores. F. M. SIMONDS (U.S.P. 1,693,702, 4.12.28. Appl., 26.10.26).—The hearth is formed in sections each of which consists of a floor and a perforated confining wall at one end forming an angle with the floor. M. E. NOTTAGE.

Treatment of iron ores. T. P. CAMPBELL (U.S.P. 1,696,188, 25.12.28. Appl., 19.5.27).—The comminuted ore is brought into a spongy state by reduction, and after being partially reoxidised is then passed over a magnetic separator. H. ROYAL-DAWSON.

Manufacture of steel. F. C. LANGENBERG and M. A. GROSSMANN (U.S.P. 1,695,594, 18.12.28. Appl., 1.6.27).—A non-hot-short steel contains <0.05% C and <0.03% O. H. ROYAL-DAWSON.

Heat treatment of steel. LE B. W. KINNEY and G. H. BIEMAN, ASSTS. to WHITE MOTOR CO. (U.S.P. 1,695,922, 18.12.28. Appl., 27.9.28).—A composition consisting entirely of kieselguhr, water, and borax serves to prevent decarbonisation during heat-treatment of the metal. H. ROYAL-DAWSON.

Carburisation of metals. G. W. HEGEL and G. R. BROPHY, ASSTS. to GEN. ELECTRIC CO. (U.S.P. 1,696,603, 25.12.28. Appl., 26.8.26).—A carburising liquid containing bone oil is used. H. ROYAL-DAWSON.

Slag and gas eliminator for molten steel. F. H. MOYER (U.S.P. 1,690,748, 6.11.28. Appl., 24.7.26).—The steel is tapped from the furnace into a ladle in the shape of an inverted, truncated cone divided by a baffle parallel to the side of the furnace and extending nearly to the bottom of the cone. The purified metal overflows from the far side of the cone into a larger receptacle for transfer to the moulds. A. R. POWELL.

Hardening of iron, mild steel, and iron alloys. H. LINDHORST (B.P. 302,740, 28.9.27).—Cementation of iron is effected by heating it at 720–820° in a closed chamber together with a small quantity of powder out of contact with the metal and which evolves carbon dioxide and other gaseous hydrocarbons. C. A. KING.

Pickling of iron and steel. V. BERTLEFF (B.P. 293,701, 9.7.28. Austr., 9.7.27).—To the bath of usual pickling liquor a protective agent is added consisting of sulphonated products of coal-tar oil, made by heating the latter with not more than an equal weight of con-

centrated sulphuric acid until complete solubility in water has been attained; or salts, e.g., those of calcium or sodium, or the purified sulphonated acids prepared from such sulphonated oils may be used. M. E. NOTTAGE.

Zirconium-treated iron-chromium alloy. F. M. BECKET, ASSR. to ELECTRO METALLURGICAL CO. (U.S.P. 1,689,276, 30.10.28. Appl., 8.5.26).—Addition of small quantities of zirconium, added as silicon-zirconium alloy, to iron-chromium alloys containing 10–60% Cr greatly increases the ductility and renders them more easily rollable. E.g., a low-carbon steel with 9–30% Cr, less than 3% Si, and up to 0.5% Mn is treated with zirconium sufficient to leave 0.02% in the alloy. A. R. POWELL.

Manufacture of metal articles and alloys therefor. W. H. HATFIELD and H. GREEN (B.P. 302,812, 31.12.27).—A steel alloy resistant to corrosion and to the staining effects of foodstuffs contains 11.5–13% Ni, 11.5–13% Cr, and not more than 0.2% C. The alloy is rendered ductile for cold-working by heating it at 950–1090°, preferably 1000–1050°, and cooling it rapidly at first and then at a slower rate. C. A. KING.

Preparation of the surfaces of iron, steel, and copper articles for nickel-plating. W. A. BURFORD (G.P. 451,620, 7.10.26).—The articles are pickled anodically in a bath containing 45–55% of sulphuric acid, using a current density of 10 amp./dm.² A higher current density may be employed if nitric acid is added to the bath, an extra 5 amp./dm.² being used for every 2% of nitric acid. The articles are rinsed after treatment, dipped into concentrated nitric acid, and plated directly. A. R. POWELL.

Copper alloys. METALLBANK U. METALLURGISCHE GES. A.-G. (B.P. 288,314, 4.4.28. Ger., 9.4.27. Addn. to B.P. 286,616; B., 1928, 898).—The workability and capability of being strengthened of these alloys can be considerably increased if the iron content be replaced wholly or partially by cadmium, tin, or manganese. Before being shaped the alloys are rolled hot, annealed above 600°, quenched to room temperature, and mechanically worked to a cross-section slightly greater than the final; the metal pieces are then re-annealed either at 300–600° and cooled slowly, or at temperatures above 600°, rapidly cooled to 300–400°, and afterwards slowly cooled. M. E. NOTTAGE.

Erratum.—B., 1928, 898, col. 2, line 21 from bottom, for “286,615” read “286,616.”

Leading-in wires for evacuated [glass] containers. J. A. YUNCK (U.S.P. 1,695,791, 18.12.28. Appl., 6.8.27).—A sheath of silver alloy composed principally of silver is welded to a core composed of approx. 40% Ni and 60% Fe. J. S. G. THOMAS.

Manufacture of bodies [e.g., wire] from metals [e.g., tungsten] having a high m.p. N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 284,990, 24.1.28. Holl., 7.2.27. Addn. to B.P. 200,879; B., 1923, 934A).—In addition to the volatile metal compound, the atmosphere in which the metal crystal is heated also contains nitrogen; preferably a current of nitrogen is caused to flow along the heated metal crystal. M. E. NOTTAGE.

Treatment of ores (B.P. 277,660).—See II. **Reduction of barium minerals** (U.S.P. 1,685,772).—See VII. **Annealing furnaces** (B.P. 273,320). **Heat-treatment and forging furnaces** (B.P. 303,222).—See XI.

XI.—ELECTROTECHNICS.

Effect of different electrolytes on the electro-deposition of copper. A. JULIARD and J. LEDRUT (Bull. Soc. chim. Belg., 1928, 37, 377—384).—Economy of time and electrical energy in the deposition of copper is effected by the addition of small amounts of potassium chlorate or perchlorate to an acid solution of copper sulphate. At 20° the presence of 1% of potassium chlorate results in 75% increase in the current for a given voltage, and reduces the time and electrical energy required for the deposition of a given amount of copper by 30% and 50%, respectively. The deposition of copper in the presence of sulphites, persulphates, permanganates, chromates, dichromates, nitrites, fluorides, and alkaline tartrates is not quantitative, there being either wastage of electrical energy or incomplete deposition. F. G. TRYHORN.

Detection of coal tar in mouldings for cable parts. E. KINDSCHER and P. LEDERER (Chem.-Ztg., 1928, 52, 1014).—10 g. of the moulding—an insulating, fusible material used for covering the ends and joints of cables—is heated for $\frac{1}{4}$ hr. with 40 c.c. of *N*-sodium hydroxide solution under a reflux. A positive reaction of a small portion of the filtrate with benzenediazonium chloride may indicate the presence not only of coal tar, but also of oxidation products of colophony. The bulk of the solution is then treated with dilute sulphuric acid, and the separated acids and phenolic substances, after extraction with ether and removal of the solvent, are heated with 5—10 c.c. of 10% sodium hydroxide solution until evolution of carbon dioxide ceases, when the carboxylic acids from colophony are destroyed. The liquid is then extracted with ether, the solvent is removed, and the residue is dissolved in sodium hydroxide solution, and tested with benzenediazonium chloride. F. R. ENNOS.

Potentiometric and conductometric analysis. CALLAN and HORROBIN.—See I. **Reduction of azo dyes.** HUBBUCH and LOWY.—See IV. **B.p. of electrolytic caustic solutions.** MONRAD and BADGER.—See VII.

PATENTS.

Electric furnace. C. B. FOLEY, Assr. to C. B. FOLEY, INC. (U.S.P. 1,694,536, 11.12.28. Appl., 13.3.20. Renewed, 14.4.27).—A tapping device, with means for closing it, is located above the surface of the melt, when the furnace is in its working position. Below the normal level of the melt is a gas inlet. F. G. CLARKE.

Electric furnace. A. N. OTIS and C. L. IPSEN, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,696,728, 25.12.28. Appl., 10.9.25).—A heating resistor is hung in a series of depending loops from parallel bars extending across the furnace chamber, and having their ends secured in opposite walls. J. S. G. THOMAS.

[Operation of] **electric annealing furnaces of the resistance type.** A.-G. BROWN, BOVERI & CIE. (B.P.

273,320, 23.6.27. Ger., 23.6.26).—Continuous current is supplied to the furnace at least until a desired temperature is attained, and is then automatically regulated so that, after the maximum permissible surface temperature of the material to be treated is reached, the energy supply is periodically reduced so as to be always equal to the energy loss from the furnace, together with that soaking into the material at that temperature.

J. S. G. THOMAS.

Electric furnaces for heat treatment and forging.

A. IMBERY (B.P. 303,222, 10.10.27).—Non-metallic heating elements, in the form of rods, are arranged in a refractory chamber near tubes extending through the chamber between the heating elements and receiving parts of the bars or rods to be heated. Electrical connexion with the heating elements is made by metal alloy terminals of low resistance. J. S. G. THOMAS.

Induction electric furnace. High-frequency induction furnace. E. F. NORTHRUP, Assr. to AJAX ELECTROTHERMIC CORP. (U.S.P. 1,694,791—2, 11.12.28. Appl., [A] 14.2.25, [B] 29.11.24. Renewed, [B], 28.7.28).—(A) In inductive heating free from inter-threading by transformer iron, using alternating currents within the range of frequency of generation by rotary, multipolar generators, the current path is confined to a single layer of turns about the charge, and the relation of the frequency to the diameter of the charge is such that the induced electromagnetic energy does not penetrate appreciably beyond the axis of the charge. (B) The loss of power in the inductor in apparatus free from transformer iron is rendered practically independent of the size of the inductor by providing a single inductor layer of greater conductor depth than thickness, and having a depth bearing radially a definite proportion to the diameter of the charge. L. A. COLES.

[Seal for heater-leads of] **electric furnaces.** BRIT. THOMSON-HOUSTON CO., LTD., Assees. of R. B. PRINDLE (B.P. 282,741, 12.12.27. U.S., 29.12.26).—Each opening in the furnace chamber for the heater-leads is provided with a glass sleeve surmounted by a metallic conducting cap to which the lead is connected. The sleeve is surrounded by a rigidly mounted protective member spaced apart from the glass and carrying the conductor from each conducting cap. J. S. G. THOMAS.

Catalytic agent in storage battery. D. W. DAVIS, Assr. to A. L. NEW (U.S.P. 1,694,530, 11.12.28. Appl., 5.8.27).—Gases evolved from the electrolyte are caused to combine by means of a catalyst which is placed in such positions that at least one portion is unsubmerged when the battery is tilted or inverted. F. G. CLARKE.

