

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

NOV. 22, 1929.

### I.—GENERAL; PLANT; MACHINERY.

**Mechanical handling of materials in and about the chemical plant.** II, III. A. K. BURDITT and W. F. SCHAPHORST (*Ind. Eng. Chem.*, 1929, 21, 649—654, 844—850).—Conveyers described include belt conveyers, the drag cable in a trough, scraper, apron, and bucket conveyers, gravity systems such as chutes and roller conveyers, and screw conveyers. Pneumatic conveyers operating by either pressure or suction are suitable for longer travels. C. IRWIN.

**So-called Kick law applied to fine grinding.** A. M. GAUDIN, J. GROSS, and S. R. ZIMMERLEY (*Min. and Met.*, 1929, 10, 447—448).—The Kick law is generally accepted to mean that for each reduction to one half in particle diameter, in unit weight, the same amount of work is required. In an actual crushing test on quartz the product was graded and the energy units supplied by Kick's law were calculated. This gave a relationship between these units and work expended as determined experimentally. By extrapolating with the help of this ratio the energy corresponding to any degree of fineness can be obtained. With finer division the efficiency, *i.e.*, the relation of this figure to the theoretical surface energy of quartz, steadily increases and greatly exceeds 100%. It is therefore inferred that Kick's law is invalid. C. IRWIN.

**Separation of particulate matter [dusts] smaller than screen sizes into graded fractions.** D. E. CUMMINGS (*J. Ind. Hygiene*, 1929, 11, 245—256).—The dust is thoroughly dried and screened to pass 270-mesh; 25 g. of this material are made into a smooth paste with 125 c.c. of 95% alcohol diluted to 250 c.c. with distilled water, and 1 litre of water is added to the suspension, with stirring. After settling for  $\frac{1}{2}$  min. the supernatant suspension is decanted carefully, allowed to settle for 1 min., again decanted, and the suspension settled for 2 min. This process is repeated at intervals each of which is twice as long as the previous one up to 64 min., then at intervals of 1, 2, and 4 days, after which no further deposition takes place. The first sediment is re-treated as before, the suspension being poured into the vessel with the second sediment and so on, treatment being continued until the supernatant liquid is clear in every case. The mean diameters of quartz particles graded in this way are, respectively, 45.3, 34.0, 25.2, 16.7, 12.1, 8.8, 6.3, and 4.2 microns for settling times of  $\frac{1}{2}$ , 1, 2, 4, 8, 16, 32, and 64 min. Stokes' law may be applied to settling quartz particles having a diameter between 430 microns (*i.e.*, about 40-mesh) and 0.5 micron. A chart is given from which can be read the settling times for quartz particles of different sizes. A. R. POWELL.

**Distillation and rectification of complex mixtures.** L. GAY (*Chim. et Ind.*, 1929, 22, 3—18; *cf. A.*, 1927, 1133).—It has been shown that for mixtures of more than four constituents continuous distillation to yield pure products is practically impossible. In such processes as petroleum refining the production of chemically pure compounds is not aimed at. The case of a single column, with source of heat at the bottom and dephlegmator at the top from different portions of which mixtures of different composition are withdrawn, is discussed in detail. Compositions are depicted spatially on a tetrahedron if there are four constituents. It is concluded that it is more economical to withdraw intermediate mixtures as vapour than as liquid. In the case of the dephlegmator, however, it is shown that fractions removed here should be in the liquid state. Discontinuous distillation cannot be treated on the theory developed with strict accuracy, but if the quantity of the mixture in the rectifying column is small compared with that in the still, so that composition changes are slow, conditions are approximately the same. The composition changes during the progress of a distillation are traced. C. IRWIN.

**Measurement of plasticity.** PARMELEE and RUDD.—See VIII. **Gas filter.** COLLES. **Alloy for vacuum chambers.** NICHOLS.—See X.

### PATENTS.

**Jaw crusher.** J. E. KENNEDY (U.S.P. 1,719,333, 2.7.29. Appl., 20.2.28).—Along one side of the rectangular jaw is a tubular portion, which is integral therewith and protrudes at both sides, so as to form trunnions for supporting the jaw in the crusher-frame. F. G. CLARKE.

**Crushing machine for hard materials.** J. IHLE-FELDT, Assr. to G. POLYSIUS (U.S.P. 1,719,979, 9.7.29. Appl., 27.6.27. Ger., 4.2.26).—A pair of vertical grinding rings are resiliently pressed against the rotary crushing ring of a ring-roller crushing mill, whereby it both grinds and crushes. F. G. CLARKE.

**Two-zone pulverising apparatus.** F. N. DANIELS, Assr. to RILEY STOKER CORP. (U.S.P. 1,719,831, 9.7.29. Appl., 12.4.27).—Coarse material enters one end of a drum near its axis, which is horizontal, and is crushed by rotary members against accumulated material at the bottom of the first zone. A second set of rotary members crushes the charge against stationary members in the second zone, the two zones communicating at the upper portion of the periphery only. F. G. CLARKE.

**Drying apparatus.** J. FARASEY (U.S.P. 1,719,603, 2.7.29. Appl., 28.3.28).—A horizontal cylindrical shell rotates upon two peripheral rings, which bear upon two

sets of wheels, one set being at the front and the other some distance from the rear of the shell. The rear wheels are carried by a transverse support located between the shell and the firebox below. The latter is divided by a bridge-wall beneath the transverse support. The front portion of the firebox communicates directly with the shell by means of apertures, and a passageway leads combustion gases from the rear portion round the end of the shell and into the interior. The shell is jacketed between the rings, and also between the rear ring and the end of the shell.

F. G. CLARKE.

**Treatment [drying] of finely-divided solids.** V. S. HENRY, A. WRIGHT, and F. W. YOUNG, Assrs. to FILTRATION ENGINEERS, INC. (U.S.P. 1,726,511, 27.8.29. Appl., 8.6.23. Renewed 7.12.27).—A pasty substance, *e.g.*, a filter cake, in a hopper, is forced down upon an endless conveyer of open-mesh material, which carries it between ironing rollers, and then through a dryer, *e.g.*, of the festoon type. A scraper regulates the quantity of substance applied to the conveyer, and endless bands, pressed on opposite sides of the conveyer, may replace the ironing rollers. After leaving the dryer the substance is detached by means of a beater.

F. G. CLARKE.

**Decohering solid substances.** P. G. M. A. PIGACHE (B.P. 292,965, 27.6.28. Fr., 27.6.27).—Solid substances (colours, catalysts, medicaments, graphite, etc.) are subjected after a preliminary crushing to a slow and continuous or intermittent trituration in the presence of one or more hydrosol colloids, *e.g.*, gelatins, hydro-celluloses, oxycelluloses, albumins, gums, etc., dispersed or expanded in an aqueous medium so as to form a paste sufficiently tacky to prevent water from dripping off during the process. Solid, liquid, or gaseous material capable of facilitating the decohering process and/or preventing putrefaction, *e.g.*, mercuric chloride, acids, glycerin, formaldehyde, ammonia, etc., may also be added, and the products may be diluted with an aqueous or non-aqueous medium or may be dried, preferably after admixture with a substance, *e.g.*, glycerin, which prevents complete desiccation. In examples the preparation of graphitic mineral oil, of hydrosol paints, and of coloured alcohol, oil, or cellulosic paints is described.

L. A. COLES.

**Machine for mixing dry and liquid materials.** G. V. ROBERTS, JUN. (U.S.P. 1,720,573, 9.7.29. Appl., 2.3.29).—The mixer is provided with a vertical screw-conveyer, mounted upon a hollow shaft, which is provided with horizontal discharge pipes disposed just below each turn of the conveyer blades.

F. G. CLARKE.

**Apparatus for mixing.** W. A. GILCHRIST (U.S.P. 1,720,549, 9.7.29. Appl., 19.6.25).—A tank encloses a chamber, open at the top and bottom and not in contact with the inside surface of the tank, and a device, *e.g.*, a propeller, is used to circulate the liquid upwards through staggered baffles in the chamber, and downwards through baffles in the space between the chamber and the tank.

F. G. CLARKE.

**Separation of liquids and solids.** W. B. EDDISON (U.S.P. 1,720,211, 9.7.29. Appl., 14.3.25).—The suspended solids are separated in the form of a filter cake,

and, while still on the filtering surface, are subjected to pressure by a flexible, impervious surface to remove further liquid.

F. G. CLARKE.

**Filtering drum.** J. B. VERNAY (U.S.P. 1,720,156, 9.7.29. Appl., 13.1.28. Ger., 18.1.27).—A filtering drum is divided into independent sectors, comprising a number of individual blocks covered with filtering cloth. Each block has a set of grooves in its outer surface, and a single channel cross-connecting such channels and disposed inwardly of the plane thereof, the channels of any two successive blocks registering endwise. Radial tubes connect distributing gear at the centre of the drum with a channel of each sector.

F. G. CLARKE.

**Liquid-clarifying apparatus.** H. MILLER (U.S.P. 1,720,185, 9.7.29. Appl., 25.4.27).—After issuing through a trap, the liquid is passed in succession through charge, mixing, washing, and precipitating tanks to a filter; the outlet of the precipitating tank is above the floor.

F. G. CLARKE.

**Compressed gas.** M. G. FARRAR, Assf. to CARBIDE & CARBON CHEM. CORP. (U.S.P. 1,726,018, 27.8.29. Appl., 1.5.28).—To prevent water which is condensed in the outlet of a vessel containing gases under pressure from freezing, about 1% of an organic substance which is soluble in water and is expelled with the water vapour, *e.g.*, alcohol or acetone, is introduced into the vessel.

F. G. CLARKE.

**Separation [of gases] by liquefaction.** R. C. TOLMAN, W. L. DE BAUFRE, J. W. DAVIS, and M. H. ROBERTS, Assrs. to S. G. ALLEN (U.S.P. 1,728,947, 24.9.29. Appl., 16.9.22).—During the condensation of a mixture of gases the composition of the mixture of the liquid and vapour is maintained approximately constant until the gas to be separated forms a large proportion of the uncondensed fraction. Final separation is effected by external refrigeration, using part of the condensed gases as a cooling medium.

C. A. KING.

**Extraction of volatile substances (U.S.P. 1,715,313).**—See III.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Comparison of the conditions of occurrence of bituminous coal and petroleum.** E. McK. TAYLOR (J. Inst. Petroleum Tech., 1929, 15, 372—384).

—Examination of the roofs of bituminous coal seams and the cap-rocks of oil sands shows both types of strata to possess identical chemical, physical, and biological properties, and to consist of material that has undergone base exchange with solutions of sodium chloride followed by hydrolysis in fresh water of the sodium clay thus formed. Evidence points to the formation of bituminous coal and petroleum by the same type of decomposition, but from different types of organic matter. Laboratory tests confirm field observation that plant material containing lignocellulose when decomposed in a sodium clay yields materials similar to those in bituminous coal; hence a source of organic matter free from lignocellulose is indicated as the material from which petroleum has been formed,

a condition satisfied by material of estuarine and marine origin.

H. S. GARLICK.

**Determination of best temperature of hydrogenation for bituminous coals.** J. P. AREND (Proc. 2nd Int. Conf. Bit. Coal, 1928, 2, 485—490).—The first deviation of the expansion curve of the heated coal indicates the first appreciable extent of hydrogenation. At the maximal rate of expansion hydrogenation gives the greatest yield of oils and the maximal quantity of light gasoline.

CHEMICAL ABSTRACTS.

**Presence of chlorine in coal.** H. TER MEULEN (Rec. trav. chim., 1929, 48, 938—940).—For 22 samples of coal examined the chlorine content varied from 0.03 to 0.33%. Extraction of the powdered coal with cold 0.2*N*-nitric acid removes inorganic chlorine (0—0.12%); the remainder is probably organic in nature. A Yorkshire coal contained as much as 0.46% Cl (0.19% extractable). Calcination of the extracted coal gives a residue free or almost free from chlorine.

H. BURTON.

**Peat as a source of nitrogen.** Z. V. LOGVINOVA (Trans. Sci. Inst. Fertilisers, Moscow, 1929, No. 56, 1—66).—Moss peat contains more adsorbed ammonia than meadow peat; the upper layers contain more than the lower. The calcium is in the adsorbed state, and is easily replaced by sodium. Moss peats, but not meadow peats, give appreciable amounts of phosphorus when treated with sodium chloride solution.

CHEMICAL ABSTRACTS.

**Significance of the hydrogen content of charcoals.** H. H. LOWRY (J. Physical Chem., 1929, 33, 1332—1342; cf. A., 1924, ii, 393).—Determinations of the hydrogen contents of coal under various conditions show that at a constant temperature, exceeding some temperature characteristic of a given material, the hydrogen content decreases regularly with an increase in the time of treatment. The range investigated varied from 800° to 1500° with times of heating between 0.5 and 141.5 hrs. Two equations of similar form, one being  $H = H_0 e^{-a(T-T_0)^n}$ , where  $H$  is the hydrogen content after heating at  $T^\circ$  for a time  $t$  with a material having an original hydrogen content  $H_0$ ,  $a$  and  $n$  being constants, represent the data for three different coals. The effect of two separate heat treatments is additive and, in all cases, the hydrogen content is more sensitive to a change in temperature than to duration of heating at a particular temperature. In the case of 14 different coals, the hydrogen contents (originally ranging from 2.13 to 4.73%) after heating for 30 min. at 900—1200° lie within a narrow range, and, in general, the greater the hydrogen content of the raw material, the less is the hydrogen content of the remaining charcoal for a specific treatment. Analyses of charcoals prepared between 900° and 1300° in hydrogen, air, and carbon dioxide show that the atmosphere in which a sample of coal is heated does not influence the hydrogen content of the resultant charcoal, indicating that this factor has not the importance in activation processes which is commonly supposed. An accurate method for the determination of hydrogen contents less than 0.2% is described.

L. S. THEOBALD.

**Decomposition of coal into definite organic compounds by oxidation.** O. HORN (Brennstoff-

Chem., 1929, 10, 362—364).—The literature bearing on the oxidation of charcoal and coal with the production of definite organic compounds is summarised.

A. B. MANNING.

**Oxidation of Willstätter's lignin with nitric acid.** O. HORN (Brennstoff-Chem., 1929, 10, 364—365).—By the treatment of Willstätter's lignin with 5*N*-nitric acid oxalic acid was obtained together with an insoluble oxidation product. By further oxidation of the latter with more concentrated acid and finally with fuming acid a small quantity of mellitic acid was obtained.

A. B. MANNING.

**"Wet" purification of coal gas—a review of past and present methods.** L. S. PICKLES (Gas J., 1929, 188, 107—114).—A historical survey of purification processes from 1792 to 1923 is given, and a new method is described in which crude gas passes into an acid liquor and is maintained in an acid atmosphere until cold, when it is treated with alkali. A small setting of two horizontal iron retorts was used, of which the hydraulic main was fitted with special sprays for the acid liquor. The annular condensers were followed by the cyanogen washer, the exhauster, and the iron oxide purifiers. The acid liquor contained 0.75% of sulphuric acid and the alkali cream was made as in the Davis-Neill process, with 5% of excess alkali. The gas and the various liquids were analysed at frequent intervals and full working data are given for an 8-weeks' operation of the plant; the results confirm the predictions made for the process. Noxious constituents of the liquor are considerably reduced, and no important difficulties arise in handling the dilute acid; corrosion due to cyanogen compounds is practically eliminated, and cyanides are more readily recoverable. The recovery of phenol is increased and treatment of effluent in order to render it harmless is far less difficult.

R. H. GRIFFITH.

**Determination of the calorific value and analysis of coal gas.** G. NEUMANN (Arch. Eisenhüttenw., 1929—30, 3, 123—132, 199—203; Stahl u. Eisen, 1929, 49, 1455—1457).—The precautions necessary for obtaining correct results with the Junker calorimeter and the calorimeter are described, and the magnitude of the errors that may be caused by various deviations from standard conditions have been determined. A deviation of  $\pm 5^\circ$  from the standard temperature of the gases passing into and out of the Junker calorimeter causes an error of  $\mp 0.77\%$  in the calorific value. The calorimeter usually gives results which are 2—3% too low. In the analysis of the gas, fuming sulphuric acid is preferable to bromine for the determination of the unsaturated hydrocarbons and phosphorus to pyrogallol for oxygen absorption. Combustion in the Drehschmidt-Winkler platinum capillary avoids the formation of nitrous oxide and gives excellent results for hydrogen and methane.

A. R. POWELL.

**Determination of tar fog in coke-oven gas.** W. KLEMP (Brennstoff-Chem., 1929, 10, 359—361).—Tests have been carried out with the following types of tar-fog filter: (1) tube packed with glass wool, (2) tube packed with cotton wool, (3) tube containing two porous discs of sintered glass, and (4) extraction thimble packed with cotton wool and fitted in a glass container so that

the gas passes successively through the thimble and cotton wool. With (1) the tar fog was not completely retained, whilst to obtain complete retention with (2) it was necessary to pack the tube so tightly that the velocity of the gas current was very greatly decreased. Only tar fogs of low concentration were absorbed completely by (3). The arrangement (4) was the most efficient of those tested. A difficulty arose from the loss of volatile constituents of the tar during drying. A partial correction for this was made by drying in a constant current of air, plotting weight against volume of air passed, and extrapolating back to zero from the constant rate of loss of weight which is eventually reached.

A. B. MANNING.

**Exactitude of measurement of gas-investigation methods.** A. SCHNEIDER (Gas- u. Wasserfach, 1929, 72, 829—831).—The errors involved in measurements with gases are discussed in connexion with calorific value, sp. gr., hydrogen sulphide, ammonia, oxygen, and naphthalene. By the methods recommended 0.1 g. of ammonia in 100 m.<sup>3</sup> of gas, 0.02% of oxygen, and 0.2 g. of naphthalene/100 m.<sup>3</sup> can be determined.

R. H. GRIFFITH.

**Inflammability of mixed gases.** G. W. JONES (U.S. Bur. Mines Tech. Paper No. 450, 1929, 38 pp.).—The limits of inflammability have been determined for mixtures of methane, of hydrogen, and of carbon monoxide with air and nitrogen or air and carbon dioxide. The inflammability of mixtures can be calculated with fair accuracy by means of a simple formula.

C. W. GIBBY.

**Inflammability of mixtures of ethyl alcohol, benzene, furfuraldehyde, and acetone.** G. W. JONES and J. R. KLICK (Ind. Eng. Chem., 1929, 21, 791—793).—The lower inflammable limits found for the solvents when mixed with air and at 125° were: ethyl alcohol 3.85%; benzene 1.53%; furfuraldehyde 2.10%; acetone (pure) 2.92%, (commercial) 3.18%. The results of tests made on mixtures of these four solvents give limits of inflammability (lower) when mixed with air which agreed closely with calculated values obtained by the application of Le Chatelier's law.

H. S. GARLICK.

**Effects of knock-suppressing and knock-inducing substances on the ignition and partial combustion of certain fuels.** R. E. SCHAAD and C. E. BOORD (Ind. Eng. Chem., 1929, 21, 756—762).—Hot-wire ignition curves were determined for *n*-propyl, *n*-butyl, and isoamyl alcohols, toluene, isoamyl acetate, kerosene, ethyl maleate, and ethyl fumarate between the lower and upper limits of inflammability. The fuel-air mixtures investigated were produced by a vapour-pressure method described and shown to be applicable to the continuous and reproducible preparation of such a series of mixtures. The current required by an electrically heated platinum wire for ignition of the most easily ignitable mixture of air and toluene, isoamyl acetate, or kerosene was increased by the addition to the fuel of a knock suppressor such as lead tetraethyl or selenium diethyl. On the contrary, the addition of one of the knock inducers decreased the hot-wire ignition current. The addition to each of the above-

mentioned fuels of either a knock suppressor or a knock inducer had no noticeable effect on the ignition curves obtained by means of direct-current break sparks. Pre-ignition combustion curves of fuels at chosen saturator temperatures were obtained by plotting filament current (in amp.), both increasing and decreasing values, against the corresponding percentages of carbon dioxide as ordinates. No measurable quantity of carbon monoxide was formed in any of these experiments. Very little combustion of toluene took place at filament currents less than that required for ignition, but an abrupt increase took place with ignition. Without exception, the addition of knock suppressors to the fuels mentioned decreased pre-ignition combustion. On the contrary, the addition of a knock inducer caused a marked increase.

H. S. GARLICK.

**Propylene from crude oil.** A. DOBRYANSKI, B. ARKHANGELSKI, and R. STEPANYAN (Nef. Choz., 1928, 15, 46—52).—When cracked (0.31 g. per min. through a copper tube at 550—750°), solar oil, of the various fractions examined, gave the highest yield of propylene. The optimal temperature was 600—650°. At higher temperatures the propylene decomposed with formation of ethylene. The yield of propylene could not be increased by using nickel or iron as catalyst.

CHEMICAL ABSTRACTS.

**Higher alcohols from petroleum hydrocarbons.** III. E. HOLZMANN and S. PILAT (Przemysl Chem., 1929, 13, 455—460; cf. Pilat and others, B., 1929, 743).—The best yields of alcohol from low-boiling benzines produced by the Cross cracking system are obtained by shaking 1 litre of the benzine with 500 c.c. of 85% sulphuric acid and 100 g. of acetic acid, at a temperature below 0°, when 147 c.c. of alcohols are obtained; in the absence of acetic acid the yield is only 88 c.c. The former figure represents a yield of 50% of the olefines present. By treatment of benzine successively with 65 and 85% sulphuric acid, a mixture in the ratio 2:1 of secondary to tertiary butyl, amyl, and hexyl alcohols is obtained. The hydrolysis of the alkyl sulphates should be carried out in an alkaline medium, otherwise olefines are regenerated and largely converted into tarry polymerides. R. TRUSZKOWSKI.

**Oxidation mechanisms of the paraffin hydrocarbons.** G. EGLOFF, R. E. SCHAAD, and C. D. LOWRY, JUN. (Ind. Eng. Chem., 1929, 21, 785—791).—The theories proposed to account for the course of oxidation of the paraffins are critically reviewed. Particular attention is paid to the hydroxylation theory, which fails to explain why primary alcohols appear not to be primary products of hydrocarbon oxidation, and to the peroxide theory, which satisfactorily explains the prominence of aldehydes among the oxidation products, but cannot be considered proved fully because of the failure to isolate and identify peroxidic oxidation products, nor does it present a complete explanation of knocking, for the tendency of hydrocarbons to knock does not parallel what is known of their ability to form peroxides. The study of flame propagation and the assumption of a chain mechanism for hydrocarbon oxidation make intelligible the checking of knocking by chemical reagents which act as inhibitors of a special

type, and also accords with the stimulation of knocking by unstable reactive substances which, in the engine cylinder, may include oxides of nitrogen and ozone, as well as peroxides. H. S. GARLICK.

**Refining of shale oil.** Y. TANAKA and R. KOBAYASHI (J. Fuel Soc. Japan, 1929, 8, 92—93).—The addition of an adsorbent substance, such as clay, before distillation of a shale oil reduces the production of pitch and gas, and improves the quality of the liquid distillate. The process may also be improved by addition of alcohol vapour, as the b.p. of the oil fractions are thereby lowered. Hydrogenation of shale oil can be carried out at 280—290° and 75 atm. in presence of a nickel catalyst, or by nascent hydrogen produced from zinc and acid; in either case the yield of solid paraffins is considerably increased. R. H. GRIFFITH.

**Fuller's earth and acid-treated earths as oil-refining adsorbents.** C. W. DAVIS and L. R. MESSER (Amer. Inst. Min. Met. Eng. Tech. Pub., 1929, No. 207, 18 pp.).—Fuller's earth and earths from which oil-bleaching adsorbents may be prepared vary considerably in composition, but a high silica:alumina ratio is characteristic. The characteristics of the earths are discussed. CHEMICAL ABSTRACTS.

**Determination of unsaturated and aromatic hydrocarbons in gasoline.** S. S. NAMETKIN and E. A. ROBINZON (Nef. Choz., 1929, 14, 775—779).—Treatment of gasoline with 98% sulphuric acid at 0° for 30 min. removes the whole of the unsaturated hydrocarbons. CHEMICAL ABSTRACTS.

**Testing of motor spirit.** R. BRUNSCHWIG and L. JACQUÉ (Compt. rend., 1929, 189, 486—489).—Potential resins in motor spirit may be determined satisfactorily by passing a current of air through the liquid while refluxing, evaporating, and weighing the residue remaining at 105°. Production of resins by irradiation with ultra-violet light does not give results in agreement with those obtained by storing for nine months. C. W. GIBBY.

**Thermal decomposition of paraffin wax in presence and in absence of hydrogen under high pressure.** H. I. WATERMAN, T. W. TE NUYL, and J. N. J. PERQUIN (J. Inst. Petroleum Tech., 1929, 15, 369—371).—A comparison of analogous fractions from the vacuum distillation of the products of thermal decomposition of paraffin wax in the presence (berginisation) and in the absence (cracking) of hydrogen under high pressure shows that the aniline point of the berginisation product is about 20° higher than that of the cracking product, the refractive index of the cracked products is 0.02—0.03 higher, their sp. gr. is higher, and the bromine value of the cracking products is much higher, substitution more readily taking place on cracking than on berginisation. Comparison with the physical constants of the normal saturated aliphatic hydrocarbons indicates the berginised product to consist possibly of 90% of saturated hydrocarbons, whilst the cracking product is strongly unsaturated and contains compounds of a cyclic character. H. S. GARLICK.

**Anti-oxygenic effects of sulphur and selenium on refined transformer oil and paraffin wax.** S. MIZUSHIMA and T. YAMADA (J. Soc. Chem. Ind. Japan,

1929, 32, 848—851).—That highly refined transformer oil is far more easily oxidisable than a less refined one is confirmed. The authors' results, contrary to those of other workers, show that sulphur contained in petroleum oils acts as an anti-oxidant. When a refined white transformer oil (originally containing 0.023% S) was mixed with 0.1% of sulphur and oxidised with oxygen at 86 cm. pressure for 100 hrs. at 120°, the oil resisted the oxidation almost perfectly, whilst the same oil without added sulphur was strongly oxidised. By mixing with a small amount of sulphur, paraffin wax resisted the oxidising action of oxygen. Further, isoamyl sulphide and selenium are, equally as is free sulphur, remarkably effective as anti-oxidants for refined transformer oil. K. KASHIMA.

**Physical properties and constitution of mineral lubricating oils.** S. KYROPOULOS (Z. physikal. Chem., 1929, 144, 22—48).—The relation between refractive index and mol. wt. is represented by separate curves for normal paraffins, isoparaffins, naphthenes, and aromatic hydrocarbons, and the determination of these two constants for a mixture of hydrocarbons of unknown character enables it to be classified. The method has been applied to a number of mineral lubricating oils in cases where the great complexity of the mixtures makes identification by chemical means impracticable. A typical group of Pennsylvanian lubricating oils ("Valvoline") and some other saturated hydrocarbon oils are shown to be mixtures consisting largely of isoparaffins. The lower lubricating value of oils containing normal paraffins is due partly to the superior lubricating properties of branched-chain as compared with straight-chain molecules, but largely also to the fact that in a mixture of given b.p. the normal paraffins have a lower mol. wt. than the isoparaffins, and consequently lower lubricating power. The removal of normal paraffins by crystallisation from oils used in internal-combustion engines is especially important, since the former are the least readily burned and therefore accumulate in the unburnt residue, thereby greatly lowering the lubricating power and leading to excessive consumption. The relation between the technical value of lubricating oils and their chemical nature, and the question of the origin of mineral oils, are also discussed. F. L. USHER.

**Iodine values of lubricating oils before and after use in automobile engines.** W. F. SEYER and J. S. ALLEN (Ind. Eng. Chem., 1929, 21, 793—794).—The iodine value, as determined on a definite sample of dried oil dissolved in carbon tetrachloride and in contact with excess iodine monochloride for 2 hrs. at 0°, shows, with one or two exceptions, that the iodine value of lubricating oil decreases in use in automobile engines in spite of dilution by unconsumed fuel, and that the decrease is greatest during the first 500 miles. H. S. GARLICK.

**Gas purification in relation to coal sulphur.** F. W. SPERR, JUN. (Proc. 2nd Int. Conf. Bit. Coal, 1928, 2, 37—64).

**Products of decomposition in the distillation of mazout.** S. A. VISHETRAVSKI (Nef. Choz., 1928, 14, 769—771).

Mazout distillation in Germany and a new process developed by the Azneft for extracting oil out of heavy bottoms. K. V. KOSTRIN (Neft. Choz., 1928, 15, 658—663).

Distillation of complex mixtures. GAY.—See I. Gas filter: COLLES.—See X. Brewers' pitch. KUTTER.—See XVIII. Determination of water [in coal etc.]. PRITZKER and JUNGKUNZ.—See XIX.