Production of storage-battery separator elements. N. FALLEK (U.S.P. 1,690,573, 6.11.28. Appl., 4.5.25).—A pulp of vegetable fibre and water is boiled, washed to remove dirt and impurities, and subjected to the direct action of steam. After boiling with caustic soda solution, it is washed to remove all traces of soda, mixed with a small quantity of pulverised rubber and sodium silicate, formed into sheets, and allowed to dry. F. R. ENNOS.

Manufacture of cathodes for electric discharge tubes. VEREIN. GLÜHLAMPEN U. ELEKTRICITÄTS A.-G.

(B.P. 289,762, 18.11.27. Austr., 30.4.27).—An alloy of an alkaline-earth metal, *e.g.*, a barium-magnesium alloy, is decomposed *in vacuo* and partially or completely vaporised so that the vapour of the alkaline-earth metal is deposited on the outer surface of the core of the cathode which contains, at least on its surface, a substance which combines with the alkaline-earth metal to form an alloy or oxide. J. S. G. THOMAS.

Manufacture of cathodes for electric discharge tubes. M-O. VALVE Co., LTD., and J. W. RYDE (B.P. 302,503, 23.12.27).—A highly electropositive metal, *e.g.*, barium, is deposited upon a support, *e.g.*, tungsten, which has been exposed to cyanogen. J. S. G. THOMAS.

Manufacture of cathodes for discharge devices. ALLGEM. ELEKTRICITÄTS.-GES. (B.P. 286,650, 6.3.28. Ger., 7.3.27. Addn. to B.P. 268,815; B., 1928, 646).—A paste consisting of a metal oxide in a viscous fluid, *e.g.*, paraffin oil, sugar, and collodion solutions, is applied to an oxide base which is heated in a reducing atmosphere to a temperature at which the metal oxide is reduced to metal which is sintered upon the base. J. S. G. THOMAS.

Electron-discharge device. K. H. STARK, ASSR. to SPERRY GYROSCOPE Co. (U.S.P. 1,695,746, 18.12.28. Appl., 13.5.19).—The device comprises a gas-tight container, an anode, and a cathode element formed of a photo-electric metallic coating sputtered upon a glass member, a discharge-controlling device, and a spark-gap for causing the cathode to emit electrons. J. S. G. THOMAS.

Electron-emitting element [coating]. L. McCULLOCH, ASSR. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,695,899, 18.12.28. Appl., 27.9.22).—A conductor coated with an aqueous solution of one or more compounds of at least one metal of the alkaline-earth group is heated in carbon dioxide to ignition temperature. J. S. G. THOMAS.

Alloy base for oxide-coated cathodes. G. W. HALLOCK, ASSR. to WESTINGHOUSE LAMP Co. (U.S.P. 1,695,845, 18.12.28. Appl., 27.10.27).—The cathode core consists of a nickel-silicon alloy. H. ROYAL-DAWSON.

Activation of [tungsten] filaments. G. D. O'NEILL, ASSR. to WESTINGHOUSE LAMP Co. (U.S.P. 1,695,819, 18.12.28. Appl., 1.4.27).—Thoriated tungsten filaments are activated by intermittently incandescing them in an atmosphere containing carbon. H. ROYAL-DAWSON.

Protection from oxidation of caps consisting of brass or similar alloys, such as bronze, for electric incandescence lamps etc. GEN. ELECTRIC Co., LTD., ASSEES. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜH-LAMPEN M.B.H. (B.P. 289,440, 25.4.28. Ger., 27.4.27).—The caps are heated at 500–600° in a reducing atmosphere between the process of cleaning by pickling and the setting of the cement. M. E. NOTTAGE.

Centrifugal apparatus for recovery of vapours from air drawn off from chromium-[plating] baths. W. G. POETZSCH (B.P. 298,226, 13.9.28. Ger.,

6.10.27).—The air current is caused to pass through one or more systems of vanes arranged in the exhaust, and to rotate either by the air current itself or mechanically. J. S. G. THOMAS.

[Plates for] secondary or storage batteries. CHLORIDE ELECTRICAL STORAGE Co., LTD. From ELECTRIC STORAGE BATTERY Co. (B.P. 302,472, 21.11.27).

Argon mercury discharge tubes. RAINBOW LIGHT, INC., and R. R. MACHLETT (B.P. 293,683, 23.9.27. U.S., 9.7.27).—See U.S.P. 1,680,271; B., 1928, 760.

Luminous electric discharge tube of high candle power. RAINBOW LIGHT, INC., ASSEES. of R. R. MACHLETT (B.P. 293,682, 23.9.27. U.S., 9.7.27).

Furnace regulation (B.P. 283,903).—See I. Carbonisation of marine algæ (B.P. 284,583). **Manufacture of hydrocarbons** (B.P. 301,949).—See II. **Preparation of articles for nickel-plating** (G.P. 451,620). **Leading-in wires** (U.S.P. 1,695,791).—See X. **Electrodeposition of rubber** (B.P. 303,214).—See XIV. **Pasteurisation of milk** (U.S.P. 1,695,300).—See XIX.

XII.—FATS; OILS; WAXES.

Formation of glycerides and their isomerides. I. T. L. GARNER (J.S.C.I., 1928, 47, 278–279 t).—The effect of various gases and reduced pressure on the preparation of glyceride from oleic acid was found to be largely mechanical with the exception that sulphur dioxide gave the isomeride trielaidin. The action of sulphur dioxide is briefly discussed and the synthesis of trielaidin with this gas is shown to be reversible. The m.p. of the trielaidin, 39°, was somewhat higher than that found by previous investigators.

Identification of fats and mixtures of fats. I. B. LUSTIG and G. BOTSTIBER (Biochem. Z., 1928, 202, 81–90).—Illustrations show the difference in microscopic appearance of cocoa butter, hardened train oil, palm-kernel fat, beef tallow, etc. when a drop of the melted fat is pressed between microscope slides and cooled in ice. Tables show for a variety of such fats the different degree and colour of fluorescence, together with its variation in different solvents, and, according to the manner of preparation of the fat, also the amount, iodine value, and m.p. of the solid fatty acid fraction, together with the normal variations of these in different samples of the fats. P. W. CLUTTERBUCK.

Oil seeds from British Guiana. [Crabwood seed oil; awarra palm oil.] (Bull. Imp. Inst., 1928, 26, 411–416). I. Crabwood seeds and oil (*Casapa guianensis*, Aubl.) have been examined. The kernels, which form 70.7% of the seeds and contain 6.4% of moisture, yield 55% of a cream-coloured oil, partly liquid and partly solid, having an intensely bitter taste. Oils extracted in British Guiana and at the Imperial Institute had, respectively, d_{20}^{20} 0.8689, 0.8572, n_D^{20} 1.4590, 1.4560, solidifying point of fatty acids 37.8°, 35.8°, acid value 36.9, 75.8, saponif. value 198.5, 197.4, iodine value (Hübl, 17 hrs.) 57.3, 64.5, unsaponifiable matter 1.1%, 0.7%. The oil is suitable only for soap making and possibly as a germicide. The residual cake

is useless as a feeding-stuff on account of the bitter taste.

II. The oil (45.5%) of awarra palm fruits (*Astrocaryum*, sp.), extracted by light petroleum from the pericarp, was orange-red and partly solid at 15.5°, with a slight odour resembling that of palm oil. The kernels yielded 36.3% of a cream-coloured hard fat with a faint odour of coconut oil. The oils from the pericarp and kernel, respectively, had d_{15}^{20} 0.8573, 0.8660, m.p. —, 30.8°, n_D^{20} 1.458, 1.4505, acid value 38.4, 0.4, saponif. value 195.8, 241.9, iodine value (Hübl, 17 hrs.) 68.0, 14.6, unsaponifiable matter 0.5%, 0.5%, soluble volatile acids 0.4, 2.4, insoluble volatile acids 0.2, 6.8, solidifying point of fatty acids 36.7°, 27.0°. The residual meal contains less protein than palm-kernel cake.

E. LEWKOWITSCH.

New oil-seed from Brazil [Arara nuts] (Bull. Imp. Inst., 1928, 26, 416—418).—Arara nuts (*Joannessia heveoides*) contained 44.8% of kernel which, on extraction with light petroleum, yielded 58.6% of a pale yellow liquid oil with a pleasant nutty odour having d_{15}^{20} 0.9239, n_D^{20} 1.467, acid value 2.1, saponif. value 188.5, iodine value (Hübl, 17 hrs.) 129.8, unsaponifiable matter 0.48%. A film of oil exposed on glass dried in 11 days (linseed oil, 8 days). The cream-coloured residual meal had a faintly bitter taste, contained substances of an alkaloidal nature, and gave on analysis: moisture 8.0%, crude proteins 47.4%, fat 0.7%, starch etc. (by diff.) 25.1%, crude fibre 6.5%, ash 12.3%, nutrient ratio 1:0.56, food units 145. Both meal and oil may possess toxic properties; the oil has semi-drying properties, and is soluble for soap-making.

E. LEWKOWITSCH.

The Wijs method as the standard for iodine absorption. J. J. A. WIJS (Analyst, 1929, 54, 12—14, and Chim. et Ind., 1928, 20, 1043—1044).—The criticism of Schmidt-Nielsen that substitution as well as addition takes place in the Wijs method for determining iodine absorption is refuted, the break required by Schmidt-Nielsen as showing a cessation of iodine absorption being in fact present. Further, Weiser and Donath's failure to obtain theoretical values with the Wijs reagent is shown to be due to the fact that their solution contained free iodine trichloride owing to 7.2 instead of 10.2 g. of iodine being used for 9.4 g. of iodine trichloride. The reagent should be made by dissolving about 9 g. of iodine trichloride in 1 litre of acetic acid of at least 99% concentration, or in a mixture of 300 c.c. of carbon tetrachloride and 700 c.c. of acetic acid. In exactly 5 c.c. of this solution the halogen content is determined, the bulk of the solution treated with 10 g. of pulverised iodine, and when nearly all is dissolved the halogen content is again determined on 5 c.c., and as soon as this is one half the previous value the solution is filtered into a stoppered bottle. No oxidisable matter must be present in the acetic acid or carbon tetrachloride, and the high coefficient of expansion by heat must be allowed for. Not more than 30% of the halogen present should be absorbed by the fat or oil.

D. G. HEWER.

Composition of Italian beeswax. D. CORTESE (Atti II Cong. Naz. Chim. pura appl., 1926, 1347—1352; Chem. Zentr., 1928, ii, 678).—Values are: d 0.9597—

0.9680, m.p. 63—64°, f.p. 60—61°, refractive index (Zeiss butyrefractometer, 70°) 27—30 (calc. at 40° 42.9—45.9), acid value 19.03—20.92, saponif. value 92—96.2, ester value 72.20—77.13, iodine value 8.0—10.3.