## PATENTS.

Production of briquettes. F. L. SCHMIDT (B.P. 294,879, 19.7.28. Ger., 30.7.27).—For the production of a hard briquette, the products obtained by fermentation of cellulose or molasses lyes are employed. The sugar substances present are thus decomposed, leaving a material from which a non-sticky substance (similar to tar pitch) may be obtained by evaporation. At the same time such valuable by-products as alcohol, acetone, acetaldehyde, etc. are obtained.

J. A. SUGDEN.

Briquetting of coal, coke, and the like. P. J. and J. P. SPENGLER (B.P. 299,859, 27.4.28. Ger., 4.11.27).—Briquettes may be weak owing to air fissures and striation. De-aeration may be effected during pressing by applying the pressure in steps and by partially or wholly removing the briquette from the mould after each application. In this way a smaller quantity or an inferior binder may be used.

J. A. SUGDEN.

Manufacture of agglomerated blocks of fuel. A. L. J. VOINCHET and A. A. LERCIU (B.P. 297,085, 11.9.28. Fr., 14.9.27).—China or other clay, to which is added an alkaline silicate, is used as the binding agent. The material is compressed, then treated with a solution of a calcium salt (e.g., chloride) containing traces of acids, glycerin, etc. in order to render the binder insoluble and capable of resisting disintegration by water, and finally dried. The product burns without smoke, and leaves no fusible ash; drying of the fuel before briquetting is not necessary. J. A. SUGDEN.

Treatment of coal dust for utilisation in burners. L. LAIS (B.P. 316,389, 21.6.28).—The dried dust particles are covered with a non-absorbent layer of a substance such as coal-tar pitch by mixing 3—6% of the finely powdered pitch with the dried coal dust at the liquefying temperature of the pitch; a small amount (3—5%) of greasy material, e.g., mazout or petroleum oil, may also be added. Risk of blockages in feed pipes due to absorption of moisture and of spontaneous combustion of the coal dust when stored in bins is thereby avoided. J. A. SUGDEN.

Preparation of granular carbon. H. H. LOWRY, Assr. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,722,055, 23.7.29. Appl., 1.6.25).—Comminuted carbonaceous material is heated slowly, so that a uniform product of low porosity is obtained, and this is subsequently heated to a higher temperature. R. H. GRIFFITH.

Carbonisation of briquettes or like agglomerate blocks of fuel. E. GEVERS-ORBAN and J. PIETERS (B.P. 294,581, 26.7.28).—To prevent briquettes from sticking together or altering their shape during carbonisation, it is necessary to embed them in some

powdered material. In the case of briquettes made from peat, lignite, or other non-caking substances, the material must first be carbonised, then agglomerated with a binder, and carbonised again. In the second carbonisation the briquettes are embedded in the powdered raw fuel, which after the carbonisation is ready for briquetting. This process is continuous. In the case of bituminous or caking materials which require only one carbonisation, the briquettes are embedded in coke breeze left over from the previous carbonisation. J. A. SUGDEN.

Carbonising or distilling material. INTERNAT. COMBUSTION, LTD., and S. McEWEN (B.P. 316,734, 8.6.28).—In a modification of the prior patent (B.P. 316,901; B., 1929, 802) the gases as evolved are drawn down through the hot coke in order to increase the yield of permanent gas. J. A. SUGDEN.

Obtaining liquid products from coal and the like. M. MELAMID (U.S.P. 1,723,431, 6.8.29. Appl., 5.12.24. Ger., 17.11.21).—The hydrogenation of coal and similar substances is carried out under pressure in the presence of tin, and at a temperature not above 600°.

R. H. GRIFFITH.

Manufacture of viscous oils from brown-coal tars or their distillation products. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 316,701, 11.5.28).—Good yields of viscous oils can be obtained from oils freed from benzine or paraffin wax, by treatment, at 100—200°, with metallic chlorides (e.g., aluminium or ferric chloride). Heavy-metal salts, particularly those of group VI, may also be added as activators. The yield depends on the fraction employed and its treatment. The formation of low-boiling constituents is almost completely prevented. J. A. SUGDEN.

Gasification of solid fuel. J. M. RUSBY and C. W. ANDREWS, Assrs. to U.G.I. CONTRACTING Co. (U.S.P. 1,720,403, 9.7.29. Appl., 5.12.22).—Bituminous fuel is subjected to a hot air-blast; when the charge becomes plastic the air supply is stopped and the fuel bed is stirred mechanically to break up the coalesced material. Fresh fuel is then added, and the process continued.

R. H. GRIFFITH.

Gas producers. I. G. FARBENIND. A.-G. (B.P. 300,277, 7.11.28. Ger., 10.11.27. Addn. to B.P. 214,544; B., 1924, 549).—In producers for gasifying small-sized fuel, as described in the prior patent, a waste-heat boiler (preferably water-tube) is built into the upper portion of the producer itself, or into the dust separator. When it is not desired to produce water-gas or producer gas, sufficient secondary air for complete combustion may be admitted above the fuel bed.

J. A. SUGDEN.

Gas producers. T. R. WOLLASTON (B.P. 316,749, 5.7.28).—The water-cooled walls of the producer are utilised as a steam boiler. At the hot zone the annular boiler itself serves as the wall, but the upper portion of the boiler (containing the water level and steam space) is protected by the ordinary refractory lining. The water level is maintained by a feed-water regulator. A central column, terminating in a conical distributing head, extends upwards towards the fuel-feed throat and, together with a series of fire bars which can be agitated,

ensures a uniform movement of the fuel. The steam generated may be utilised in heating installations or to saturate the blast. J. A. SUGDEN.

**Decomposition of gas mixtures, more particularly coke-oven gas.** GES. F. LINDE'S EISMASCHINEN A.-G. (B.P. 284,213, 21.1.28. Ger., 24.1.27).—Coke-oven gas is compressed to 10 atm. and the carbon dioxide, hydrogen sulphide, etc. are removed; the purified gas is then cooled in a countercurrent heat exchanger so that the higher-boiling constituents separate. Most of the methane is subsequently condensed in other coils around which circulates additional compressed nitrogen; the cooling effect produced by evaporation of the methane is thus transferred to the nitrogen and carried to another point, when the latter evaporates at a lower pressure. After this treatment the residual gas is rich in carbon monoxide and hydrogen; the former is readily condensed, and the hydrogen is finally purified by washing with liquid nitrogen. R. H. GRIFFITH.

**Cracking of hydrocarbon oils.** SINCLAIR REFINING Co., Assees. of E. C. HERTHEL (B.P. 303,879, 3.11.28. U.S., 12.1.28).—An initial charge of oil is brought to cracking conditions of temperature and pressure and the vapours are subjected to a refluxing operation in direct contact with fresh oil. At the same time the reflux condensate and the unvaporised raw oil from the refluxing operation are circulated through the heating zone mixed with the unvaporised oil recirculated from the vaporising zone until a substantial amount of cracked oil has been taken off. Then, before the oil circulating through the heating zone becomes saturated with pitch, the recirculation of unvaporised oil from the vaporisation zone through the heating zone is stopped, while continuing to subject vapours taken off from the vaporisation zone to the refluxing operation and to circulate admixed reflux condensate and unvaporised fresh oil from the refluxing operation through the heating zone until a further substantial amount of cracked oil has been taken off. During the second-mentioned period of operation unvaporised oil from the vaporisation zone is discharged. Finally, discharge of unvaporised oil is stopped and recirculation through the heating zone continued. H. S. GARLICK.

**Catalytic treatment of hydrocarbon oil.** G. EGLOFF and J. C. MORRELL, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,705,180, 12.3.29. Appl., 10.5.22. Renewed 5.9.28).—Into the oil raised to cracking temperature are injected regulated quantities of hydrogen chloride, and the mixture is passed into an enlarged zone containing disintegrated aluminium, a pressure above atmospheric being maintained during the cracking process. The vapours produced are condensed and collected. F. G. CROSSE.

**Conversion [cracking] of hydrocarbon oil.** G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,705,181, 12.3.29. Appl., 4.12.25).—Fuel oil, or other heavy crude oil containing no light fractions, is heated, e.g., at 200 lb./in.<sup>2</sup> and 440°, and discharged into a reaction chamber simultaneously with kerosene oil heated separately at, e.g., 2000 lb./in.<sup>2</sup> and 595°. The expansion chamber is connected with the usual dephlegm-

ator, condenser, and receiver, and the phlegms are returned to a separate heating tube and discharged into the reaction chamber at, e.g., 1200 lb./in.<sup>2</sup> and 540°. R. BRIGHTMAN.

**Distillation of hydrocarbon oils.** W. W. HOLLAND, Assr. to STANDARD OIL Co. (U.S.P. 1,723,698, 6.8.29. Appl., 11.12.24).—Vapours from the still pass into a dephlegmator, in the upper part of which the partly fractionated vapours are in indirect heat exchange with a current of steam at 105–120°. Condensate returns to the still in countercurrent with ascending vapours, whilst the steam heated thereby to, e.g., 160–220° is passed directly into the distilling oil. R. BRIGHTMAN.

**Purification of hydrocarbons.** I. H. DERBY (Assr. to P. C. REILLY) and K. R. DAVIS (U.S.P. 1,727,052, 3.9.29. Appl., 13.2.22).—Normally solid hydrocarbon compounds are atomised in a molten condition and subjected to a current of inert gas. H. ROYAL-DAWSON.

**Treatment of carbon from cracking stills.** G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,705,179, 12.3.29. Appl., 23.4.21. Renewed 20.2.28).—Oil-soaked carbon is discharged from the expansion chamber of a cracking still into a rotating, centrally heated, cylindrical still, preferably provided with steel balls to disintegrate the carbon. The liquid residue distils off at atmospheric pressure and is stored or returned for cracking, and the dry carbon is discharged. R. BRIGHTMAN.

**Production of lubricating oil from petroleum oil.** J. B. TERRY and R. A. HALLORAN, Assrs. to STANDARD OIL Co. of CALIFORNIA (U.S.P. 1,721,719, 23.7.29. Appl., 14.2.23. Renewed 16.5.28).—Petroleum is distilled under reduced pressure until asphalt remains; about one half of this residue is then destructively distilled to leave a brittle pitch, which is then extracted with solvent naphtha in order to recover the lubricating oils that are mixed with it. R. H. GRIFFITH.

**Lubricating composition.** A. L. KLEES, Assr. to COMBUSTION UTILITIES CORP. (U.S.P. 1,727,109, 3.9.29. Appl., 16.7.24).—The composition comprises a mixture of cyclic unsaturated non-benzenoid hydrocarbons with naphthenic and paraffin hydrocarbons of similar boiling range. L. A. COLES.

**Separation of hydrogen from gaseous mixtures [coke-oven gas].** G. F. JAUBERT, Assr. to SOC. AMMONIA (U.S.P. 1,723,425, 6.8.29. Appl., 26.12.24. Fr., 6.11.24).—See B.P. 242,583; B., 1926, 440.

**Cracking of hydrocarbon oils.** E. C. HERTHEL, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,721,779 and 1,721,780, 23.7.29. Appl., 12.1.28).—See B.P. 303,879; preceding.

**Manufacture of carburetting liquids similar to petrol.** E. A. PRUDHOMME, Assr. to SOC. INTERNAT. DES PROC. PRUDHOMME (S.I.P.P.) (U.S.P. 1,711,855, 7.5.29. Appl., 20.1.25. Fr., 19.5.24).—See B.P. 238,931; B., 1925, 950.

**Treatment by pressure and heat of heavy mineral oils and carbon material.** M. HOFSSÄSS, Assr. to INTERNAT. BERGIN COMP. VOOR OLIE- EN KOLEN-CHEMIE

S'GRAVENHAGE (U.S.P. 1,729,943, 1.10.29. Appl., 14.6.22. Ger., 14.6.21).—See B.P. 213,661; B., 1924, 503.

**Liquid fuel burning apparatus.** HOMATRA, LTD., and M. TRACHTENBERG (B.P. 319,468, 18.8.28).

**Decohering solid substances** (B.P. 292,965).—See I. **Extraction of naphthenic acid** (U.S.P. 1,720,821).—See III. **Emulsion paints** (U.S.P. 1,691,765—8).—See XIII. **Liquors for treating leather** (B.P. 318,070).—See XV.

### III.—ORGANIC INTERMEDIATES.

**Catalysts for the formation of alcohols from carbon monoxide and hydrogen.** IV. **Decomposition and synthesis of methyl alcohol by catalysts composed of zinc and chromium oxides.** D. S. CRYDER and P. K. FROLICH (Ind. Eng. Chem., 1929, 21, 867—871; cf. A., 1929, 153).—The close similarity between the decomposition and the synthesis of methyl alcohol previously noted with zinc-copper catalysts (A., 1928, 1112) is also evident with zinc-chromium oxide catalysts. Catalysts containing less than 50 mol.-% Cr are mixtures of zinc oxide and chromium trioxide, and can be dehydrated and reduced by methyl alcohol at 220°; above 50% the excess chromium is added as the sesquioxide, since then the higher oxide is irregularly reduced. The decomposition experiments, carried out at 1 atm. and 340°, show that a maximum yield of carbon monoxide, equivalent to 85% of the methyl alcohol converted, occurs with a catalyst containing 75 mol.-% Zn. Catalysts richer in chromium gave relatively large yields of carbon dioxide and unsaturated hydrocarbons, whilst those richer in zinc tended to produce methyl formate. The activity of the catalysts, as shown by the amount of alcohol decomposed, has also a sharp maximum at 78% Zn, a result in general agreement with that of Smith and Hawk (A., 1928, 488). In the synthetic experiments, carried out at 204 atm. and 350°, the maximum production of methyl alcohol occurred with a catalyst containing 77 mol.-% Zn, in which case 18% of the carbon monoxide was converted. The optimum temperature for a catalyst of this composition is 370—390°; the liquid product formed is shown by fractionation to be almost pure methyl alcohol. The yield of formaldehyde was almost constant throughout the decomposition experiments, and it is suggested that this is an intermediate product, which either breaks down to carbon monoxide and hydrogen or yields methyl formate as a condensation product.

T. H. MORTON.

**Determination of iodoform.** A JÁNSKÝ (Časopis Českoslov. Lék., 1928, 8, 215—221; Chem. Zentr., 1929, i, 2454—2455).—A study of published methods.

A. A. ELDRIDGE.

**Preparation of isovaleric acid from fusel oil.** A. PIOTROWSKI (Przemysl Chem., 1929, 13, 413—418).—Fusel oil obtained from the rectification of potato spirit contains about 80% of isoamyl alcohol, b.p. 128—132°. A mixture of fusel oil vapour with air (820 c.c. of air per g. of alcohol) is passed at a rate of 1.75 m./sec. through a tube containing two copper gauze spirals at 370—390°. A yield of about 75% of oily products, consisting chiefly of unreacted alcohol and of isovaleralde-

hyde, is obtained, and this product gives on oxidation with permanganate a final yield of 39.2% of isovaleric acid. Whilst this yield is smaller than that obtained by direct oxidation by permanganate of the fusel oil, the cost per unit weight of isovaleric acid is considerably lower, using the above indirect method.

R. TRUSZKOWSKI.

**Manufacture of benzaldehyde.** P. SHORIGIN, I. KIZBERG, N. TROITZKI, and E. SMOLYANINOVA (J. Chem. Ind. Moscow, 1929, 6, 258—260).—For the oxidation of toluene with manganese dioxide the optimal conditions consist in the use of 65% sulphuric acid, ratio of toluene to manganese dioxide 4:1, temperature 18—19°; the yield (calculated on the active manganese dioxide) was 55—56% of the theoretical. A small quantity of nitric acid has a slight accelerating action. Substitution of manganese dioxide by pyrolusite which had been finely divided or heated under pressure with sodium hydroxide was unsatisfactory. Oxidation of toluene vapour by atmospheric oxygen in the presence of a vanadium oxide catalyst gave poor yields of benzaldehyde contaminated with benzoic acid and anthraquinone. Chlorination of toluene vapour followed by treatment with sodium hydroxide and oxidation with bleaching powder and sodium carbonate at 40—60° gives favourable industrial yields of a mixture of benzaldehyde and benzoic acid. The catalytic oxidation of benzyl alcohol or benzyl chloride using a vanadium oxide catalyst is described, yields of 51% and 74%, respectively, being recorded. CHEMICAL ABSTRACTS.

**Preparation and conversion [into the nitro- and amino-naphthols] of 5-nitro- $\alpha$ -naphthylamine.** N. N. VOROSHTZOV and A. A. KULEV (Bull. Inst. Polytech. Ivanovo-Vosniesensk, 1928, 9, 87—93).—1:5-Dinitronaphthalene was obtained from the commercial product by means of sulphuric acid or aniline, and was reduced with sodium sulphide and ammonium sulphate to the nitronaphthylamine in 40% yield. The method of Morgan and Jones for the preparation of this substance (J.S.C.I., 1923, 42, 342 r) was modified. The best yield of 5-nitro- $\alpha$ -naphthol obtained was 41.7%, the corresponding aminonaphthol being obtained from it in 88.1% yield. CHEMICAL ABSTRACTS.

**Nitration of  $\beta$ -naphthol-6-sulphonic acid.** N. N. VOROSHTZOV and N. M. KASATKIN (Bull. Inst. Polytech. Ivanovo-Vosniesensk, 1928, 11, 75—85).—Schaeffer's salt, when treated with nitric and anhydrous sulphuric acids, afforded 13% of 1-nitro- $\beta$ -naphthol-6-sulphonic acid, and 32% of a red dinitro-compound; dinitration afforded 78% of the latter. CHEMICAL ABSTRACTS.

**Meso-derivatives of anthracene and dianthryl.** B. P. FEDOROV (Bull. Inst. Polytech. Ivanovo-Vosniesensk, 1928, 9, 103—109).—The action of sodium sulphite on 9-nitroanthracene affords sodium anthracene-9-sulphonate; the SO<sub>3</sub>Na group is readily replaced by H, OH, or NH<sub>2</sub>. There is also formed sodium dianthryl-*ms*-sulphonate, from which *ms*-hydroxydianthryl and *ms*-aminodianthryl, m.p. 305° (decomp.), are readily obtainable. CHEMICAL ABSTRACTS.

**Oxidation of lignin.** HORN. **Propylene from crude oil.** DOBRYANSKI and others. **Alcohols from**



petroleum hydrocarbons. HOLZMANN and PILAT.—See II. Purification of ethyl acetate. VON MIKÓ.—See XX.

## PATENTS.

Distillation and vapour-phase extraction of volatile substances [crude pyroligneous acid]. H. SUIDA (U.S.P. 1,715,313, 28.5.29. Appl., 9.2.26. Austr., 21.2.25).—Crude diluted pyroligneous acid flows through a series of distillation retorts at successively lower pressures and temperatures. Steam is injected into the first retort and the vapours from the retort ascend a tower down which a stream of extraction agent descends and carries the acetic acid with it into a collecting conduit at the bottom of the tower. Steam passes from the top of the tower to the succeeding still and thereby distils off the residual liquor from the first still under a reduced pressure. Residual liquor from the final still affords tar and a little acetic acid. R. BRIGHTMAN.

Manufacture of esters [*sec.*-butyl acetate]. H. E. BUC and W. W. CLOUGH, Assrs. to STANDARD DEVELOPMENT Co. (U.S.P. 1,726,945, 3.9.29. Appl., 23.8.23).—A mixture of about 55% of *sec.*-butyl alcohol, 45% of concentrated acetic acid, and 0.1% of sulphuric acid is distilled and the upper layer settling from the condensed liquid is refluxed continuously until stratification practically ceases; the still contents are then distilled in fractions to within a few degrees of the b.p. of *sec.*-butyl acetate and of that of acetic acid, respectively, and water is added to the latter fraction to form with the *sec.*-butyl acetate present a constant-boiling mixture which is then separated by distillation.

L. A. COLES.

Aryl alkyl esters. R. H. VAN SCHAAK, JUN., Assr. to VAN SCHAAK BROS. CHEM. WORKS (U.S.P. 1,700,960, 5.2.29. Appl., 14.9.27).—Aryl alkyl esters of phthalic acid, or, e.g., succinic, citric, or phenylmalonic acid, obtained from saturated alcohols, b.p. below 150°, are used as pyroxylin solvents. *Benzyl ethyl phthalate* has b.p. 199°/3 mm.

R. BRIGHTMAN.

Hexyl [ $\alpha\delta$ -dimethylbutyl] esters. R. H. VAN SCHAAK, JUN., Assr. to VAN SCHAAK BROS. CHEM. WORKS, INC. (U.S.P. 1,702,180, 12.2.29. Appl., 20.1.28).—The esters are prepared from methylisobutylcarbinol in benzene in presence of sulphuric acid, e.g., 0.02%, as catalyst.  $\alpha\delta$ -Dimethylbutyl propionate has b.p. 163°,  $d_{25}^{25}$  0.87.

R. BRIGHTMAN.

Manufacture of *N*-[hydr]oxyethyl derivatives of 2-amino-1-oxybenzene [*o*-aminophenol]. I. G. FARBENIND. A.-G. (B.P. 292,950, 13.6.28. Ger., 27.6.27).—*o*-Aminophenol is heated at 100° with aqueous ethylene chlorohydrin in presence of an acid-binding agent (calcium carbonate). *o*- $\beta$ -Hydroxyethylaminophenol, m.p. 80—81°, and *o*-*di*-( $\beta$ -hydroxyethyl)aminophenol are obtained according to the proportion of chlorohydrin.

C. HOLLINS.

Manufacture of *o*-chloronitrobenzene-*p*-sulphonic acid. H. W. HILLYER, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,701,367, 5.2.29. Appl., 30.1.20).—Chlorobenzene is sulphonated with concentrated sulphuric acid at 90—100°, and the mixture is cooled to 20° and treated at 30—50° with mixed acid (nitric acid 29%, sulphuric acid 65%, water 6%). The product

may be salted out after dilution with water, or isolated by the process of U.S.P. 1,564,239 (B., 1926, 122).

R. BRIGHTMAN.

Manufacture of 6-ethoxy-2:4-dimethylquinoline. H. T. CLARKE and E. R. TAYLOR, Assrs. to EASTMAN KODAK Co. (U.S.P. 1,701,144, 5.2.29. Appl., 7.7.23).—*p*-Phenetidine is condensed with acetone in presence of iodine at 92—96° for 36 hrs. Acetone and water are distilled off until the temperature reaches 140°, the mixture is washed with sodium hydroxide, and the *p*-ethoxyacetoneamide distilled at 165—180°/10 mm. and treated with hydrogen chloride at 180—200° for 8 hrs.

R. BRIGHTMAN.

Manufacture of naphthalene [naphthaquinone] derivatives. BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLPIN, and E. W. KIRK (B.P. 315,331, 7.2.28).— $\beta$ -Benzoylpropionic acids, obtained by condensing succinic anhydride or its halogen derivatives with quinol, toluquinol, 3:6-dihydroxy-*o*-xylene, *p*-aminophenol, *p*-chlorophenol, 3:4-dichlorophenol, or other *p*-disubstituted benzene derivatives, are cyclised and at the same time oxidised by means of sulphuric acid in presence of boric acid. The condensation, cyclisation, and oxidation may be performed in one operation. The preparation of naphthazarin from quinol is described.

C. HOLLINS.

Extraction and purification of naphthenic acid. S. P. COLEMAN, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,720,821, 16.7.29. Appl., 30.7.27).—High-boiling organic acids are separated from admixed oil by conversion into the corresponding alkali salts, which are extracted with water. The free acids are then obtained by adding mineral acid, are converted again into their salts, and heated under reduced pressure to remove the final traces of oil.

R. H. GRIFFITH.

Manufacture of condensation products of the benzodiazine [quinazoline] series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 309,102, 31.12.26).—2:4-Dichloro- or -dibromo-quinazoline is condensed with 2 mols. (*A* and *B*) of compounds containing reactive hydrogen attached to nitrogen, oxygen, or sulphur; the 2 mols. may be identical except in the case of water, ammonia, an alkali hydrogen sulphide, or a monohydric aliphatic alcohol. Examples are described in which *A* = *B* = H-acid, J-acid,  $\gamma$ -acid, S-acid, *m*-aminobenzoyl-J-acid, *p*-nitroaniline-*m*-sulphonic acid (reducible to a diamine), *m*-phenylenediamine-4-sulphonic acid, 5-sulphinosalicylic acid (to give a bis-sulphone), 5-aminosalicylic acid,  $\alpha$ -aminoanthraquinone (m.p. above 265°), aniline, sodium phenoxide, thio-*p*-cresol,  $\alpha$ -naphthol, or 2:5-diaminobenzoic acid; *A* = H-acid, *B* = J-acid, phenylhydrazine, aniline, methylamine, naphthylamine, 2:5-diamino-4'-methylidiphenyl sulphone, ammonia, phenol, toluene-*p*-sulphinic acid, potassium methoxide, benzyl alcohol, cyclohexanol, *p*-aminobenzeneazosalicylic acid, dithioglycol (0.5 mol.), resorcinol (0.5 mol.); *A* = J-acid, *B* = sodium hydrogen sulphide; *A* = S-acid, *B* = sodium hydroxide; *A* = *p*-phenylenediamine (0.5 mol.), *B* = H-acid, *m*- or *p*-phenylenediaminesulphonic acid, 4:4'-diaminodiphenyl-3-sulphonic acid, ammonia, *p*-aminophenyl-5-hydroxy-7-sulpho-2-naphthylcarbamide; *A* = phenol, *B*

= sulphanilic acid; *A* = sulphanilic acid, *B* = H-acid; *A* = *m*-phenylenediamine-4-sulphonic acid, *B* = J-acid, *m*-aminofornanilide; *A* = 4-*m*-nitrobenzamidometanilic acid, *B* = 6-*m*-nitrobenzamido-*o*-toluidine-4-sulphonic acid; *A* = 0.5 mol. of 4:4'-diaminodiphenylamine-2-sulphonic acid or 4:4'-diaminostilbene-2:2'-disulphonic acid, *B* = *m*-phenylenediamine-4-sulphonic acid or 2:6-dichloro-*p*-phenylenediamine; *A* = *p*-nitroaniline-*m*-sulphonic acid, *B* = dimethylamine, aniline, *o*-anisidine, aminophenylpyrazolones, J-acid,  $\gamma$ -acid; *A* = 4-nitro- $\alpha$ -naphthylamine-6-sulphonic acid, *B* = glycol (convertible into sulphato-compound), diethyl- $\beta$ -methylaminoethylamine; *A* = phenol, *B* = H-acid; *A* = 5-sulphinosalicylic acid, *B* = H-acid, ethyl-H-acid, methyl-J-acid, 1-*m*-aminophenyl-5-pyrazolone-3-carboxylic acid, *m*- or *p*-phenylenediaminesulphonic acid; *A* = 3-*p*-aminophenylureidobenzoyl-K-acid, *B* = sodium sulphite; *A* = acetyl-1:4-naphthylenediamine-6-sulphonic acid, *B* = ammonia (acetyl group removed).

C. HOLLINS.

**Manufacture of unsaturated aldehydes.**

A. KNORR and A. WEISSENBORN, Assrs. to WINTHROP CHEM. CO., INC. (U.S.P. 1,716,822, 11.6.29. Appl., 7.3.27. Ger., 2.7.26).—See B.P. 284,458; B., 1928, 634.

**Production of aldehyde-sulphoxylates.**

C. SCHUMANN, E. MUNCH, O. SCHLICHTING, and B. CHRIST, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,714,636—7, 28.5.29. Appl., 9.4.28. Ger., 22.4.27).—See B.P. 308,229; B., 1929, 549.

**Manufacture of depolymerisation products from carbohydrates of high mol. wt.** F. LANGE, Assr. to I. G. FARBEIND. A.-G. (U.S.P. 1,714,565, 28.5.29. Appl., 16.3.27. Ger., 17.3.26).—See B.P. 290,377; B., 1928, 515.

**Purification of phthalic anhydride.** A. O. JAEGER and F. A. CANON, Assrs. to SELDEN CO. (U.S.P. 1,702,871, 19.2.29. Appl., 6.9.27).—See B.P. 285,017; B., 1929, 672.

**Preparation of 2-aminonaphthalene-3-carboxylic acid [2-amino-3-naphthoic acid].** E. HOTZ and V. LANTZ, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,690,785, 6.11.28. Appl., 16.12.27. Ger., 17.12.26).—See B.P. 282,450; B., 1929, 467.

**Manufacture of  $\alpha$ -aminoanthraquinone- $\beta$ -carboxylic acids.** K. WILKE, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,698,900, 15.1.29. Appl., 3.3.27. Ger., 8.3.26).—See B.P. 267,164; B., 1928, 361.