A. A. ELDRIDGE.

Emulsions of fatty substances and of hydrocarbons and their industrial applications. L. MEUNIER (Chim. et Ind., 1929, 21, 3—19).

Transformation of fatty acids. STADNIKOV and others. **Water-miscible mineral oil preparations.** HART.—See II. **Dental materials.** COLEMAN.—See X.

PATENTS.

Manufacture of fatty acid and soap derived from mineral oil. Manufacture of mineral oil derivatives. G. ALLEMAN, Assr. to SUN OIL CO. (U.S.P. 1,694,461—2, 11.12.28. Appl., [A] 18.7.25, [B] 26.3.27).—(A) A mixture of fatty acids derived from mineral oil is claimed, the mixture having m.p. below —30°, being insoluble in water, practically free from mineral oil and petroleum resins, and readily converted into soaps resembling those of animal and vegetable origin, the soaps being miscible in all proportions with mineral oil and water and capable of forming homogenous emulsions with mineral oil and water together. (B) A water-soluble soap practically free from mineral oil and containing a reduced petroleum resin content is separated from an emulsion of soap, petroleum resin, and mineral oil, and is subsequently converted into a soap insoluble in water by double decomposition with a suitable metal salt.

L. A. COLES.

Removal or elimination of fatty acids, resins, and bitter and mucous substances from oils and fats. K. F. WILHELM (B.P. 289,801, 24.1.28. Ger., 2.5.27. Addn. to B.P. 226,767; B., 1925, 411).—To the solvent used in the process of the prior patent is added an equal amount of another solvent, which will dissolve fat, but which will not combine with the de-acidifying liquid, e.g., trichloroethylene, methylene chloride, or benzene. The reagent is completed by the addition of 1% of Glauber's salts, calculated on the weight of the crude oil to be treated.

E. HOLMES.

Treatment of cashew nut-shell and allied oils. HARVEL CORP., INC., Assees. of M. T. HARVEY (B.P. 275,574, 23.6.27. U.S., 6.8.26).—Non-drying cashew nut-shell oil, consisting mainly of anacardic acid and cardol, may be converted into a drying oil by interaction with copper, nickel, monel metal, lead, magnesium, calcium, or manganese, if necessary at elevated temperatures and pressures, followed by the addition of another vegetable oil such as linseed oil. Copper itself is the most effective reagent, although the oxides, hydroxides, carbonates, oleates, or stearates of any of these metals may be used. The resulting products are of use as impregnating and coating materials for waterproofing and insulating purposes.

E. HOLMES.

[Bar-forming device for] soap-making. L. H. NELLES (B.P. 299,960, 21.9.27).

Lubricating oils (B.P. 295,230).—See II. **Degreasing raw wool** (B.P. 300,941). **Compositions from cellulose derivatives** (U.S.P. 1,690,515).—See V.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Ageing processes of paints. A. V. BLOM (Farben-Ztg., 1929, 34, 892—895).—The ageing of paints is discussed with special reference to changes in colloid systems. Outside influences affecting a paint film at early stages of its life control its subsequent behaviour. Under unfavourable conditions of application etc. a paint very quickly enters the "danger zone" in which it is readily vulnerable to circumstances that would not affect the same paint applied satisfactorily. The chief properties, deterioration in which connotes "ageing," are elasticity, hardness, and adhesion. An accelerated test is detailed wherein dry paint films on suitable metal strips are tested in an extensometer. The film is observed under a lens and the experiment is stopped at the first appearance of cracks, when the extension of the strip is recorded. The particular form of cracking is noted and the film is rated by reference to a graded series of "standard cracking types," photomicrographs illustrating which are discussed. The "degree of cracking" and the maximum extension of the film must both be taken into consideration, since both adhesion and elasticity are concerned in the test. Hardness is measured by scratching tests carried out on the same films. Accelerated ageing is accomplished by subjecting the films to continuous cooling, intermittent cooling in dry and moist atmospheres, continuous heating and intermittent heating. The conclusions to be drawn from extensometer tests on these "aged" films at elevated, low, and ordinary temperatures are indicated.

S. S. WOOLF.

Behaviour of white pigments [and paints] under ultra-violet light. C. P. VAN HOEK (Farben-Ztg., 1929, 34, 833—834, 895—896, 951—956, 1006—1009).—Mainly a digest of the literature on the subject. The destructive influence of ultra-violet rays on paints is discussed with reference to the absorbing and transmitting powers of the various white pigments.

S. S. WOOLF.

Photometric determination of the colour of various lamp-blacks. L. HOCK (Kautschuk, 1928, 4, 266—268).—There is a close relationship between the specific surface, i.e., the total surface exposed by the particles in unit weight, of different finely-divided forms of amorphous carbon and their blackness. The specific surface was determined by the heat of wetting with benzene. Blackness was determined by mixing with 100 pts. of lithopone in a medium, preferably rubber, and measuring the intensity of the light reflected from a smooth surface by means of a polarisation photometer or a leucometer.

D. F. TWISS.

Viscosity of cellulose acetate. DESCHIENS.—See V. **Selenium-red in rubber.** DITMAR.—See XIV.

PATENTS.

[Manufacture of] ink. B. KALISCHER (B.P. 303,200, 30.9.27).—An ink which penetrates almost immediately into ordinary paper contains water and/or glycerol or glycol together with 2—10% of an ester of a polyhydric alcohol, e.g., glycol or glycerol acetate. Gelatin, dextrin, gum arabic, etc. may also be added to vary the fluidity of the liquid.

L. A. COLES.

Uninflammable nitrocellulose lacquers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,390, 15.9.27).—Alkylene chlorides are introduced into nitrocellulose lacquers in amount not less than that of the nitrocellulose solvents already present.

S. S. WOOLF.

Manufacture of plastic compositions, lacquers, filling or priming compositions, etc. from nitrocellulose. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,961, 22.9.27).—Plasticising agents which do not volatilise and thereby render the products permanently elastic, comprising phthalic acid esters having one carboxyl group esterified with the radical of an aromatic or hydroaromatic alcohol and the other with the radical of an aliphatic or aromatic alcohol, e.g., *n*-propyl benzyl phthalate, cyclohexyl benzyl phthalate, isoamyl cyclohexyl phthalate, etc., are used in the manufacture of the compositions.

L. A. COLES.

Refining of wood rosin. G. E. JENKS, Assr. to HERCULES POWDER CO. (U.S.P. 1,694,179, 4.12.28. Appl., 15.5.23).—Wood rosin is externally heated *in vacuo* while superheated steam is injected into the body of the rosin. A portion of the distillate is condensed at not less than 174°.

S. S. WOOLF.

Manufacture of condensation products from phenols and aldehydes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 302,609, 14.7.27).—Phenols are condensed with formaldehyde etc. in presence of strong aliphatic or alicyclic bases, e.g., *n*-butylamine, dicyclohexylamine, β -hydroxyethylcyclohexylamine, di-(β -hydroxyethyl)butylamine.

C. HOLLINS.

[Phenol-formaldehyde] condensation product. E. S. HOLE (B.P. 302,098, 20.6.27).—Water-soluble phenol-formaldehyde condensation products are obtained by boiling the reagents in the presence of a small quantity of an alkaline condensing agent (preferably an alkali hydroxide) until the requisite viscosity is attained, when water is added, the mixture is cooled, and a further quantity of water containing a suitable salt of chromic acid is added. The incorporation of alcohol and/or ammonia into the boiling mixture opposes any tendency to precipitation of the final water-soluble product.

S. S. WOOLF.

Manufacture of condensation products of urea and formaldehyde. I. G. FARBENIND. A.-G. (B.P. 278,698, 27.9.27. Ger., 8.10.26. Addn. to B.P. 258,289; B., 1928, 376).—The duration of the heating necessary to harden the condensation products is reduced by the addition, during or after evaporation, of substances giving acids during the heating (e.g., magnesium chloride, formic esters, formamide).

B. FULLMAN.

Production of artificial masses. I. G. FARBENIND. A.-G. (B.P. 282,635, 29.10.27. Ger., 23.12.26).—Soluble formaldehyde-carbamide (etc.) products are mixed with fillers, such as wood-meal, lignin, etc., which have been impregnated with carbamide, thio-carbamide, phenol, etc., or non-sticky compounds of these with formaldehyde etc. (excluding soluble form-

aldehyde-carbamide products). The mixture is dried and hardened in the press. C. HOLLINS.

Composition from cellulose derivatives (U.S.P. 1,690,515).—See V. **Cashew nut-shell oil etc.** (B.P. 275,574).—See XII. **Perishable goods** (B.P. 302,447).—See XIX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Latices of *Euphorbia tirucalli*, *E. candelabro*, and *E. abyssinica*. E. DE'CONNO, S. CAPALBI, and L. FRUITIER (Annali Chim. Appl., 1928, 18, 540–549).—The latices of these plants, in the order named, which grow profusely in Erythrea, have the following percentage compositions: water 46.75, 30.05, 49.60; gum 7.25, 8.30, 8.44; euphorboresene 25.50, 40.50, 28.39; euphorbone 9.25, 10.20, 10.23; euphorbinic acid traces in each case; fat trace, —, 1.03; insoluble residue (inorganic) 7.40, 7.30, 0.29; (organic) 2.70, 2.50, 1.73. In *E. tirucalli* the gummy matter is perfectly elastic and resembles caoutchouc in its physical properties, the euphorboresene is soft and pale yellow, and the euphorbone has m.p. 112–113°; in the other two species the gummy matter is only slightly elastic and resembles Para rubber, the euphorboresene is solid and dissolves in carbon disulphide and acetic acid, and the euphorbone has m.p. 114–115°.

D. H. POPE.

Evaluation of anti-oxidants [for rubber]. T. L. GARNER (India-Rubber J., 1929, 77, 31).—The relative effect of various anti-oxidants is influenced considerably by the type of mixture used; for general comparative purposes the composition rubber 100, sulphur 3.5, zinc oxide 3, diphenylguanidine 1, and anti-oxidant 0.5—I is recommended. The protective effect of anti-oxidants is much more marked with over-vulcanised rubber. Most anti-oxidants cause discoloration of white articles, and their presence may lead to staining of black goods.

D. F. TWISS.

Selenium-red [in rubber]. R. DITMAR (Gummi-Ztg., 1929, 43, 759–760).—Selenium-red forms a colloidal dispersion in rubber and increases the plasticity of the mixing. It has strong pigmenting action for hard or soft rubber vulcanised by the hot process, but is discoloured on vulcanisation with sulphur chloride. Its colour is not affected by organic accelerators and anti-oxidants.

D. F. TWISS.

Causes of variation in plasticity [of rubber]. REPORT FROM Ceylon Rubber Research Committee (India-Rubber J., 1929, 77, 32–36).—The addition of sodium bisulphite to latex before coagulation leads to a marked loss of plasticity in the finished rubber; the plasticity is also notably reduced by prolongation of the period between coagulation and crêpeing. Dilution of the latex before coagulation has little influence on the plasticity, but excessive machining of the coagulum on the crêpeing rolls increases plasticity, as also does drying the crêpe rubber in hot air. D. F. TWISS.

Influence of zinc oxide on the coefficient of vulcanisation [of rubber]. S. A. BRAZIER and L. A. RIDGWAY (J.S.C.I., 1928, 47, 351–356 T, 368 T).—The

effect of the combination of sulphur with zinc oxide on the coefficient of vulcanisation of various rubber mixings containing organic accelerators and compounding ingredients has been studied for different temperatures and periods of vulcanisation. No general rules can be deduced from the results obtained, as, although an appreciable amount of zinc sulphide is formed in the presence of organic accelerators in common use in the rubber industry, the amount is dependent on a number of variables. The extent of zinc sulphide formation is also shown to affect appreciably analytical work where specifications limiting the coefficient of vulcanisation are concerned. The paper includes preliminary results of experiments using cadmium oxide, where a similar effect is noted.

Effect of temperature on the stress-strain properties of vulcanised rubber. A. A. SOMERVILLE and W. H. COPE (Trans. Inst. Rubber Ind., 1928, 4, 263–286, and Kautschuk, 1928, 4, 271–272).—In view of the spontaneous heating of some rubber goods, e.g., tyres, during service, fuller investigation of the alteration in physical properties with temperature change is desirable. Tests are now described with ring test pieces at temperatures from 0° to 100°; the characteristics examined include the stress-strain curve, fatigue, permanent set, and adhesion, the variable factors examined in addition to temperature including time of vulcanisation, proportion of sulphur and accelerator, and presence of an anti-oxidant. D. F. TWISS.

Analysis and classification of regenerates [reclaimed rubber]. E. LINDMAYER (Kautschuk, 1928, 4, 278–280).—Determination of the proportion of caoutchouc hydrocarbon in reclaimed rubber is effected by extracting a comminuted sample successively with acetone, aqueous potassium hydroxide, and concentrated hydrochloric acid, and determining the carbon and hydrogen content of the dry, purified residue by a combustion method using lead chromate. From the hydrogen found, the proportion of rubber C_5H_8 is estimated, any carbon in excess of the requirement for this purpose being regarded as present as carbon black or lamp-black. Mineral rubber is estimated by dissolving the chloroform extract of the material in carbon tetrachloride and comparing the colour with that of a solution of "mineral rubber" of known concentration in the same solvent. On an assumption that the reclaiming process is dependent on a conversion of vulcanised rubber, thus: $(C_5H_8)_6 \cdot S \cdot (C_5H_8)_6 \rightarrow (C_5H_8)_3 \cdot S \cdot (C_5H_8)_3 + (C_5H_8)_6$, the latter formulæ represent the portions of the product respectively insoluble and soluble in chloroform; the limit of the reclaiming process will therefore be represented by a conversion of about 48% of the vulcanised caoutchouc into a condition soluble in chloroform.

D. F. TWISS.

PATENTS.

Electrodeposition of rubber. S. O. COWPER-COLES (B.P. 303,214, 3.10.27).—In the electrodeposition of rubber, inclusion of gas bubbles in the anode deposit is prevented by employing a cathode or cathodes bearing perforations which permit the escape of bubbles to the

side remote from the anode and its rubber deposit. The current may be intermittent, the cathode or cathodes may be rotatable, and the anode or anodes may be immersed only for intermittent periods. D. F. TWISS.

Production of rubber coatings. K.D.P., LTD. (B.P. 277,375, 12.9.27. Ger., 10.9.26).—Latex, if desired in admixture with filling materials such as lamp-black, pigments, sulphur, etc., is sprayed on the base to be coated, *e.g.*, metal, wood, stone, leather, etc., provision being made for evaporation of the water by using a hot, gaseous medium for the atomising process. Rubber layers so produced on different bases may, if desired, be united by compression and vulcanisation.

D. F. TWISS.

Accelerator of vulcanisation [of rubber]. GOOD-YEAR TIRE & RUBBER CO., ASSESS. OF L. B. SEBRELL (B.P. 278,689, 19.9.27. U.S., 8.10.26).—Vulcanisation is accelerated by a reaction product of a mercaptan and an amine other than guanidine; compounds of mercaptobenzthiazole with an aliphatic amine, *e.g.*, a dialkylamine, are indicated. [Stat. ref.]

D. F. TWISS.

Recovery of rubber. W. H. YEANDLE, ASST. TO INTER-CONTINENTAL RUBBER CO. (U.S.P. 1,695,676, 18.12.28. Appl., 13.12.27).—Vegetable matter containing rubber enclosed within the plant cells is permeated with steam, the pressure being then increased and suddenly reduced. The material being thereby disintegrated and the cells disrupted, the rubber is then separated.

D. F. TWISS.

Jelutong products and their production. E. C. R. MARKS. FROM BEECH NUT PACKING CO. (B.P. 302,850, 29.3.28).—Oxidation of jelutong is prevented by introduction of an ionisable anti-oxidant, *e.g.*, ammonium phosphate, tartrate, acetate, etc., or sodium phosphate or phosphoric acid. The jelutong may either be heated in an aqueous solution of the anti-oxidant, or the aqueous solution may be mixed into the molten jelutong.

D. F. TWISS.

XV.—LEATHER; GLUE.

Determination of tannin [in beers and worts]. HARTONG.—See XVIII.

PATENTS.

Tanning. P. PAVLOVITSCH (B.P. 302,408, 20.9.27).—Unhaird hides are plumped with a tanning solution (*e.g.*, quebracho liquor, *d* 1.075), which may be at 30–37°, and have p_H 6–12 (preferably obtained by adding sodium hydroxide), according to the tanning material used. After complete penetration of the hides (3–10 days) they are transferred to tan liquors of p_H 2–5. [Stat. ref.]

D. WOODROFFE.

Moulding of casein under pressure. P. HAESSLER (B.P. 302,545, 21.5.28).—Powdered casein, subjected in the pan of a spray-casting machine to a pressure of, *e.g.*, 150–400 atm. at about 90–110°, is forced through a sieve and thence through an injection nozzle into a mould, where it is exposed to high pressure for a short time.

L. A. COLES.

Tanning agent (B.P. 272,541).—See XXIII.

XVI.—AGRICULTURE.

Catalytic properties of soils. K. SCHARRER (Z. Pflanz. Düng., 1928, 12A, 323–329).—The published literature of the catalytic properties of soils toward hydrogen peroxide and iodides is summarised and discussed, and the essential soil factors concerned are differentiated in the two cases.

A. G. POLLARD.

Properties of heavy alkaline soils containing different exchangeable bases. A. F. JOSEPH and H. B. OAKLEY (J. Agric. Sci., 1929, 19, 121–131).—Clays saturated with different bases exhibit very divergent physical and chemical properties. The commonly accepted clay-like properties are shown most definitely by lithium, sodium, and magnesium clays. The plasticity of a soil bears no relationship to the proportion of very fine material (remaining suspended in a 10 cm. column for 14 days) it contains. In base-exchange properties potassium and sodium clays resemble each other much more than calcium clay, but differ considerably in plasticity and permeability. Comparison of the absorption of calcium, sodium, and potassium by percolation with solutions containing two-base mixtures showed calcium and potassium to be absorbed in equivalent amounts, whilst only one sixth of the equivalent amount of sodium was absorbed.

A. G. POLLARD.

Dispersion and mechanical analysis of heavy alkaline soils. A. F. JOSEPH and O. W. SNOW (J. Agric. Sci., 1929, 19, 106–120).—Examination of soil fractions, of smaller ranges of mean diameter than the customary "clay" fraction, shows that after a single dispersive treatment the fractionation is affected by the nature of the bases with which the soil is saturated. The proportion of very fine particles ($< 0.5 \mu$) cannot be correlated with other important soil properties, and gives no measure of the colloidal properties of soil when the pipette method of analysis is used. Other conditions being similar, "sodium soils" show better dispersion than "ammonium soils." In no case examined was the "clay" fraction influenced quantitatively by pre-treatment with hydrogen peroxide. In the decantation process using sodium carbonate for dispersion, pre-treatment with acid did not affect the number of decantations required nor the proportion of clay determined. It is impossible to disperse soils in a single treatment as required for the pipette method, and for this process sodium carbonate gives better dispersion than ammonia. In Sudan soils decantation methods with sodium carbonate are recommended, with the omission of the hydrogen peroxide-hydrochloric acid treatment.

A. G. POLLARD.

Soil examination by means of conductivity measurements. W. BENADE (Z. Pflanz. Düng., 1928, 12A, 293–309).—Electrical conductivity measurements of aqueous suspensions of soils during the growth of seedlings mark the period of the intake of nutrients by remaining practically constant. Control soils without seedlings show a steady rise in conductivity with time. This increased conductivity is accompanied by a change of soil reaction towards the alkaline side. Conductivity of soil suspensions is influenced by the length of the dis-

solution period, atmospheric carbon dioxide, and the activity of micro-organisms. Soils which have been once extracted with water, filtered, and resuspended in water show an increased conductivity. The new value is characteristic for each soil, and is relatively greater for clays than for sandy soils. Mathematical relationships are obtained to express changes in conductivity in soils with continuous leaching. Soils from which practically all soluble salts have been leached show a general parallelism between conductivity and humus content. A. G. POLLARD.

Liming as a factor in the amelioration of deteriorated tropical soils. P. E. TURNER (J. Agric. Sci., 1929, 19, 83—89).—Liming trials with acid soils showed that finely-ground limestone was more effective than slaked lime as an ameliorant. Large single dressings were more quickly responded to than smaller annual dressings, there being little penetration of lime below the depth of cultivation. A satisfactory dressing must render the soil neutral in reaction and raise the degree of saturation to 80%. A. G. POLLARD.

Lime requirement of soils. S. GOY (Z. Pflanz. Düng., 1928, 12A, 317—318).—A brief outline of the chemical methods of examining acid soils, and their significance. A. G. POLLARD.

Rapid electrometric method for measuring "lime requirements" of soils. F. HARDY and A. H. LEWIS (J. Agric. Sci., 1929, 19, 17—25).—The soil sample (10 g.), is mixed with 40 c.c. of 0.2 *M*-calcium chloride solution (neutralised to p_H 7.0 with bromothymol-blue) and titrated with 0.03*N*-lime water in successive portions of 5 c.c. with shaking for 3 min. between each addition until p_H 7.0 is passed. From a curve showing the changes of p_H with lime added the exact amount of lime required to produce p_H 7.0 is determined. Comparison with the Hutchinson-MacLennan method (B., 1915, 565) shows the new method to be more rapid and reliable. A. G. POLLARD.