**$\alpha$ -Di(methylamino)- $\alpha$ -dihydroxyanthraquinone-disulphonic acids and their manufacture.** R. E. SCHMIDT and W. TRAUTNER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,702,022, 12.2.29. Appl., 13.4.26. Ger., 17.4.25).—See B.P. 250,968; B., 1927, 743.

**Production of ketones of the anthraquinone series.** A. LÜTRINGHAUS and F. KÄSER, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,730,081, 1.10.29. Appl., 26.1.28. Ger., 16.2.27).—See B.P. 289,585; B., 1928, 516.

**Preparation of condensation products of the benzanthrone series.** G. KRÄNZLEIN and M. CORELL, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,714,677, 28.5.29. Appl., 5.3.26. Ger., 9.3.25).—See B.P. 248,791; B., 1927, 550.

**Isatin derivatives and their manufacture.** M. P. SCHMIDT and O. HERRMANN, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,700,814, 5.2.29. Appl., 13.9.26. Ger., 6.6.25).—See B.P. 282,863; B., 1928, 151.

**Manufacture of isatins [and *N*-arylsulphonyl derivatives thereof].** K. SCHIRMACHER and K. RENN, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,698,894, 15.1.29. Appl., 27.1.27. Ger., 28.1.26).—See B.P. 265,224; B., 1928, 224.

**Fatty acid peroxide** (U.S.P. 1,718,609).—See XII. **Butyl alcohol and acetone by fermentation** (B.P. 319,079).—See XVIII.

## IV.—DYESTUFFS.

**Synthesis of alizarin.** V. I. MINAEV and B. P. FEDOROV (Bull. Inst. Polytech. Ivanovo-Vosniesensk, 1928, 9, 95—101).—The method of G.P. 241,806 is modified; 5 pts. of sodium hydroxide, 1 pt. of anthraquinone, and water to produce a 28% concentration of sodium hydroxide are kept at 180—200° for 18—30 hrs. after a period of 6 hrs. at 130—138° in the absence of sodium nitrate. Meso-derivatives of anthracene give poor yields of alizarin if the fusion is carried out without addition of sodium sulphite; the presence of sodium nitrate is necessary for the mononitro- but unnecessary for the dinitro-derivative. CHEMICAL ABSTRACTS.

**Spectroscopic examination of food dyes.** B. V. HEATH (J. Soc. Chem. Ind. Victoria, 1929, 29, 159—165).—A 1:200 aqueous solution of the dye is examined with a direct-vision spectroscope, and the position of the absorption bands noted with reference to the Fraunhofer lines, an accuracy of 50 Å. sufficing. Further readings are then taken after halving the concentration at each stage, and the results plotted. Similar absorption curves are made for solutions of the dye in 10% sulphuric acid and 10% sodium hydroxide, the series of curves thus obtained being characteristic for a particular dye. In order to identify the dye present in a food product, 20 g. of the latter are finely ground and extracted several times with methylated spirit. The residue is macerated with water, an equal volume of methylated spirit is added, and the solution filtered. The filtrate is concentrated, a large excess of absolute alcohol added to precipitate starch, and the filtrate from this added to the original extract. The united extracts are evaporated to dryness, the residue is dissolved in water, and the absorption curves are mapped and compared with those of known dyes. An approximate estimate of the amount of dye present can also be obtained in this way. The method is not suitable for the examination of black dyes, nor are those which show considerable general absorption. H. F. HARWOOD.

## PATENTS.

**[Manufacture of] vat dyes of the anthraquinone-acridone series.** O. UNGER and G. BÖHNER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,690,913, 6.11.28. Appl., 25.10.26. Ger., 29.10.25).—See B.P. 260,588; B., 1928, 225.

**Azo dyes and their manufacture.** E. HOFFA, E. THOMA, and H. HEYNA, Assrs. to GRASSELLI DYE-

STUFF CORP. (U.S.P. 1,698,884, 15.1.29. Appl., 11.7.27. Ger., 21.7.26).—See B.P. 296,473; B., 1928, 849.

**Monoazo dyes [for acetate silk] derived from naphthylaminocarboxylic [aminonaphthoic] acids.** L. LASKA and F. WEBER, Asssts. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,702,832, 19.2.29. Appl., 3.5.26. Ger., 8.5.25).—See B.P. 275,307; B., 1927, 776.

**Manufacture of indigoid dyes.** E. HOFFA, H. HEYNA, and F. MÜLLER, Asssts. to GEN. ANILINE WORKS, INC. (U.S.P. 1,730,209, 1.10.29. Appl., 30.11.26. Ger., 4.12.25).—See B.P. 262,457; B., 1927, 772.

**Dyes for leather** (B.P. 316,822).—See VI.

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

**Structure of vegetable fibres. I. Spiral structure of cotton, wood, manila, straw, bamboo, and sugar cane (bagasse) fibres.** M. NAKANO (J. Cellulose Inst., Tokyo, 1929, 5, 202—203).—The spiral structure (spiral cuticle) and the bead necklace effect on swelling in cuprammonium solution, which are both shown by cotton and wood fibres, are obtained with many other vegetable fibres such as manila, straw, bamboo, and sugar cane (bagasse). The spiral structure was successfully shown by swelling the fibres after weak xanthation. B. P. RIDGE.

**Hydrolysis of wool by sodium sulphide. II.** W. KÜSTER and W. IRION (Z. physiol. Chem., 1929, 184, 225—240; cf. A., 1928, 535).—The acetic acid precipitate from the sodium sulphide hydrolysate of wool contains less histidine than the original wool. Ammonia is produced during hydrolysis, although histidine itself is not deaminated by sodium sulphide. An attempt to prepare a histidine peptide by linking up benzoylhistidine methyl ester with hydrazine hydrate gave a crystalline *azlactone*, m.p. 215°. Of arginine, tyrosine, cystine, and asparagine, only the last splits off ammonia with sodium sulphide. No cystine could be isolated from the acetic acid precipitate, although the latter contained sulphur. This was shown to be present as a new dibasic *diamino-acid*,  $C_7H_{14}O_4N_2S$ , which crystallised from the neutralised hydrolysate after the tyrosine. When recrystallised from ammonia it formed tetragonal spheroids, m.p. 275° (decomp.). Further attempts to isolate this substance were unsuccessful. J. H. BIRKINSHAW.

**Decomposition of maize stalks by chlorine.** E. HORVÁTH (Papier-Fabr., 1929, 27, 626—639).—The cut-up stalks are treated with warm dilute sodium hydroxide solution, then with chlorine, and finally with cold sodium hydroxide solution. At the same temperature the amount of sodium hydroxide used up in steeping the material depends on the concentration of the liquor and on the time of treatment. With  $\frac{1}{2}$ , 1, or 2% solutions the reaction is completed in 90 min. If equal amounts of material are treated under the same conditions with equal quantities of 1 or 2% sodium hydroxide liquor, the products, after chlorination, consume almost equal amounts of sodium hydroxide, even if the amount of chlorine used is increased 8-fold. The yield obtained depends on the conditions of steeping and chlorination. With increasing

chlorine consumption the yield decreases quickly at first and then gradually. In all the experiments performed, under different conditions, the ash and silica contents of the final products were approximately the same. Loss of lignin increases with increasing chlorine consumption quickly at first, but subsequently reaches an approximately constant value. It is better to use a more concentrated sodium hydroxide solution in the first steeping process, followed by low chlorine consumption, than a dilute solution and high chlorine consumption, since in the former case greater dissolution of lignin results. B. P. RIDGE.

**Removal of fermentation-restraining humic substances from the hydrolysis products of hemicelluloses.** C. SCHMIDT, M. ATTERER, and H. THALER (Cellulosechem., 1929, 10, 153—155).—Humic substances, formed during the acid hydrolysis of hemicelluloses, which render completely inaccurate the determination of the hexoses by fermentation, may be removed by treatment with a dilute aqueous solution of chlorine dioxide. This treatment is shown not to affect the fermentation value of hexose in solution. The hemicelluloses from archegoniates and phanerogams (cf. Schmidt and co-workers, A., 1929, 1113) may be hydrolysed by refluxing with 5% oxalic acid; the resulting solution is neutralised, treated with a 1% solution of chlorine dioxide, after 12 hrs. extracted with ether, and again neutralised. The hexoses may now be determined by fermentation with *Schizosaccharomyces Pombe* or *Saccharomyces Vordermannii* (*loc. cit.*). To hydrolyse the hemicellulose from the wood fungus (*Fomes fomentarius*) it is necessary first to treat with 75% sulphuric acid at ordinary temperature, and then to reflux with 5—10% acid for 2½ hrs. The hexose may then be determined as described above. The hexose contents of hemicelluloses from different sources are given: *Pteridium aquilinum* 10.3%, rye straw 9.0%, *Fagus sylvatica* 8.3%, *Fomes fomentarius* 81.0%. T. H. MORTON.

## PATENTS.

**Manufacture of artificial textile filaments or fibres.** H. DREYFUS (B.P. 317,097—8, 6.2.28).—(A) Hollow filaments of good strength and capable of being drawn out to low deniers may be dry-spun from solutions of cellulose derivatives in organic solvents by introducing into the spinning liquor one or more high-boiling solvents or plasticisers in quantities varying from 3 to 80% (on the weight of the cellulose ester) and depending on the volatility of the high-boiling solvent. Suitable compounds include diacetone alcohol, acetophenone, di- and tri-acetin, ethyl phthalate, alkyl ethers of ethylene glycol, etc. The residual plasticising agent may be removed from the spun filaments by extraction with water or other suitable solvents. For a 24.8% solution of cellulose acetate in 3:1 acetone-diacetone alcohol 83° is a suitable spinning temperature. (B) The same result may be achieved by spinning a solution of cellulose acetate in a mixed solvent the components of which differ in their b.p. by 35—100° or more, the temperature of spinning being below or not substantially above the b.p. of the higher-boiling component of the spinning solution. The less is the

difference between the b.p. of the components of the solvent mixture the greater should be the proportion of the lower-boiling constituent and the lower may be the spinning temperature. Thus hollow filaments are obtained by spinning a 20.7% solution of cellulose acetate in a 1:3 acetone-acetaldehyde mixture at 65°, whilst at lower spinning temperatures cellular filaments result.

D. J. NORMAN.

**Packing material for soft soap.** H. FREHRS (B.P. 319,517, 15.10.28).—The packing paper is coated with a layer, impervious to gases and liquids, containing lime or a lime salt (*e.g.*, waxes or hard fats with calcium carbonate); on contact with the soft soap an insoluble lime soap layer, impervious to soft soap, is produced.

E. LEWKOWITSCH.

**Manufacture of paper.** A. L. KENNEDY, Assr. to PLASTIC, INC. (U.S.P. 1,730,009, 1.10.29. Appl., 25.3.27).—See B.P. 287,538; B., 1928, 668.

**Grinding-stones used on wood-pulping machines.** H. AKEROYD (B.P. 319,469, 22.8.28).

**Stabilisation of lead tetra-alkyl (U.S.P. 1,724,640).**—See VII.

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

**Reflecting power of coloured fabric.** CUNLIFFE and FARROW.—See XXI.

### PATENTS.

**Dyeing of cellulose derivatives.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 308,242, 13.12.27. Cf. B.P. 307,813; B., 1929, 639).—Cellulose esters and ethers are dyed with alkylthiolanthraquinones, *e.g.*, ethylthiolanthraquinone (yellow), 1:4-di(ethylthiol)-anthraquinone (orange), 1-amino-4-ethylthiolanthraquinone (bordeaux), 1-methylamino-2-ethylthiolanthraquinone (pink), 1-amino-2-isopropylthiolanthraquinone (red), 1-methylamino-2-isobutylthiolanthraquinone (pink).

C. HOLLINS.

**Dyeing of leather and manufacture of dyes for use therein.** J. C. BOTTOMLEY & EMERSON, LTD., and W. D. EARNSHAW (B.P. 316,822, 29.10.28).—Dyes produced by coupling 2 mols. of *o*- or *p*-toluidine, *m*- or *p*-xylydine, or their sulphonic acids, or 1 mol. each of any two of these substances with 1 mol. of resorcinol or  $\alpha$ - or  $\beta$ -resorcylic acid in the presence of caustic soda are suitable for dyeing leather in brown shades; those dyes containing sulphonic groups are more soluble in water and yield deeper shades. Dyeing is effected at 45° in an acid liquor on bark-, chrome-, or alum-tanned leather.

A. J. HALL.

**Treatment of textile materials.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 307,948, 14.9.27).—Hydroxyalkylamines, *e.g.*, "triethanolamine" [tri-( $\beta$ -hydroxyethyl)amine], cyclohexyl- $\beta$ -hydroxyethylamine, and cyclohexyldi-( $\beta$ -hydroxyethyl)amine, are added to aqueous baths for the cleansing, dyeing, carbonising, stripping, scouring, or bowking of textiles (in absence of gums). (Cf. B.P. 270,293; B., 1928, 704.) C. HOLLINS.

**Delustring of cellulose acetate silk, films, and the like.** SILVER SPRINGS BLEACHING & DYEING CO., LTD., and A. J. HALL (B.P. 316,169, 24.4.28).—Cellulose

acetate silk is treated so as to absorb the leuco-compounds of anthraquinone and such of its derivatives as are colourless in the fully oxidised state, and is then suitably oxidised. The fully oxidised anthraquinone compound is thereby deposited within the silk, which thus becomes reduced in lustre; the resultant delustring effect is fast to soaping. Anthraquinone and 2-chloro-anthraquinone are particularly suitable, and are applied from an alkaline reducing liquor containing ammonia and sodium hyposulphite. The silk may be dyed and delustred simultaneously if use is made of dyes such as Hydron Pink FF, which are capable of colouring cellulose acetate under such conditions.

A. J. HALL.

**Delustring of cellulose acetate silk materials and of other materials containing cellulose acetate.** SILVER SPRINGS BLEACHING & DYEING CO., LTD., and A. J. HALL (B.P. 316,638, 28.4.28).—The material containing cellulose acetate is treated with aqueous suspensions of esters of aliphatic acids, particularly ethyl oxalate, and then with ammonia, whereby oxamide is formed within the material, thus reducing its lustre and giving to it a scroop handle. The delustring is fast to soaping, and the process can be readily adapted for dyeing or printing methods.

A. J. HALL.

**Production of resists in dyeing with vat dyes.** J. HÖPKER, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,730,211, 1.10.29. Appl., 30.4.28. Ger., 6.5.27).—See B.P. 290,177; B., 1929, 849.

**Preparation of fulling liquors and emulsions [for softening of wool etc.].** K. DAIMLER, G. BALLE, and F. JUST, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,715,410, 4.6.29. Appl., 28.1.26. Ger., 28.1.25).—See B.P. 246,867; B., 1927, 451.

**Dye baths.** T. PARKINSON (B.P. 319,432, 6.7.28).

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

**Detection of hydrogen peroxide with potassium permanganate solution according to D.A.B. VI.** G. FRERICHS (Apoth.-Ztg., 1929, 44, 225—226; Chem. Zentr., 1929, i, 1974—1975).—A modification of the procedure is recommended.

A. A. ELDRIDGE.

**Manufacture of manganese borate.** V. V. ERIN (J. Chem. Ind. Moscow, 1929, 6, 254).—A mixture of finely-divided pyrolusite (9 pts.) and non-caking coal (1 pt.) is heated at 800—900°; the yield of manganous oxide is 85%. The hot mass is boiled with a 5—10% excess of 20% sulphuric acid for 1 hr., the solution is filtered through asbestos, the iron oxidised by boiling the solution with a 10% excess of nitric acid, and precipitated at 50° with sodium carbonate solution. The liquid, after settling, is filtered and treated with 4% borax solution, the end-point being denoted by the disappearance of cloudiness when a manganous salt solution is added to the filtered liquid. After settling, the precipitate is centrifuged, filter-pressed, and dried at 50°, then at 110°.

CHEMICAL ABSTRACTS.

**Oxygen and hydrogen in industry.** W. P. DOBSON and A. S. L. BARNES (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 11 pp.).—Methods of

production and fields of utilisation of oxygen and hydrogen are reviewed. Further possible outlets for these gases are considered and the prospects of the electrolytic process for producing them discussed.

H. J. T. ELLINGHAM.

**Determination of phosphorus in "Phosphor solutus" according to D.A.B. VI.** W. BÖTTGER (Apoth.-Ztg., 1928, 43, 1551—1553; Chem. Zentr., 1929, i, 1845).—Determinations based on titration of the acid liberated from an iodide-iodate mixture gave concordant results; iodometric determinations gave results 25% higher.

A. A. ELDRIDGE.

**Molybdenum-blue method for micro-determination of phosphate and arsenate ions.** G. DENIGÈS (Mikrochem., 1929, Pregl Fest., 27—45).—An acid solution of ammonium molybdate gives in presence of phosphate or arsenate ions on treatment with reducing agents stable blue compounds of the composition  $(4\text{MoO}_3 \cdot \text{MoO}_2)_2 \cdot \text{XO}_4\text{H}_3$ , where X = P or As. A colorimetric method, based on the above reaction, has been devised for the determination of phosphate in wines and other beverages; the determination is carried out on the original sample without previous destruction of organic matter, so that the figure obtained represents the mineral phosphate present. The total phosphate can be found by carrying out a second determination after removal of organic matter. The method is also applicable to the determination of phosphate in urine, milk, and other physiological products, as well as to fertilisers and mineral phosphates. Arsenate may be determined in a similar manner.

H. F. HARWOOD.

**Rapid determination of nitrogen.** F. M. WIENINGER and M. LINDEMANN (Woch. Brau., 1929, 46, 406—407).—The methods of Lundin and Ellburg (B., 1929, 596) and Kühl and Gottschalk (Z. ges. Getreidewesen, 1929, 114—115) are accurate and rapidly completed, but they require as much attention as the ordinary Kjeldahl method. The use of phosphoric acid in the digestion mixture causes appreciable loss of material from the flasks. Until a rapid accurate method for the determination of moisture is devised such processes are of little advantage in cereal analyses, and in this direction the "D.K." (condenser) method (B., 1929, 373) appears promising.

F. E. DAY.

**Peat as a source of nitrogen.** LOGVINOVA. Fuller's earth and acid-treated earths. DAVIS and MESSER.—See II. Catalysts for methyl alcohol synthesis. CRYDER and FROLICH.—See III. Selenium and its compounds for ruby glass. KRAK.—See VIII. Precipitation of lead and copper from solutions. OLDRIGHT and others.—See X. Electrolysis of water. SCHNURMANN.—See XI. Antiseptic value of chlorine. HOWARD and others.—See XV. Iron albuminate. OBERHARD.—See XX.

#### PATENTS.

**Catalytic reactions.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 315,900, 13.4.28).—Catalysts for reactions in which hydrogen is employed under pressure, e.g., the synthesis of ammonia, are, prior to the reaction, irradiated by Röntgen rays, rays of radioactive substances, etc. in the presence of inert gases

or of gases, vapours, or liquids of the same nature as those which are to take part in the catalytic reaction, the catalysts being in the same physical and chemical state as that in which their activity is to be exerted.

W. G. CAREY.

**Obtaining sodium chloride.** G. B. BURNHAM, Assr. to BURNHAM CHEM. CO. (U.S.P. 1,701,295, 5.2.29. Appl., 16.11.25).—The brine is brought to supersaturation by, e.g., atmospheric evaporation, and circulated upwardly to a conical tank and through crystals deposited on a perforated partition a short distance above the bottom (apex) of the tank. Liquor overflowing at the top of the tank is returned to the evaporating pond, and crystals fall through the partition into a vertical collecting conduit parallel with the inlet conduit and are removed to storage by a conveyor.

R. BRIGHTMAN.

**Refining of borax.** C. F. RITCHIE and W. A. GALE, Assrs. to AMER. POTASH & CHEM. CORP. (U.S.P. 1,724,420, 13.8.29. Appl., 27.1.25).—Liquors containing phosphates and borates are treated with a reagent which by reducing the  $p_{\text{H}}$  value increases the solubility of the phosphates and thus facilitates the crystallisation of borates free from phosphates.

L. A. COLES.

**Manufacture of sodium xanthate.** W. HIRSCHKIND, Assr. to GREAT WESTERN ELECTRO-CHEM. CO. (U.S.P. 1,701,264, 5.2.29. Appl., 28.11.24).—Ethyl alcohol (91% denatured) is added to 46% sodium hydroxide solution, and carbon disulphide is added at 25—30°. The crystalline xanthate ( $2\text{H}_2\text{O}$ ) is centrifuged and the mother-liquor treated cold with 55% potassium hydroxide to give a further precipitate of potassium xanthate. The final mother-liquor containing, e.g., 16% of caustic alkali, is useful as an auxiliary froth-floatation agent.

R. BRIGHTMAN.

**Manufacture of alkali xanthates.** T. W. BARTRAM and H. P. ROBERTS, Assrs. to RUBBER SERVICE LABS. CO. (U.S.P. 1,718,937, 2.7.29. Appl., 8.11.26).—Carbon disulphide is added during 50—1000 min. to alcoholic alkali (1 : 1 mol.) at 0—50°, and the mixture is heated below 70° to decompose by-products.

C. HOLLINS.

**Manufacture of inorganic [iron] oxides [for pigments].** E. H. MCLEOD, Assr. to AULT & WIBORG CO. OF NEW YORK (U.S.P. 1,726,851—2, 3.9.29. Appl., 26.10.22).—(A) A solution of ferrous sulphate, e.g., waste pickling liquor, is oxidised with bleaching powder and treated with sodium carbonate to precipitate a mixture of calcium carbonate and ferric hydroxide. The precipitate is agitated with a further quantity of ferrous sulphate to produce, by interaction with the calcium carbonate and ferric hydroxide, a mixture of ferrosiferrous oxide and calcium sulphate of any desired tint. (B) Burnt pyrites cinder is dissolved in hydrochloric acid, sodium carbonate is added in excess, and the mixture treated at 82° with ferrous sulphate.

A. R. POWELL.

**Stabilisation of lead tetra-alkyl and its compositions.** W. S. CALCOTT and A. E. PARMELEE, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,724,640, 13.8.29. Appl., 19.6.28).—Primary, secondary, or tertiary alkylamines or their derivatives are used; e.g.,

1% of methylamine or diethylamine is added to a mixture of lead tetraethyl, ethylene dibromide, and chloronaphthalene. R. BRIGHTMAN.

**Manufacture of silica or masses containing the same.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 315,675, 16.3.28. Addn. to B.P. 270,040; B., 1927, 481).—A decomposable silicon compound and a decomposing agent are mixed at a low temperature in a single pouring action within a few seconds or by simultaneous confluence in such concentration that a non-alkaline silica sol is produced containing at least 9 g. of silica/100 c.c. of total liquid. W. G. CAREY.

**Production of gas strong in sulphur dioxide.** G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,724,421, 13.8.29. Appl., 26.3.23).—Sulphur burner gases are passed through a confined space in which they are brought in contact with non-reacting surface material and a countercurrent of water, and the solution obtained is subsequently heated and treated in a confined space in the presence of non-reacting material with a countercurrent of the residual gas from the first treatment to yield a gas mixture with a high sulphur dioxide content. L. A. COLES.

**Production of phosphorus.** W. H. WAGGAMAN and H. W. EASTERWOOD, Assrs. to VICTOR CHEM. WORKS (U.S.P. 1,728,948, 24.9.29. Appl., 17.1.24).—Briquetted mixtures of natural phosphates, silica, and a solid reducing agent are heated in continuous rotary kilns to not above 1500°, the composition of the mixture being such as not to be readily fusible at this temperature. C. A. KING.

**Production of sodium nitrate.** R. GRIESSBACH and K. RÖHRE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,696,197, 25.12.28. Appl., 22.9.27. Ger., 14.10.26).—See B.P. 290,860; B., 1928, 522.

**Purification of chemicals [used in preparation of per-salts, etc.].** W. WEBER and M. JACOBI (U.S.P. 1,722,871, 30.7.29. Appl., 7.9.27. Ger., 24.12.26).—See B.P. 282,302; B., 1928, 157.

**Protection of pipes (U.S.P. 1,700,995—6). Treatment of ores for separation of salts (B.P. 318,301).—See X. Electrolytic cells (B.P. 292,130).—See XI. Base-interchange agent (B.P. 286,307).—See XXIII.**

## VIII.—GLASS; CERAMICS.

**Volatility of selenium and its compounds in the manufacture of ruby glass.** J. B. KRAK (J. Amer. Ceram. Soc., 1929, 12, 530—537).—Glass batches were melted in a gas-fired furnace to determine the amount of selenium lost in making ruby glass, to investigate the conditions for producing "pot" ruby, and to discover whether the use of selenium compounds decreases the loss of selenium. About 75% of the selenium added to a batch was lost by volatilisation, this amount varying with the composition of the batch. Although sodium and barium selenite are stable at 950°, no saving of selenite was effected by substituting either of these compounds for the selenium itself. Methods of preparing sodium and barium selenites and of determining selenium in glass are described. F. SALT.

**Viscosity measurements in glass.** H. R. LILLIE (J. Amer. Ceram. Soc., 1929, 12, 516—529).—For low

viscosities (10—10<sup>4</sup> poises) the method already described (*ibid.*, 505) was used. For higher viscosities (10<sup>8</sup>—10<sup>8</sup>) the same apparatus was used, but the outer cylinder was turned through a small angle and stopped, the return of the inner spindle towards its point of rest being then timed. The tests were carried out in a molybdenum-wound furnace, in which temperatures between 1400° and 1500° were attained on 110 volts. The glass samples were in the form of cylinders, 5 $\frac{3}{4}$  in. long and 2 $\frac{1}{4}$  in. in diam. The results obtained are compared with those published by English, Stott, and by Washburn. F. SALT.

**Deformation study of cobalt oxide-alumina-silica mixtures.** T. M. FELTON (J. Amer. Ceram. Soc., 1929, 12, 548—551).—The study was undertaken to investigate the tendency of cobalt oxide to "spread" in glazes. The various mixtures were made up into cones and the temperatures of deformation and of fusion were noted. The deformation eutectic temperature of the cobalt oxide-silica series was 1325°, the composition being 32% SiO<sub>2</sub> and 68% Co<sub>3</sub>O<sub>4</sub>. The composition of the alumina-cobalt oxide deformation eutectic was approx. 10% Al<sub>2</sub>O<sub>3</sub> and 90% Co<sub>3</sub>O<sub>4</sub>; that of the cobalt oxide-alumina-silica system was 38% SiO<sub>2</sub>, 20% Al<sub>2</sub>O<sub>3</sub>, and 42% Co<sub>3</sub>O<sub>4</sub>, the temperature being 1200°. The addition of silica in amounts up to 70% greatly reduced the melting temperatures of mixtures of alumina and cobaltocobaltic oxide. In the presence of alumina and silica, cobalt oxide is an active flux, which may account for the "spreading" tendency in glazes. F. SALT.

**Capillary suction of some ceramic materials.** A. E. R. WESTMAN (J. Amer. Ceram. Soc., 1929, 12, 585—595).—A simple experiment with a porous pot, a graduated glass tube, and a flask containing mercury is described, by means of which capillary suction can be demonstrated. Evaporation from the surface of a saturated clay causes compression of the material with the development of tension in the pore water and a retreat of the water-to-air contact surface from the outside of the material toward the interior. Capillary suction is defined as the maximum difference which can exist between the pressure of the pore water and the pressure of the air in contact with the material without causing the water-to-air contact to retreat to the interior. Apparatus is described for determining this maximum pressure by using air at pressures greater than atmospheric and keeping the pore water at atmospheric pressure. The capillary suction of flint, felspar, ball clay, kaolin, and mixtures of these were determined. The addition of non-plastic material to clays caused a marked decrease in capillary suction. The figure obtained for ball clay was three times that for kaolin. Owing to capillary suction, water under atmospheric pressure was made to enter a bomb in which the gas pressure was 200 lb./in.<sup>2</sup> F. SALT.

**Burning of calcareous clays.** O. LECHER (Chem.-Ztg., 1929, 53, 669—671).—In the form of gypsum, the presence of calcium in a clay is definitely undesirable for the production of ceramic ware. Calcium is usually present as the carbonate, and in this form its effect depends largely on its physical condition. It is practically impossible to grind coarse nodules of lime in a

clay to sufficient fineness to render them harmless. When the clay is fired, lime particles, even as small as 1 mm. and less, are not completely "dead-burned," but are partially converted into quicklime. The most effective way of removing coarse grains of limestone is by washing the clay. If in a finely-divided state, lime may be present in considerable amount in clays used in making face-bricks and tiles, stove tiles, etc. It largely destroys the colouring effect of iron oxide in such clays fired to high temperatures, but the bricks produced have great crushing strength and density. The firing of clays rich in lime is somewhat difficult, because the softening point and the m.p. of the clay are near to each other. The temperature of the kiln must therefore be reasonably uniform; hence down-draught kilns are not suitable, and some modification of the annular kiln is necessary to fire these clays successfully. F. SALT.