Determination of harmful soil acidity. B. TACKE and T. ARND (Z. Pflanz. Düng., 1928, 12A, 362—390).—Of the total soil acidity, the active and the base-exchange acidity are injurious to plants. The different sensitivities of various plant species to acidity cannot be fully described in terms of soil acidity alone. It is probable that the neutral point in respect of plant physiology is the same as the chemical neutral point. For the effective determination of injurious acidity, soils should be exhaustively extracted with neutral salt solutions by percolation. A shorter and rapid process consists in shaking soil for 1 hr. with *N*-potassium chloride and calcium carbonate. The carbon dioxide evolved is a measure of the injurious soil acidity. Both organic and mineral soils limed on this basis have a reaction of p_H 7. A. G. POLLARD.

Comparison of methods for determining the saturation capacity of soils. P. A. KUTSCHINSKY (Z. Pflanz. Düng., 1928, 12A, 392—411).—The methods of Hissink, Kappen, and of Gedroiz for determining the exchangeable bases in soils (*S*) give results in close agreement. For carbonate-free soils, Kappen's method

is the most rapid. The determination of the value ($T - S$) (Hissink) is unsatisfactory in soils of high p_H value, or of considerable buffer capacity, owing to the turbidity of the liquid to be titrated. Duplicate tests, however, show moderately close agreement. The value ($T - S$) can be calculated as $6.5 \times$ hydrolytic acidity (Kappen). The lime required to bring an acid soil to a definite p_H value can also be calculated from the hydrolytic acidity (Kappen), using the factors 3 for p_H 7.0, 4 for p_H 7.5, 5 for p_H 8.0, and 6.5 for p_H 8.5. Of the methods for determining the degree of saturation (*V*), those of Kappen and Gehring show closest agreement with each other and with field-trial results. Hissink's method gives low results and indicates only 50% of the saturation of alkali soils. Gehring's method is advantageous in that the final saturation of the soil with bases takes place at practically the same soil reaction as obtains under natural conditions. Kappen's method is quicker and more convenient in practice.

A. G. POLLARD.

Comparison between the culture methods of Mitscherlich and Wiessmann [for determining nutrient values of soils]. W. U. BEHRENS (Z. Pflanz. Düng., 1928, 12A, 412—415).—The basis of the methods is examined mathematically from the point of view of the dependence of the constants of the growth curves on the nature of the soils and experimental conditions. In Wiessmann's method the constants refer to one experiment only; those of Mitscherlich are of general application. The accuracy of the results of the pure sand cultures in Wiessmann's method is of vital importance, since they control the corrected values for plant growth in the experimental sand-soil cultures. A. G. POLLARD.

Influence of the carbon : nitrogen ratios of organic material on the mineralisation of nitrogen. H. L. JENSEN (J. Agric. Sci., 1929, 19, 71—82).—The relationship between the extent of nitrification of various organic materials in acid and alkaline soils to the carbon : nitrogen ratios is examined. In acid soil accumulation of nitrate and ammonia took place only when pea-pod meal with a C : N ratio of 13.3 was used. In alkaline soil materials with a C : N ratio less than 26 : 1 were not nitrified. Nitrification was the more rapid as the ratio narrowed below the limiting value. The residual unnitrifiable nitrogen amounted to 1.5—2.2% of the original material, and was greater with highly nitrogenous matter. All materials examined increased the α -humus content of the soil, but to varying extents. Alkaline soil favoured the production of α -humus from plant residues, but not from farmyard manure. The decomposition of straw, sweet clover, lupin, and farmyard manure is associated with their lignin contents, since they increased the nitrogen and methoxyl content of the humus, but the dried mycelium of *Polyporus* contained a "humic acid" fraction of high nitrogen content, but without methoxyl, which persisted in the soil. A. G. POLLARD.

Determination and characterisation of organic substances in soil. U. SPRINGER (Z. Pflanz. Düng., 1928, 12A, 309—317).—Existing methods for examining soil organic matter are discussed. Oxidation processes for determining organic matter may give fictitious

values if the usual relationship, $\text{humus} = 1.724 \times \text{carbon}$, is adopted. From 40 to 70% of the soil organic matter is humified, the balance being mainly cellulose, lignin, and pectin, with carbon contents of 44, 56, and 40%, respectively, and the humus content is more accurately regarded as $2 \times \text{carbon}$. Wet combustion methods for this type of analysis are recommended. In alkaline extraction processes for determining humified material values depend on concentration and temperature of the solvent and the period of extraction. Non-humified material may be partially extracted. Use of ammonia extraction leads to difficulty in the separation of deflocculated clay from the extract. 50% pyridine is not a specific solvent for humus. Where alkaline-extracted humus is determined by oxidation with permanganate, results vary somewhat with the concentration of permanganates used and the period of boiling. The ratio humus-carbon:total carbon is valuable in the characterisation of soil types; and in the examination of soil profiles the alkali-extractable carbon expressed as a percentage of the total carbon content yields information as to the process of soil formation.

A. G. POLLARD.

Relationship between stability of soil structure and its colloid and sand content. A. T. TIULIN (Rep. Dept. Agric. Chem., Perm, 1928, 2, 22; Bied. Zentr., 1929, 58, 7—8).—The comparison of colloid and sand contents in several soils with their stability of structure showed that the stability was related directly to the colloid and inversely to the sand content. Four tschernozem and two podsol soils were compared, stability of structure being measured directly by washing samples with a constant pressure of water, and colloid content estimated from adsorption capacity. The product of adsorption capacity and sand content varied as between the two types of soil, but was roughly constant within a type. Sodium acetate treatment was found to increase the amount of colloid and decrease sand; ferrous sulphate had the opposite effect.

H. L. RICHARDSON.

The second approximation of the theory of growth factors. E. A. MITSCHERLICH (Z. Pflanz. Düng., 1928, 12A, 273—282).—The theory of growth factors is extended to include the depression of crop yields resulting from the supply of nutrient in amounts greater than the optimum. Experimental work can be expressed in mathematical form. The growth-depression factor is influenced by the nature of the deficient nutrients and of the soil, the buffer capacity and water-retaining capacity of the soil, and by climatic conditions. Much of the experimental evidence of the opponents of the constancy of the growth factor is considered unsatisfactory owing to lack of consideration of the "depression factors."

A. G. POLLARD.

Sparingly soluble phosphates of physiological importance to plants. E. UNGERER (Z. Pflanz. Düng., 1928, 12A, 349—362).—The amount of phosphoric acid extracted from the tertiary phosphates $\text{Mg}_3(\text{PO}_4)_2 \cdot 22\text{H}_2\text{O}$, $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$, $\text{Ca}_3(\text{PO}_4)_2$, AlPO_4 , and FePO_4 is affected by the reaction of the extracting solution and the presence in it of electrolytes and univalent permutites. Neutral salts decrease the solubility

of iron and aluminium phosphate and increase the p_{H} value of the solution. Clay and potassium- and ammonium-permutites adsorb calcium and magnesium ions from the above phosphates, and an equivalent amount of phosphate appears in solution. In soils, iron and aluminium phosphates in the presence of neutral salts are involved in base-exchange activities, and titratable hydrated alumina is found in solution. The value of these phosphates as plant nutrients depends on the water and chalk content, and reaction of the soil. When the soil moisture rises to 90% of the maximum capacity, aluminium phosphate has a greater nutrient value than calcium monohydrogen phosphate. Soil reactions more acid than p_{H} 5 decrease the nutrient values of iron and aluminium phosphate.

A. G. POLLARD.

Physiology of urea in the higher plants. G. KLEIN (Z. Pflanz. Düng., 1928, 12A, 390—391).—Urea is present in plants in maximum amounts in seedlings, young buds, and leaves, and in active tissue. In old leaves and seeds none occurs. Urea in a nutrient medium for plants is partially decomposed outside the plant roots and partially absorbed and decomposed within the plant tissue, and the plant may be poisoned by ammonia produced. Urea in young plant tissue is to a large extent combined with aldehydes.

A. G. POLLARD.

The washing-out of chloride [added to soils]. H. J. WITTEVEEN (Landb. Tijdschr., 1928, 40, 528; Bied. Zentr., 1929, 58, 9—10).—In a field experiment in the wet winter of 1926—27, chloride added as heavy applications of 20% potash salts was soon washed out. Chloride was added at the rate of nearly 500 and 600 kg. per hectare, and samples were taken every four weeks to a depth of 20 cm. After two months only 100—130 kg. of chloride per hectare remained in this depth, and further washing out took place in the following months. It was concluded that by seed time most of the added chloride would be out of reach of all but deep-rooted plants, and thus would not be harmful.

H. L. RICHARDSON.

Influence of irregularities in the level of soil on its fertility. N. N. KOURTIAKOFF (Compt. rend., 1929, 188, 189—191).—Owing probably to the fact that carbonates are washed to a lower depth below depressions in a field than in the higher parts, the yield of barley is least in the portions of lowest level.

G. A. C. GOUGH.

Fertilising with iodine. E. G. DOERELL (Z. Pflanz. Düng., 1928, 12A, 344—349).—Application of iodine (as potassium iodide) at the rate of 3.2—4.3 kg. per hectare increased the yield of hops, but higher proportions (5.4 kg. per hectare) caused considerable crop reduction. In no case was the increased value of the crop as great as the cost of iodide used. The iodine present in Chilean saltpetre and in superphosphate is sufficient to show a small but definite effect in the crop. Iodides improve the colour of the hops and decrease the iodine number and tannin content.

A. G. POLLARD.

Field experiments with Zeotokol. E. BLANCK (J. Landw., 1928, 76, 317—326).—"Zeotokol" (powdered dolerite) applied at 5 and 10 cwt./acre had no good effect on growth or yield with sugar beet, nor any residual

value for oats in the following year. This result, agreeing with earlier pot experiments of the author, contradicts Popp's conclusion (1926) that Zeotokol improved yield and soil condition. His results are criticised on the basis of experimental error, whilst any beneficial action on soil condition could be secured more cheaply with other materials. H. L. RICHARDSON.

Sterilisation of green fodder by liquids. C. BRAHM [with G. ANDRESEN and R. PRILLWITZ] (Forts. Landw., 1928, 3, 769; Bied. Zentr., 1929, 58, 24—26).—Fingerling's suggestion for preserving fodder materials with dilute hydrochloric acid was tested, but it gave a product no better than ordinary silage at a greater cost. Analyses of the grass used, before and after treatment, and of the press liquors over a period of nine months, showed that the hydrochloric acid tended to disappear during storage, and was not able to prevent the occurrence of other processes than lactic acid fermentation, nor prevent the disappearance of lactic acid.

H. L. RICHARDSON.

Influence of clay on plant growth. E. BLANCK and H. KEESE (J. Landw., 1928, 76, 309—316).—In pot experiments with oats the addition of increasing proportions of clay to sand at first improved and then injured the plants. Quartz sand and a "pure clay" containing only traces of bases were used in mixtures of 10:0, 9:1, 8:2, 6:4, and 4:6, with the addition of a complete fertiliser. Height at harvest and dry weight were both greatest with the 9:1 mixture, about the same with either 10:0 or 8:2, and greatly reduced with 6:4 and 4:6 mixtures. Subsequently reaction measurements showed that whereas the sand was neutral the clay was acid (p_H 4.23); the injurious action of the clay was therefore attributed to its acid nature. The difference in the properties and action of individual clays was emphasised.

H. L. RICHARDSON.

Effect of sodium silicate in increasing the yield of barley. R. A. FISHER (J. Agric. Sci., 1929, 19, 132—139).—Sodium silicate increases the yield of barley most markedly in the absence of superphosphate dressings. Although the phosphate content of the barley plant is not notably increased by silicate manuring, the total phosphate removed by the crop is greatly increased and the silicate content of the plant ash is also increased. The higher yield of grain and straw following the use of sodium silicate can be quantitatively ascribed to the increased availability of soil phosphate under these conditions.

A. G. POLLARD.

A type of bacteria abundant in productive soils, but apparently lacking in certain soils of low productivity. H. J. CONN (N.Y. State Agric. Exp. Stat. Bull. No. 138, 1928, 26 pp.).—The organism (*Bacterium globiformis*), which consists of a rod in young cultures and a coccus in older ones, will not grow in certain unproductive soils which are acid, even after liming, unless addition is made of certain simple nitrogen and carbon sources, e.g., amino-acids, ammonium salts, or nitrates, and carbohydrates, polyhydric alcohols, or various organic acids even as simple as acetic acid. A further study of the nutritive requirements of the organism is being made in order to throw light on the unproductiveness of these soils.

F. R. ENNOS.

Lime status of soil in relation to an insect pest of sugar cane. P. E. TURNER (J. Agric. Sci., 1929, 19, 26—35).—The blight of sugar cane caused by the "frog-hopper" (*Monecphora [Tomaspis] saccharina*, Dist.) is associated with the acidity and calcium reserve of soils. The ratio exchangeable calcium : (clay + fine silt) averaged 0.28—0.63 in unblighted soils and 0.11—0.25 in blighted areas.

A. G. POLLARD.

Seed treatment for black-leg disease of crucifers. E. E. CLAYTON (N.Y. State Agric. Exp. Stat. Bull. No. 137, 1928, 58 pp.).—The efficiency of various methods of seed treatment for black-leg on cabbage and brussels sprouts were tested both in the greenhouse and in field plats. Dust disinfectants, e.g., copper carbonate, or the organic mercury disinfectants Semesan and Uspulun, or liquid chemical treatments, e.g., mercuric chloride, Germisan, etc., are considered unsatisfactory on account of their uncertain action or of their liability to injure the seed. The most effective control of the disease was obtained by hot water. Treatments of 25 min. at 50°, 10 min. at 55°, and 4½ min. at 60° were about equally effective; the lowest temperature is recommended since the chance of seed injury is least.

F. R. ENNOS.

Tar distillate trials in Kent and West Sussex in 1928. F. V. THEOBALD (J. Pomology, 1928, 7, 199—211).—Data are cited with reference to the effects of various tar distillate washes in freeing the following trees and bushes from certain blights, particularly aphid and capsid bugs: gooseberry, cob-nut, black currant, and apple. In the case of the apple tree, data are also given with reference to the effect of the spraying on the subsequent crop.

E. A. LUNT.

Plant colloids. XXIII. SAMEC.—See XVII. Tobacco products. POPP.—See XX.

PATENTS.

Products from marine algæ (B.P. 284,583).—See II. **Calcium nitrate-ammonium nitrate** (B.P. 301,486). **Calcium cyanamide etc.** (B.P. 279,419).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Plant colloids. XXIII. Soluble starches obtained by oxidation methods. M. SAMEC (Kolloidchem. Beih., 1929, 28, 155—165).—Methods of obtaining soluble starch by oxidation processes involving sodium peroxide, hydrogen peroxide, ammonium persulphate, potassium permanganate, sodium perborate, chlorine, and air in alkaline solution are described. 1% solutions of each of these preparations were made and the following properties were studied: viscosity, electrical conductivity, hydrogen-ion concentration, alkali-binding capacity, osmotic pressure, and behaviour towards a collodion membrane. The viscosity of all the preparations was lower than that of native starch and, although the viscosity of ordinary starch is raised by addition of bases, the soluble starches were unaltered, except for a few cases where a decrease was observed. The electrical conductivity of the soluble starches was about 10 times as great as that of native starch. This result, together

with the increase in potentiometrically active hydrogen ions and alkali-binding capacity, suggests that during oxidation some acidic groups are produced. The degree of dispersion of the soluble starches is higher. The phosphorus content remains unchanged.

E. S. HEDGES.

Effect of heat on tragacanth and its mucilage. L. F. GABEL (J. Amer. Pharm. Assoc., 1928, 17, 1206—1210).—The viscosity of mucilage of tragacanth prepared under different conditions has been examined. A boiling period of 1 or 2 min. produces an initial maximum viscosity which increases very markedly on ageing. A 3 min. boiling period produces a mucilage of high viscosity which, however, is not materially increased on ageing. The viscosity rapidly decreases with prolonged boiling, and previous heating of the powdered tragacanth causes a very high loss in the viscosity of the mucilage prepared from it. Deterioration on heating or drying over calcium chloride is partially due to chemical change, as is evidenced by the development of acidity.

E. H. SHARPLES.

Insect pest of sugar cane. TURNER.—See XVI.

PATENTS.

Saccharification of cellulosic materials. H. SCHOLLER (B.P. 273,317, 23.6.27. Ger., 23.6.26).—Diluted acid is forced through the cellulosic material heated under pressure, whereby the l  vulose solution formed is immediately and continuously removed from the system and cooled to prevent decomposition. The yield is practically quantitative.

W. J. BOYD.

Esters of carbohydrates (B.P. 302,191).—See V.
Gums from tobacco (B.P. 302,863).—See XX.

XVIII.—FERMENTATION INDUSTRIES.

Continuous pasteurisation of black beers. R. SELIGMAN (J. Inst. Brew., 1929, 35, 10—17).—Stouts or black beers are sterilised in a plate heat-exchanger through which they flow at high speed and with great turbulence between walls so close together that at no time is any particle $\frac{3}{8}$ in. distant from a heated surface. In the heating section of the exchanger the beers are raised in 20 sec. to 71  , at which temperature the yeast is instantaneously killed. At this temperature they re-enter the regenerative section in which they are pre-cooled to 40   by unpasteurised stout. After being cooled by water to 15   in the cooling section the beers pass into a tank in which they can be carbonated to the degree required. The use of the plate heat-exchanger is not an alternative to pasteurisation in bottle, as it gives a beer which is sterile when it leaves the machine, but is subject to the ordinary risks of the bottle and the bottling plant.

C. RANKEN.

Determination of tannin [in beers and worts]. B. D. HARTONG (Woch. Brau., 1929, 46, 11—15).—From a boiled aqueous extract of hops, previously extracted with ether, the adsorption of tannin was studied by titration with 0.01*N*-permanganate in presence of indigotin. From the results of titrations of such extracts before and after treatment with varying

quantities of adsorbents the adsorption curves (amount adsorbed per g. of adsorbent against amount unadsorbed per 100 c.c. of extract) were plotted. Norit gave a normal curve, and alumina one suggesting chemical combination, as already described by Wislicenus. Casein, hide powder, and gelatin gave parallel curves of regular form, but indicating relative weakening of adsorptive power from more dilute solutions. The adsorptive action of kaolin, asbestos, and cellulose pulp was negligible. The curves for the five active adsorbents examined were capable of extrapolation and at maximum adsorption met at the zero point, indicating complete adsorption of oxidisable matter. Gravimetric determination of tannin on three hop extracts by Chapman's cinchonine method indicated an equivalent of 45.0 for hop tannin as regards oxidation by permanganate. Tests on beers indicated that norit adsorbs matters other than tannin, but alumina and casein gave curves which on extrapolation showed the same amount of unadsorbable oxidisable matter. The authors therefore assume that these only adsorb tannin, and, since their adsorptive power appears lower in beer and wort, that the tannin in these liquids is adsorbed on the proteins. The tannin from the malt greatly exceeds that from the hops in normal beers, and in a case examined 80% of the tannin present before boiling was still present in the beer after 2½ months' storage. Since the proportion of tannin adsorbed from worts and beers by any definite proportion of casein appears to be constant, to avoid the construction of an adsorption curve for each determination, two proportions only are employed and empirical factors applied to the result. Two 50 c.c. portions of beer or wort are treated with 1 g. and 4 g. of casein, respectively, shaken every 5 min. for 1 hr. and filtered bright. A 5 c.c. sample with 100 c.c. of water and indigotin is titrated with 0.01*N*-permanganate, run in as a steady stream of drops, with constant shaking, till the colour changes. Several titrations are obviously necessary. Under these conditions, and using Hammarsten's caseinum purissimum, the proportions of total tannin adsorbed from worts and beers was 50% and 74%, respectively. By applying these factors the duplicate results so obtained agreed within about 3%.

F. E. DAY.

Cooling and drying of materials [yeast]. E. B. BROWN, ASST. to FLEISCHMANN CO. (U.S.P. 1,694,807, 11.12.28. Appl., 1.6.25).—A current of air at about 15.5   containing about 1.25 grains of moisture per cub. ft. is passed through a thick, comminuted mass of moist yeast at the rate of about 2 cub. ft. per min. per lb. of yeast.

W. J. BOYD.

Retting of flax (B.P. 302,300).—See V.

XIX.—FOODS.

Correlation of ash content of wheat and of flour. R. C. SHERWOOD and C. H. BAILEY (Cereal Chem., 1928, 5, 437—444).—Ash content of straight-grade flour is positively correlated, with a small proportion of exceptions, with the ash content of the wheat; the relation appears to be linear. Variations in weight per bushel do not cause variations in the relation between the ash content of the wheat and that of the flour. The ash

content of a highly refined middlings flour was correlated with the ash content of the wheat in the same degree as was that of the straight-grade flour. The ash content of a flour cannot be used as a measure of the percentage extraction of that flour unless the ash content of the wheat is known.

W. J. BOYD.

Aminodicarboxylic acid fraction in [wheat] gliadin. D. B. JONES and R. WILSON (Cereal Chem., 1928, 5, 473—477).—The greater portion of the glutamic acid was precipitated from the hydrolysate as the hydrochloride, and the remainder of the dicarboxylic acid fraction was obtained as the barium salts insoluble in 70% alcohol. From this glutamic acid was separated as the hydrochloride, aspartic acid as the copper salt, and hydroxyglutamic acid as the silver salt. The following yields were isolated: glutamic acid 43.0%, aspartic acid 0.5%, hydroxyglutamic acid 7.7%. Time of hydrolysis had no significant effect on the proportions isolated.

W. J. BOYD.

Viscosity of flour suspensions. G. VAN DER LEE (Cereal Chem., 1928, 5, 484—485).—Replying to Denham, Watts, and Scott-Blair (*ibid.*, 330), the author maintains his objections (B., 1928, 282) to the application of Einstein's equation for the viscosity of colloidal solutions to the viscosity of flour suspensions.