**Torsional apparatus for measuring plasticity [of clays].** C. W. PARMELEE and R. D. RUDD (J. Amer. Ceram. Soc., 1929, 12, 552—555).—Methods and apparatus previously described (cf. Talwalkar and Parmelee, B., 1928, 193) were critically examined and altered to reduce variations due to personal manipulation. The specimens were made in squeeze moulds instead of by hand. The shape of the test-piece was changed so that the ratio of the stress in the outer fibres of the square end-section to that of the circular portion was 1:3.8. The method of applying the load by adding weights to a suspended pan was abandoned in favour of a flowing-water device, which applied the load continuously. Data obtained on a ball clay and a china clay are briefly analysed. F. SALT.

**Liquefaction of difficultly workable fireclays.** P. P. BUDNIKOV, S. A. SCHCHAREVITSCH, and I. G. SCHACHNOVITSCH (Kolloid-Z., 1929, 49, 174—178).—Measurements have been made of the rate of flow of clays diluted with water with the addition of varying quantities of water-glass. An optimum concentration of water-glass exists for each dilution with water. The minimal amount of water required was with a concentration of water-glass of 0.1—0.125*N*. Hard water must be given a preliminary softening. E. S. HEDGES.

## PATENTS.

**Tunnel kiln.** W. L. HANLEY, JUN. (U.S.P. 1,720,550, 9.7.29. Appl., 11.9.25. Renewed 11.12.28).—The kiln comprises two consecutive firing zones, in addition to the preheating and cooling zones. Each firing zone is heated by separate, directly-fired furnaces, which are on opposite sides of the kiln and can be regulated so as to give a maximum temperature at a point between the two firing zones. The combustion products enter the latter directly and are withdrawn through a series of ports. Two parallel conveyors, movable in opposite directions throughout the entire length of the kiln, are provided.

F. G. CLARKE.

**Refractory product.** J. T. LITTLETON, JUN., Assr. to CORNING GLASS WORKS (U.S.P. 1,728,350, 17.9.29. Appl., 8.6.23).—Suitable material is melted and poured into a mould, unheated material of approximately the same composition in granular form being then added to the molten material, which is thus cooled, and a heterogeneous crystalline mass is formed. F. SALT.

**Repairing of refractory walls.** D. W. ROSS and J. M. LAMBIE (U.S.P. 1,727,675, 10.9.29. Appl., 27.7.25. Renewed 12.4.29).—A mixture of a granular aluminous material, a flux, and a bonding clay is applied to the inner surface of glass-tank walls, and the tanks are heated to the point where mullite crystals are formed. F. SALT.

## IX.—BUILDING MATERIALS.

**Florescence [in building materials].** W. A. MCINTYRE and R. J. SCHAFER (Trans. Ceram. Soc., 1929, 28, 363—380).—The term "florescence" is used to cover the general phenomenon of the crystallisation of soluble salts in building materials, the term "efflorescence" being restricted to crystallisations on the surface. The word "cryptoflorescence" is suggested to describe the salts concealed in the body of the material. Most florescences consist of the sulphates of the alkalis and alkaline earths, but they vary in composition within wide limits. The sources of soluble salts which cause florescence are indicated, and the forces causing disintegration are discussed. The chemical action of atmospheric acids in dissolving out certain constituents weakens the structure; salts which crystallise with a decrease in volume may exercise a powerful disruptive force by axial growth of the crystals. The expansion of sodium sulphate on hydration may be a contributory cause of disruption, and soluble salts, such as sodium sulphate, may have a disruptive effect by accelerating the hydration of calcium sulphate present in the body of a brick. The use of chemicals to remove florescences from brickwork is deprecated, periodic washing with water being advocated. F. SALT.

**Tensile autogenous heating of Portland cement mixtures.** H. J. GILKEY (Engineering, 1929, 128, 450—452).—Broken briquettes made and used for tensile strength tests generally "heal" if the broken parts are refitted and kept in position by suitable binding, and allowed to remain in water. The tensile strength of such healed briquettes is seldom more than 10% of the original strength, but much higher recoveries have been noted. Healing is due to formation of crystalline silicates, hydrates, and carbonates of calcium which form across the break. S. I. LEVY.

**Respiratory diseases in a Portland cement plant.** L. R. THOMPSON and D. K. BRUNDAGE (J. Ind. Hygiene, 1929, 11, 266—277).

**Corrosion of lead pipes in cement etc.** BADO.—See X.

## PATENTS.

**Manufacture and use of oxysalt [calcium oxychloride] composition.** C. CATLETT (U.S.P. 1,726,472, 27.8.29. Appl., 26.7.23).—A mixture of calcium chloride and hydroxide is ground with 2% or less of an oleaginous material, e.g., cottonseed oil or beef tallow. The substantially dry product, which is not sensitive to atmospheric conditions, is used to regulate the setting of Portland cement. F. G. CLARKE.

**[Bituminous mixture for] the making of roads, paths, etc.** J. T. HINES (B.P. 316,017, 31.7.28).—Stone is sprayed with a fluxing oil containing sulphur (2—10%) and then mixed mechanically with commercial bitumen

and Trinidad bitumen containing clay in a colloidal state; fine mineral filler is subsequently added. Alternatively, the fluxing oil and bitumens are mixed at a suitable temperature and the stone is treated with this mixture.

W. G. CAREY.

**Hydraulic cement.** C. PONTOPPIDAN (B.P. 293,035, 23.6.28. U.S., 30.6.27).—See U.S.P. 1,722,480; B., 1929, 816.

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

**Heat and material balance of some melts in the Brackelsberg furnace.** P. BARDENHEUER and K. L. ZEYEN (Stahl u. Eisen, 1929, 49, 1393—1398).—The advantages of the Brackelsberg coal-dust-fired furnace for melting cast iron and the economics of the process are discussed. The metal becomes coated with a thin layer of slag directly it melts so that absorption of oxide and gases and oxidation of the metal are avoided. When tapped into the ladle the metal remains quiet and the castings are free from pores and internal cavities. Owing to its high degree of fluidity the metal is especially suitable for making thin-walled castings and articles of intricate shape. The consumption of coal dust with 8.2% of ash and a calorific value of 7309 kg.-cal./kg. varied from 9.6 to 14.3% of the weight of metal melted, the slag produced was 5—6—9.2%, and the reduction in the carbon content of the metal about 0.5%. Of the total heat generated, 30—40% was taken up by the iron, 2.5—4.5% by the slag, and 55—45% by the flue gases, the loss by incomplete combustion being 4—8%. Full heat-balance sheets of the three tests made are reproduced.

A. R. POWELL.

**Microscopical study of cast iron and its relation to the foundry.** E. HOWELL (J. Soc. Chem. Ind. Victoria, 1927, 27, 1397—1407).—Improved physical strength of cast iron is aimed at by both the Lanz and the Emmel-Thyssen processes, the former restraining the formation of graphitic carbon by adopting a low-silicon iron (1% Si) and casting in preheated moulds, and the latter depending on the superheating of the metal together with low carbon content. Addition of steel scrap to the cupola charge does not decrease the total carbon below 3% owing to absorption of carbon from the fuel; for this reason good free-burning coke should be used to obtain a finely-divided graphite together with a close-grained pearlitic structure in the casting.

C. A. KING.

**Testing of permanent-magnet steel.** W. OERTEL (Stahl u. Eisen, 1929, 49, 1449—1454).—A detailed description is given with reference to photographs and wiring diagrams of the construction, standardisation, and method of operation of a new Bosch magnet-testing apparatus of the type in which a thick soft-iron yoke with a revolving coil is used for measuring the intensity of the lines of force.

A. R. POWELL.

**Effect of alumina on the properties of steel-furnace slags.** G. MARS (Arch. Eisenhüttenw., 1929—30, 3, 103—116; Stahl u. Eisen, 1929, 49, 1339—1340).—The replacement by alumina of part of the lime in the refining slag of a basic electric furnace and of a Siemens-

Martin furnace reduces the dephosphorising action almost to zero, especially when less than 20% of ferrous oxide is present. On the other hand, an aluminous slag effects a very efficient desulphurising and deoxidising action owing to the formation of aluminium carbide which dissolves in the molten steel, giving up its carbon to the iron and producing aluminium sulphide and oxide which enter the slag; the desulphurising action is, however, obtained only in the electric furnace and not in the open-hearth furnace. Owing to the affinity of alumina for ferrous oxide at high temperatures, the addition of alumina to open-hearth slag results in a more efficient removal of ferrous oxide from the molten iron and thus reduces the tendency to hot-shortness. As a result of these experiments a mixture of 90% of lime and 10% of alumina is recommended as a basic lining for steel-refining furnaces.

A. R. POWELL.

**Determination of manganese in steel by Wald's method.** J. KASSLER (Chem.-Ztg., 1929, 53, 719).—The following modification of Volhard's method as modified by Wald is recommended for the determination of manganese in chromium, nickel-chromium, and high-speed tool steels containing less than 0.2% Co. A sample of the metal (2.75 g.) is dissolved in 50 c.c. of 1:5 sulphuric acid, the solution is oxidised with 10 c.c. of 1:1 nitric acid, and the iron precipitated with zinc oxide. After dilution to 250 c.c. an aliquot portion (100 c.c.) is filtered through a dry paper, treated with 20 c.c. of 25% sodium acetate solution, boiled, and titrated with a solution containing 0.79 g./litre of permanganate until a slight excess is present, which is then reduced by the cautious addition of 10% alcohol. The aldehyde formed is expelled by boiling for 5 min., and the solution cooled and treated with 10 c.c. of 5% potassium iodide solution and 20 c.c. of hydrochloric acid; the liberated iodine is then titrated with thiosulphate and the amount due to the manganese added as permanganate is deducted. The results are correct to  $\pm 0.005\%$  Mn.

A. R. POWELL.

**Detection of sulphide segregations in the presence of phosphide segregations [in iron and steel] by Feigl's reagents.** M. NIESSNER (Arch. Eisenhüttenw., 1929—30, 3, 157—161; Stahl u. Eisen, 1929, 49, 1378—1379). The mercuric chloride method for detecting sulphide and phosphide segregations in iron and steel does not give characteristic black prints for sulphide when the amount of sulphur present is small, owing to the formation of orange to yellow sulphochlorides of mercury instead of black mercuric sulphide. These spots can be distinguished from phosphide spots on the dried print by placing on them a drop of a solution 0.1N with respect to potassium iodide and 0.2N with respect to sodium azide. If sulphur is present small bubbles of nitrogen will form in the drop within a few seconds (cf. Feigl, A., 1928, 1106); phosphides give no reaction.

A. R. POWELL.

**Soil corrosion studies, 1927—8.** K. H. LOGAN (Bur. Stand. J. Res., 1929, 3, 275—302. Cf. B., 1929, 56).—Further data on ferrous pipe and lead cable-sheath buried in various soils in 1922, and unearthed in 1928, are given. Previous conclusions are confirmed, the rate of pitting and of loss of weight decreasing with



time. The later corrosion is largely the result of general spreading of the corroded areas, penetration progressing more slowly than loss in weight. Part of the corrosion is due to electrolytic action when a pipe passes through two different soils. No conclusions as to the relative merit of different materials are yet justified.

C. J. SMITHELLS.

**[Corrosion of] lead pipes embedded in cement or lime mortars.** A. A. BADO (Anal. Asoc. Quím. Argentina, 1929, 17, 5—12).—Instances of corrosion of lead pipes set in damp mortar are described. The corroded metal contains a high proportion of carbonate (13.2%  $\text{CO}_2$ ), and is coloured by oxides of lead. In comparative experiments with pipes set in (a) 1 pt. of artificial Portland cement and 5 pts. of sand, (b) 1 pt. of artificial Portland cement, 1 pt. of lime, and 5 pts. of sand, and (c) 1 pt. of hydraulic cement and 5 pts. of sand, most corrosion occurred with mixture (b) and least with (c). It is suggested that lead pipes in damp positions should be set in a jacket of clay, or be treated with asphalt or pitch varnish.

R. K. CALLOW.

**Final report on the relative corrodibilities of various commercial forms of iron and steel. Results of prolonged exposure to the atmosphere, and general conclusions from all the tests.** J. N. FRIEND (Iron & Steel Inst., Carnegie Schol. Mem., 1929, 18, 61—71; cf. B., 1927, 843).—The loss in weight and the effect of corrosion on the surface appearance of bars of cast iron, wrought iron, and various plain carbon and alloy steels, after exposure for seven years in the Birmingham atmosphere, and after exposure for six years in the atmosphere of a gas works, have been determined. The wrought-iron specimens showed a tendency to corrode in furrows along the bar, and slight furrowing was also noticed with the plain carbon and nickel-chromium steels; all these metals suffered heavy losses in weight. Steels with 0.12% Si and those with 0.15% Cu had very smooth surfaces, and lost much less in weight, stainless steels were very little affected, manganese steel became pock-marked all over, and cast irons developed a rough surface, but were otherwise in good condition, without any signs of graphitisation. The tests made at the gas works showed similar results, but the losses in weight were generally much more severe. The most important conclusions drawn from all the tests described in the four reports are summarised.

A. R. POWELL.

**Metals in dairy equipment; corrosion caused by washing powders, chemical sterilisers, and refrigerating brines.** O. F. HUNZIKER, W. A. CORDS, and B. H. NISSEN (J. Dairy Sci., 1929, 12, 252—284).—The effect of solutions of sodium hydroxide, carbonate, phosphate, hypochlorite, chloride, chromate, and silicate, calcium chloride, chloramine-*T*, and commercial products on aluminium, tinned copper, tinned iron, galvanised iron, tin, nickel, resistant steel, and commercial alloys was studied.

CHEMICAL ABSTRACTS.