W. J. BOYD.

Determination of hydrogen-ion concentration of flour-water mixtures. P. HALTON and E. A. FISHER (Cereal Chem., 1928, 5, 445—460).—The electrometric method of determining p_H is much more reliable than the colorimetric method. The quinhydrone electrode gives results in agreement with those obtained by means of the hydrogen electrode if gold or gold-plated electrodes are used. With platinum electrodes errors in p_H of 0.4 may occur. Concordant p_H values are obtainable with centrifuged extracts and with decantates from suspensions, but filtered extracts usually give slightly low, and suspensions high, values. The p_H of flour extracts increases with increasing ratio of flour to water, and very slightly with increasing time of extraction, but the concentration- p_H curves differ widely for different flours. The p_H of flour extracts cannot be correlated with the p_H of the corresponding doughs. The apparent buffer action of a flour is $1\frac{1}{2}$ — $3\frac{1}{2}$ times that of the extract.

W. J. BOYD.

Staling and hydrogen-ion concentration [of bread]. L. P. KARACSONYI (Cereal Chem., 1928, 5, 477—481).—During ordinary keeping of bread at room temperature acidity remains constant or shows some decrease. The acidity of stale bread may increase during further storage owing to the activity of micro-organisms.

W. J. BOYD.

Volume displacement of salt-sugar solutions [for dough mixtures]. E. GREWE (Cereal Chem., 1928, 5, 470—472).—In order to facilitate the addition of the salt and sugar in the form of solution, in the standard baking test (cf. Blish, B., 1928, 463), data are supplied whereby the water contained in given volumes of solutions of different concentration can be ascertained.

W. J. BOYD.

Effect of variation in the method of manufacture on the baking quality of dry skim milk. E. GREWE and G. E. HOLM (Cereal Chem., 1928, 5, 461—469).—Baking tests were carried out on three flours, from hard spring wheat, hard winter wheat, and soft winter wheat, using six samples of dry skim milk prepared by the spray process, but held for 30 min. at 50°, 63°, 73°, 83°, 93°, and 100°, respectively, before drying. The greatest improvement was obtained with the soft winter wheat flour and the least with that from hard winter wheat (cf. Grewe, B., 1928, 798). The use of dry skim milk increases the range of permissible fermentation times. The best baking results were obtained with the samples held at 73°, 83°, 93°, and 100°, which showed little variation among themselves, and the poorest results in all-round properties were obtained with that held at 50°.

W. J. BOYD.

Gasometric determination of carbon dioxide [in baking powders, flours, etc.] by the Chittick method. R. HERTWIG and J. S. HICKS (Cereal Chem., 1928, 5, 482—484).—In the Chittick method (A.O.A.C. Book of Methods, 1925, 305) for the gasometric determination of carbon dioxide in baking powder, self-raising flour, etc., the omission of a correction for the influence of the vapour tension of dilute sulphuric acid on the carbon dioxide volume reading may cause considerable errors.

W. J. BOYD.

Chemistry of sour milk. L. L. VAN SLYKE (N.Y. State Agric. Exp. Stat. Bull. No. 140, 1928, 14 pp.).—Experiments in connexion with the factors involved in the manufacture, ripening, and digestion of cottage cheese, the chemical changes taking place in certain constituents of milk at different stages of souring, and the determination of lactic acid in sour milk are summarised.

F. R. ENNOS.

Alkali number of milk ash of various animals. A. SCHNECK (Milch. Zentr., 1928, 57, 373—379).—The alkali number ($K_2O:Na_2O$ ratio) for the ash of milk of various animals differs according to the type of food normally consumed. In any one class of animal it varies with the stage of lactation and the method of feeding, which probably accounts for the variation in the available figures for cows' milk. Except for the initial and final stages of lactation, the alkali number for cows' milk is more or less constant at 2.5 provided the food contains sufficient soda; further addition of soda in whatever form causes very little change in this ratio.

F. R. ENNOS.

Factors affecting the composition of dates. M. T. FATTAH and W. V. CRUESS (Plant Physiol., 1927, 2, 349—355).—Dates grown in Mesopotamia contained more sugar and less moisture than the same variety grown in California. Differences in sugar content exhibited by different varieties can often be attributed to arrested ripening by drying on the tree. Unripe dates contain much sucrose; except in the Deglet Noor variety, this decreased greatly during ripening. Soluble tannin decreased markedly during ripening under various conditions.

CHEMICAL ABSTRACTS.

PATENTS.

Pasteurisation of milk. J. O. TEMPLETON, Assr. to ELECTROPURE CORP. (U.S.P. 1,695,300, 18.12.28. Appl., 13.3.25.).—In pasteurising milk by passing an electric current through it, a fluid of the same conductivity as milk is first passed between the electrodes until it acquires the required temperature, and then the milk is introduced.

W. J. BOYD.

Preservation of perishable goods. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 302,447, 17.10.27).—Eggs, or other perishable goods, are treated with an aqueous solution of a urea-formaldehyde condensation product, such as dimethylolurea, to which small amounts of organic solvents such as ethyl alcohol, glycol, or glycerol, and/or other preserving agents may be added, and the goods are then dried.

W. J. BOYD.

Kneading machines [for dough]. ARTOFEX ENGINEERING WORKS, LTD. (B.P. 300,960, 10.11.28. Switz., 21.11.27).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Incompatibility of sodium salicylate and sodium bicarbonate mixtures. J. C. KRANTZ, JUN. (J. Amer. Pharm. Assoc., 1928, 17, 1203—1206).—The rapid darkening of mixtures of sodium salicylate and sodium bicarbonate in aqueous solution has been investigated. The reaction is accelerated by oxygen and does not occur with the sodium salts of the *m*- and *p*-hydroxybenzoic acids. Exposure to ultra-violet light or changing the alkali ion does not influence the rate of darkening.

E. H. SHARPLES.

Testing of Liquor cresoli saponatus D.A.B.6. W. MEYER (Chem.-Ztg., 1929, 53, 43).—For the detection of fish oils in lysols a rapid test is proposed: 3 pts. of the lysol, stirred with 7 pts. of boiling water, should develop during 3 min. no smell of fish oils, but should retain the pure odour of cresol, together with the typical "alkaline" odour.

C. HOLLINS.

Determination of chloral in syrup of chloral. P. FLEURY and M. MALMY (J. Pharm. Chim., 1928, [viii], 8, 537—542).—A critical examination of François' method (cf. B., 1928, 243). Both the presence of sugar and the temperature have a considerable influence on the rate of decomposition of chloral by alkalis. If the conditions described by François be strictly followed, chloral can be accurately determined in presence of syrup only if the temperature is above 21°. At lower temperatures decomposition is very slow, but ultimately is complete (cf. Andron, B., 1929, 35).

E. H. SHARPLES.

Commercial civet. C. T. BENNETT and W. M. SEABER (Perf. Ess. Oil Rec., 1929, 20, 14—15).—The analyses of 19 commercial samples of civet are critically compared with previously published figures and standards. Four samples appear to be adulterated with petroleum jelly, and 3 others with butter fat.

E. H. SHARPLES.

Indian ephedras. B. E. READ and C. T. FENG (J. Amer. Pharm. Assoc., 1928, 17, 1189—1192).—The

amounts of ephedrine and ψ -ephedrine in the following ephedras from Northern India have been determined: *E. intermedia* (dry broken stems and unbroken stems), *E. Gerardiana*, *E. sinica*, and *E. equisetina*. The results are compared with other assays and their therapeutical significance is discussed. *E. intermedia*, containing 60—70% of ψ -ephedrine in the total alkaloid, and *E. Gerardiana*, containing 20—30%, are not likely to yield identical physiological results. In the sample of *E. intermedia* the length, weight, and number of nodes or joints has been critically examined.

E. H. SHARPLES.

Chemistry, pharmacology, and therapeutics of *Agave Salmiana*. G. G. COLIN (J. Amer. Pharm. Assoc., 1928, 17, 1182—1188).—A crystalline substance from the juice of *Agave Salmiana* has been examined for its alleged anti-syphilitic properties. Some therapeutic properties have been observed without the use of cathartics or sudorifics, but the results were not conclusive, and the effects on the Wassermann reaction were not consistent.

E. H. SHARPLES.

Examination of β -amino- α -phenylethyl alcohol sulphate. S. M. GORDON (J. Amer. Pharm. Assoc., 1928, 17, 1195—1199).—A crystallographic and chemical examination of β -amino- α -phenylethyl alcohol sulphate, m.p. 250—254°, are described. An aqueous solution, when treated with copper sulphate and sodium hydroxide, yields a purple colour which is discharged by acids, and is insoluble in ether. Benzaldehyde and the corresponding hydrochloride are formed when the base is extracted from alkaline solution with chloroform. On boiling the sulphate with chloroform and alcoholic potassium hydroxide solution a strong odour of carbylamine is produced. In aqueous solution it yields precipitates with phosphotungstic acid, palladium chloride, and with Millon's reagent a yellow precipitate which is unchanged on heating. The compound forms a *picrolonate*, m.p. 200—201° (corr.), and a *monobenzoyl* derivative, m.p. 149.5—150° (corr.). Other published reactions are outlined and a series of standards is suggested.

E. H. SHARPLES.

Odour and constitution among the mustard oils [thiocarbimides]. V. Blending of mustard oils. G. M. DYSON (Perf. Ess. Oil Rec., 1929, 20, 3—5; cf. A., 1928, 283, 514, 748, 1127).—4-Aldehydophenylthiocarbimide ("thiotrope base"), m.p. 32°, has an odour strongly resembling heliotropin, for which it may be substituted without alteration in certain types of floral oils. Examples of blending of both the above and mesitylthiocarbimide, which exercises a softening action in perfumes of the "new-mown-hay" type, are given. Neither compound has any value for perfuming toilet soap base, but *p*-tolylthiocarbimide, either alone or blended, is a useful soap perfume. Rideal-Walker coefficients of some aromatic thiocarbimides have been determined.

E. H. SHARPLES.

Tobacco products poor in nicotine. M. POPP (Z. Pflanz. Düng., 1928, 12A, 334—344).—The nicotine content of tobacco and products is discussed, and the relative proportions of toxic material in tobacco smoke are examined. Fertiliser trials are recorded showing

their effects on the nicotine content of the crop and compared with the market value. A. G. POLLARD.

PATENTS.

Treatment of oils and fats [medicinal paraffin]. E. CANALS (B.P. 302,954, 21.9.27).—Liquid paraffin etc. (e.g., 90 pts.) is mixed with a solution of, e.g., 0.05 pt. of saccharin in 9.95 pts. of olive oil.

L. A. COLES.

Production of narcotics. E. HESSE, Assr. to GEHE & Co. A.-G. (U.S.P. 1,695,656, 18.12.28. Appl., 17.11.27. Ger., 10.3.27).—Substances containing cocaine are treated with adonidine; the product is an anaesthetic containing both drugs.

F. G. CLARKE.

Preparation of tannic acid compositions. R. S. PATERSON (U.S.P. 1,690,175, 6.11.28. Appl., 4.2.26).—Tannic acid is ground with zinc oxide, mixed with glycerol, and after hardening with a little water the mass is dissolved in water and used for urethral injection.

R. BRIGHTMAN.

Extraction of essential principles or gums from tobaccos of all kinds. B. SEFERIADIS (B.P. 302,863, 25.5.28. Fr., 19.3.28).—Tobacco or tobacco residues are treated with acetone at normal or higher temperature and pressure. The gums obtained on évaporation of the solvent are incorporated in tobacco to impart an aroma.

B. FULLMAN.

Destruction of germs in and sterilisation of cigarette tobacco. "UNIVERSELLE" CIGARETTEN-MASCHINEN FABR. J. C. MULLER & Co. (B.P. 294,537, 12.7.28. Ger., 25.7.27).—Air containing ozone is used for the pneumatic conveyance of the cut tobacco to the tobacco-loosening and dust-removing plant. The introduction of injurious substances (such as nitrous oxide, obtained in the production of the ozone) into the air-feed pipe is avoided.

B. FULLMAN.

Cosmetic (B.P. 272,541).—See XXIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photochemistry of silver halides. IV. New method of determining excess silver in unexposed photographic films, and studies on the processes involved in exposure. H. H. SCHMIDT and F. PRETSCHNER (Z. wiss. Phot., 1929, 26, 259—274).—The new method consists in centrifuging a neutral solution. From analyses by this method and by that using dichromate-sulphuric acid, the "silver value" has been split into three components. The first and greatest part is dispersed in the gelatin and can be obtained by centrifuging or by dichromate-sulphuric acid solution without altering the photographic properties of the film. The second part is absorbed in the grain and can be dissolved out by means of dichromate-sulphuric acid. The third part is similar to the second, but is not soluble. This third component is believed to cause sensitivity. The emulsions used, in this case poor in gelatin, had a smaller "silver value" than those rich in gelatin which were used previously (B., 1928, 625). A better method of deter-

mining "free silver" is required before the correlation of "free silver" with sensitivity is proved.

W. E. DOWNEY.

Influence of the nature of the fixing agent on the development after fixation of inverted or solarised photographic plates. H. BELLIOU (Compt. rend., 1929, 188, 167—169; cf. B., 1929, 151).—Development after fixation of inverted or solarised plates resembles that produced by a chemical developer when the fixing agent has strong reducing properties, the evolution of the development being inverted if the latter is acidified. The image produced is attributed to molecules directly reduced by the action of light, and, in cases where the fixing agent has strong powers of reduction, to activated molecules and molecules in their immediate neighbourhood.

J. GRANT.

XXII.—EXPLOSIVES; MATCHES.

Decomposition of explosives. J. P. KOETTNITZ (Z. Elektrochem., 1928, 34, 768—783).—The author's previous view, that an explosive process may be regarded as the evaporation or sublimation of the liquid or solid explosive, with simultaneous, or almost simultaneous, dissociation and the evolution of large quantities of heat, is further developed. On the above assumption, an approximate value for the detonation temperature, T_v , can be calculated from $Q_v/\log_e 10RT_v = n_r(1.75 \log T_v + C_\delta)$, where Q_v is the heat of explosion at constant volume, n_r the number of gas molecules reacting as a result of the detonation, and C_δ is a constant. From this, $n_r = Q_v/(4.58 T_v)(1.75 \log T_v + C_\delta)$. If t_v is the detonation temperature in °C., and n_A the number of atoms at the end of the reaction, then, for mercury fulminate, by calculation, $n_r = 7 = n_A$ at $t_v = 150^\circ$; corresponding values for cyanuric triazide are 15, 193°; for nitrogen tetrasulphide 8, 206°; and for nitroglycerin 80, 195°. Assuming that in the explosion of the following substances $n_r = n_A$, t_v (calc.) is 190° for pentaerythritol tetranitrate, 180° for mannitol hexanitrate, 170° for nitrohexamethylenetetramine (hexogen), 140° for methyl nitrate, and 190° for ethylene glycol dinitrate. In cases of incomplete combustion (i.e., when no water is formed among the reaction gases from a hydrocarbonaceous explosive), it is calculated that for picric acid $n_r = 18 = n_e$ (number of mols. at the end of the reaction) at $t_v = 424^\circ$; corresponding values for trinitrotoluene are: 18, 377°; for diazobenzene nitrate 10, 83°; for guncotton 38, 183°. Considering metallic explosives: for silver oxalate $n_r = 2 = n_g$ (number of gaseous mols. at the end of the reaction) at $t_v = 140^\circ$; the values of n_r and t_v are: for lead trinitroresorcinate 19, 260°; for lead azide 3.5, 337°; for silver acetylde 4, 200°. It is also calculated that for nitrogen trichloride $Q_v = 114.3$, and for ammonium nitrate $n_r = 7 = n_e$ at $t_v = 164^\circ$. The significance of these results is discussed at some length, with special reference to the classification and action of anti-knock materials. A number of experiments are recorded on the explosions of alkali metals with halogenated hydrocarbons. Numerous equations are developed, and a new theory of detonators is outlined, according to which the

detonator is regarded as a system consisting of a very large number of molecular elements or condensers.

L. L. BIRCUMSHAW.

Vaseline for smokeless powders. POGGI.—See II.

PATENTS.

Priming mixture for small-arms ammunition.

H. T. PECK, Assr. to PETERS CARTRIDGE CO. (U.S.P. 1,694,890, 11.12.28. Appl., 27.6.27).—The mixture consists of mercury fulminate, barium nitrate, lead chromate, and lead thiocyanate. H. ROYAL-DAWSON.

Priming compositions (B.P. 302,961). See XIII.

XXIII.—SANITATION; WATER PURIFICATION.

Dissolved oxygen absorption test. III. E. A. COOPER and S. D. NICHOLAS (J.S.C.I., 1928, 47, 320—322 T).—Solutions of sodium permolybdate accelerate the rate of dissolved oxygen absorption by sewage effluents. The oxidation of sugars, glycine, and sometimes ammonium salts in dilute aqueous solution is also accelerated by the permolybdate activator. The increased oxygen uptake is due partly to stimulated bacterial activity, and also to an induced direct chemical oxidation. An improved method for preparing the active solution is described.

Hydrogen sulphide removal and water softening at Beverly Hills, Cal. R. L. DERBY (J. Amer. Water Works' Assoc., 1928, 20, 813—818).—In order to augment the water supply of Beverly Hills, California, a supply of well water was tapped, but the presence of hydrogen sulphide averaging 6—10 p.p.m. and an average hardness of 275 p.p.m. caused many complaints. Difficulty also arose owing to a luxuriant growth of beggiatoa in the settling basins and service pipes. Chlorination was fairly successful in removing the trouble, but the coating of beggiatoa in the mains was so thick that it was impossible for this to be effective at a reasonable distance from the station without having an objectionable quantity present for those nearer to the point of supply. A combination of aeration, alum, and lime precipitation, and chlorination followed by filtration, produced a water having a hardness of 160—170 p.p.m. and free from hydrogen sulphide.

C. JEPSON.

Composition and use of ferric hydroxide as a coagulant [in river water]. E. S. HOPKINS (Ind. Eng. Chem., 1929, 21, 58—60).—Laboratory tests carried out on litre samples of river water indicate that the normal working p_H value 9.4 is the most efficient value for quantitative precipitation of ferric hydroxide at all concentrations, less than 1 p.p.m. remaining in solution. The maximum turbidity removal takes place at p_H 8.4 with the average at 9.4. When 17—170 p.p.m. of ferrous sulphate ($7H_2O$) are used the floc consists entirely of ferric hydroxide, but with higher concentrations, 428 and 1710 p.p.m., definite evidence of adsorption of sulphates is obtained. A definite basic sulphate is not formed under these conditions as occurs with alum.

C. JEPSON.

Chlorine absorption as a substitute for oxygen consumed [in water analysis]. H. HALE, M. SULLI-

VANT, and C. B. DEWITT (J. Amer. Water Works' Assoc., 1928, 20, 847—853).—The oxygen consumed test gives an indication of the carbohydrate but not of the nitrogenous matter present, and is somewhat affected when the chloride content of the sample is high. Chlorine absorption gives a measure of the nitrogenous matter present, and in low concentrations is unaffected by carbohydrates. It is recommended that the test be carried out on 100 c.c. of the sample, heated just to boiling, with a chlorine dose of 2 mg. in the form of chlorine water. After 10 min. the excess chlorine may be determined in the usual way with *o*-tolidine.

C. JEPSON.

Corrosion of iron and steel by brackish waters. FORREST and others.—See X.

PATENTS.

Sewage treatment with the aid of a screen acting like a filter. K. IMHOFF (U.S.P. 1,696,846, 25.12.28. Appl., 18.6.27. Ger., 31.7.26).—Sewage is not allowed to pass through the screen until a mat of sludge has been formed on it. This acts as a fine filter when the sewage is passed through, and the deposited solids are finally removed.

C. JEPSON.

Production of a substance having disinfectant, cosmetic, tanning, or like properties, and suitable for the treatment of textile materials. K. KELETI (B.P. 272,541, 10.6.27. Hung., 12.6.26).—The product comprises a mixture of soap, an aldehyde other than acetaldehyde, and chlorine and/or bromine or substances yielding them, *e.g.*, hypochlorites etc., with or without the addition of a heavy-metal compound, *e.g.*, chromium sulphate, and carbon disulphide. [Stat. ref.]

L. A. COLES.

Distillation of water for feed water make-up. N.V. NECKAR WATERREINIGER MAATSCHAPPIJ, and H. NOORDENDORP (B.P. 301,666, 10.1.28).—Sludge water blown down from the boiler is passed direct into an expansion and pressure-reducing chamber, and the steam derived is conveyed to the make-up evaporator and there used for the distillation of make-up feed water which has been preheated and de-aerated by the sludge water remaining after the pressure reduction.

W. G. CAREY.

Purifying the water used for steam-boiler purposes. A. H. WHITE (U.S.P. 1,693,066, 27.11.28. Appl., 7.12.25).—Sufficient phosphoric acid to form insoluble phosphates with the calcium, magnesium, and iron carbonates present is added to the water together with sodium phosphate to precipitate other salts of these metals, the amounts added being such as to neutralise the water after the carbon dioxide has been removed.

W. G. CAREY.

Removal of easily-soluble salts from boiler feed water. Purification system for boilers. Clearing and purifying water for steam boilers of all kinds. J. OSTERTAG (U.S.P. 1,700,714—6, 29.1.29. Appl., [A, B] 7.7.22, [C] 10.7.22. Poland, [A—C] 8.2.22).—See B.P. 200,263 and 204,352; B., 1923, 905 A, 1195 A.

Water-softening silicate (U.S.P. 1,693,873).—See VII.