**Precipitation of lead and copper from solution on sponge iron.** G. L. OLDRIGHT, H. E. KEYES, V. MILLER, and W. A. SLOAN (U.S. Bur. Mines Bull. No. 281, 1928, 131 pp.).—Sponge iron is produced by the reduction of magnetite or hæmatite with carbon monoxide at

950°. It resembles porous, coarse sand and offers a large amount of surface. Lead is usually extracted by wet processes as chloride, and the precipitation of lead chloride by sponge iron was therefore studied. It was found that for quick precipitation a temperature of 60° is desirable. Some agitation is necessary, but too vigorous stirring produces balling. Excess of iron has little effect. Percolation methods were unsuccessful, and agitation with air oxidises the iron. The best results were obtained by slowly rabbling sponge iron downward over a series of trays against an upward current of lead solution. Using commercial solutions, complete stripping from lead was obtained, together with a product containing about 70% Pb. For this purpose 30% over the theoretical of iron was used. Comparative results obtained with scrap iron (as generally used) show that a considerable saving in labour cost would be obtained. The precipitation of copper from sulphate solutions was also studied. It was found that sponge iron precipitates copper from a given solution much faster than does scrap iron, but the product is less pure. An excess of 7% over the theoretical amount of iron is usually sufficient. The temperature rises rapidly during precipitation and is not easily controlled. Rate of precipitation increases with temperature. The temperature should later be allowed to fall or redissolution of copper may occur. An increase of acidity increases the rate of precipitation in richer solutions, but decreases it in poorer solutions. Dissolution of copper is associated with the action of air drawn in by agitation; it is negligible if air is excluded. The possibility of a continuous countercurrent method for copper is discussed. The type of precipitator operating on the principle of the MacDougall furnace, and described above as applied to lead, appears satisfactory, but much more thorough agitation is required than with lead.

C. IRWIN.

**Bearing metals with a lead-antimony-tin basis.** H. MÜLLER (Z. Metallk., 1929, 21, 305—309).—The properties and structure of bearing metals with a high lead content are described, and the effect of adding small percentages of the various copper or nickel antimonides, either alone or together, to eutectic alloys of the system lead-tin-antimony is illustrated by photomicrographs. The compound  $\text{Cu}_2\text{Sb}$  appears unchanged as needles in the ground mass of eutectic, whereas the compounds  $\text{Cu}_3\text{Sb}$  and  $\text{Cu}_5\text{Sb}_2$  are both converted into  $\text{Cu}_3\text{Sb}$  and the ground mass becomes hypoeutectiferous with small lead areas irregularly distributed throughout. Of the nickel antimonides  $\text{NiSb}$  alone remains unchanged in the eutectic alloy,  $\text{Ni}_2\text{Sb}_3$  giving up antimony to form  $\delta$ -tin-antimony crystals and  $\text{Ni}_5\text{Sb}_2$ , and  $\text{Ni}_4\text{Sb}$  taking up antimony from the eutectic. Hence, in the systematic investigations of these alloys only the compounds  $\text{Cu}_2\text{Sb}$  and  $\text{NiSb}$  need be taken into account. The binary system  $\text{Cu}_2\text{Sb}$ - $\text{NiSb}$  contains no double compounds, but  $\text{NiSb}$  crystals retain about 5%  $\text{Cu}_2\text{Sb}$  in solid solution;  $\text{NiSb}$  affects the temperature of the  $\beta$ - and  $\delta$ -transformations of  $\text{Cu}_2\text{Sb}$  and also causes a more regular distribution of the copper compound in lead bearing metals.

A. R. POWELL.

**Rapid determination of lead.** P. F. THOMPSON (J. Soc. Chem. Ind. Victoria, 1929, 29, 154—159).—

Lead in solution may be determined directly by titration with ammonium molybdate without previous separation as sulphate. The ore is dissolved in nitric acid, the solution evaporated, and acetic acid added, followed by ammonia until the liquid is practically neutral. The diluted solution is then heated to boiling and titrated rapidly with standard molybdate solution, using as external indicator a 1:300 solution of tannic acid containing 10% of potassium fluoride and a little acetic acid. Among the ordinary metals only iron, copper, cobalt, and nickel interfere; the effect of iron is eliminated by the addition of potassium fluoride to the tannic acid indicator as above, whilst if copper be present the indicator should contain some potassium cyanide. A determination of lead in galena can be carried out by the above method in 12 min., and the results appear to be as accurate as those obtained by the original long method. (Cf. Dawkins and Weldon, B., 1924, 670.)

H. F. HARWOOD.

**Possible use of beryllium in aircraft construction.** H. W. GILLET (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 4 pp.).—The physical properties of beryllium are briefly reviewed and possible applications of the metal and its alloys discussed. An outstanding property of beryllium is its high modulus of elasticity.

H. J. T. ELLINGHAM.

**Comparison between sodium cyanide and potassium cyanide silver-plating solutions.** E. B. SANIGAR (Metal Ind., 1929, 34, 539—540).—The plated articles were indistinguishable; the finishing losses were slightly less for sodium cyanide.

CHEMICAL ABSTRACTS.

**Effect of current density on the hardness of electrodeposited chromium.** R. J. PIERSON (Amer. Electrochem. Soc., Sept., 1929. Advance copy. 6 pp.).—Chromium is electrodeposited on steel sheets from a bath containing 250 g. of chromic acid and 3 g. of chromic sulphate per litre at 49°, using various current densities ranging from 16 to 109 amp./dm.<sup>2</sup> The hardness of the deposits is measured by the time required for an emery wheel, operated under standardised conditions, to cut through a plate 0.025 mm. thick. The hardness is found to increase enormously with current density up to about 62 amp./dm.<sup>2</sup>, after which it falls off somewhat. The maximum hardness observed was 43 times that of a deposit produced at 16 amp./dm.<sup>2</sup>

H. J. T. ELLINGHAM.

**Properties of industrial gold alloys.** E. A. SMITH (Metal Ind., 1929, 34, 342—344, 352, 373—374).

#### PATENTS.

**Blast furnace.** C. L. T. EDWARDS, Assr. to BETHLEHEM STEEL Co. (U.S.P. 1,727,100, 3.9.29. Appl., 23.10.24).—In the smelting of ores in a blast furnace, the pressure in the shaft is increased by the imposition of back pressure without impeding the free escape of solids entrained by the combustion gases.

A. R. POWELL.

**Annealing furnaces [for wire].** INTERNAT. GEN. ELECTRIC Co., Inc., Asses. of ALLGEM. ELEKTRIZITÄTS-GES. (B.P. 300,242, 9.11.28. Ger., 9.11.27).—The furnace comprises an electrically-heated tube, inclined downwards at an angle of about 30° to the horizontal, the

upper end of the tube outside the heating zone being bent downwards at right angles to the rest of the tube. A small vertical tube entering the main tube in the underside of the bend provides a means of passing a protective gas, e.g., hydrogen, through the furnace.

A. R. POWELL.

**Protecting pipes from corrosion.** FREEPORT SULPHUR Co., Asses. of (A) H. S. BURNS and L. S. BUSHNELL, (B) L. S. BUSHNELL (U.S.P. 1,700,995—6, 5.2.29. Appl., [A] 13.7.26, [B] 4.5.28).—(A) The surfaces of pipes for conveying corrosive liquids, e.g., "fermentation" water in the underground fusion system of mining sulphur, are protected by first forcing through the pipes a hot solution of calcium and magnesium salts, e.g., sulphates and carbonates, to deposit a scale on the pipes. (B) Suitable apparatus is described.

R. BRIGHTMAN.

**Hardening of tool steels.** (SIR) A. HERBERT and A. H. LLOYD (B.P. 318,060, 28.11.28).—Tool steel containing, e.g., 5% Cr, 17% W, 13.5% Co, and 1.25% V is heated at 1310—1340°, cooled in an air blast, reheated, and again cooled as before, and finally tempered at 600° for 30 min.

A. R. POWELL.

**Galvanising of steel or iron sheets.** G. S. MALIPHANT and F. J. REES (B.P. 318,806, 8.12.28).—A bath for galvanising iron or steel sheets comprises a rectangular vessel filled with molten zinc into which protrudes one arm of a broad U-shaped trough filled with molten lead and terminating inside the zinc bath some distance below the surface of the zinc and outside the zinc bath, either well above the zinc surface or in a second rectangular vessel forming a lead supply bath. The sheets are passed through the lead trough into the zinc, which is covered with a protective flux, thence out of the zinc through a pair of rollers partially immersed in the zinc.

A. R. POWELL.

**Welding of rails or the like by means of superheated molten metal.** ELEKTRO-THERMIT GES.M.B.H., and A. G. CRÜM (B.P. 318,705, 10.7.28).—Molten steel or iron is poured into a mould surrounding the parts to be welded, and oxygen or chlorine is forced through it to cause superheating by the vigorous exothermic action which ensures. The vigour of the reaction may be increased by adding readily oxidisable substances such as carbon, aluminium, manganese, or silicon to the molten iron.

A. R. POWELL.

**Fluxes for joining metals by welding, soldering, etc.** K. WERNER, and "PROGRESS" GES. FÜR TEXTILMASCHINEN M.B.H. (B.P. 318,377, 22.8.28).—A flux for hard soldering, brazing, or welding is made by mixing the powdered product obtained by melting equal parts of brass turnings, lead, and borax with one third of its weight of borax, one third of its weight of sodium carbonate, and one fifteenth of its weight of sodium chloride. The borax and sodium carbonate may be replaced by equal weights of potassium and ammonium sulphates.

A. R. POWELL.

**Heat-treatment of oxidised copper ores.** T. J. and B. TAPLIN, and METALS PRODUCTION, LTD. (B.P. 318,314, 5.6.28).—The gases evolved in the chloridising treatment claimed in B.P. 250,991 (B., 1926, 590) are passed over copper carbonate ores or limestone to absorb

the hydrogen chloride present, and the resulting products are used again in the chloridising treatment.

A. R. POWELL.

**Treatment of ores etc. for the separation of metals and their salts.** E. A. ASHCROFT (B.P. 318,301, 5.4., 27.7., 20.8.28, and 29.1.29).—Oxidised ores containing lead, zinc, and copper are heated with ammonium chloride at 300–350°, whereby ammonia is evolved and may be collected in water or converted into carbonate, and chlorides of zinc, lead, and copper are produced. The chloridised product is leached with water, the lead chloride collected on a filter, and the solution further purified by treatment with hydrogen sulphide or with zinc dust. Zinc is then precipitated by careful addition of ammonium carbonate; if lime or magnesia is present these separate first during this treatment and may be collected separately. The basic zinc carbonate is calcined to oxide and the carbon dioxide used for the regeneration of ammonium carbonate. The filtrate from the zinc carbonate is evaporated to recover ammonium chloride for use again. The lead chloride residue is extracted with ammonium acetate or with ammonium thiosulphate and the lead eventually recovered by electrolysis of the fused chloride or sulphochloride. The process is applicable to the treatment of mixed sulphide ores; in these cases blende is much more resistant to chlorination than galena, so that by heating with a regulated quantity of ammonium chloride only the galena is chlorinated and may be extracted first. The residual blende is roasted and heated with ammonium chloride until the ammonia is expelled, and the zinc chloride is recovered by distillation at 750–900°.

A. R. POWELL.

**Thermal disintegration of chrome ores or minerals containing chromium.** BOZEL-MALÉTRA SOC. IND. DE PROD. CHIM. (B.P. 288,250, 27.2.28. Fr., 6.4.27).—The roasting of chromite ores with lime and sodium carbonate is carried out in mechanically rabbled, multiple-hearth furnaces of the type used in roasting pyrites.

A. R. POWELL.

**Production of rare metals.** J. W. MARDEN and M. N. RICH, ASSRS. to WESTINGHOUSE LAMP Co. (U.S.P. 1,728,941, 24.9.29. Appl., 19.2.27).—Tantalum, vanadium, and niobium are prepared in the form of a powder free from hydrogen and nitrogen by heating a mixture of the metal oxide, calcium, calcium chloride, and an alkali metal in a sealed or evacuated container.

C. A. KING.

**Production of uranium and uranium-zinc alloys.** J. W. MARDEN, ASSR. to WESTINGHOUSE LAMP Co. (U.S.P. 1,728,940 and 1,728,942, 24.9.29. Appl., [A] 1.3.26, [B] 29.8.28).—(A) Uranium oxide or a mixture of uranium oxide and zinc chloride is reduced by means of calcium in the presence of calcium chloride. (B) A compound of uranium is reduced and alloyed with zinc.

C. A. KING.

**Vanadium-aluminium-silicon alloy.** B. D. SAKLATWALLA, ASSR. to VANADIUM CORP. OF AMERICA (U.S.P. 1,727,180, 3.9.29. Appl., 2.2.28).—The alloy contains 40–90% V, 3–15% Al, 5–30% Si, and the remainder chiefly iron.

H. ROYAL-DAWSON.

**Manufacture of annealed thorium.** W. B. GERO,

ASSR. to WESTINGHOUSE LAMP Co. (U.S.P. 1,719,975, 9.7.29. Appl., 1.3.26).—Articles of highly reactive, rare refractory metals, *e.g.*, thorium, are annealed by heating them in a gas-tight container packed with a finely-divided, non-contaminating substance, *e.g.*, thorium, which will react with residual atmospheric gas to form stable compounds at the temperature of annealing, but is inert to the refractory metal. F. G. CLARKE.

**Ruthenium alloy.** M. M. GOLDSMITH, and W. H. FALCK, ASSR. to GOLDSMITH BROS. SMELTING & REFINING Co. (U.S.P. 1,730,003, 1.10.29. Appl., 3.1.27).—An alloy comprising about 75% Ru, 17.5% W, and 7.5% Ni is claimed as a substitute for osmiridium.

F. G. CROSSE.

**Working-up materials containing precious metals.** G. BÜRG (B.P. 317,752, 26.9.28. Ger., 21.8.28).—Ores, minerals, and residues containing precious metals in a colloidal form are subjected to sudden heating or cooling, to electrical conditions, or to pressure so as to set up sudden differences of stress which are claimed to make the precious-metal particles coalesce into visible form, which is recoverable by ordinary methods.

A. R. POWELL.

**Electrolytic production of aluminium.** ALUMINIUM IND. A.-G. (B.P. 305,458, 31.10.28. Ger., 4.2.28).—In the electrolytic refining of aluminium by the process described in B.P. 265,170 (B., 1928, 412), the anode comprises a plate of crude aluminium with deep corrugations so as to have a low current density at the anode, thus preventing iron and silicon from dissolving in the bath.

A. R. POWELL.

**Electrolytic production of aluminium.** VEREIN. ALUMINIUM-WERKE A.-G. (B.P. 318,431, 15.11.28. Ger., 3.9.28).—In the usual method of obtaining aluminium by electrolysis of alumina in a molten cryolite bath, the active surface of the electrodes is kept equal by using a single anode having a diameter slightly greater than that of the cathode which forms the base of the furnace, and one or more channels through which the charge can be introduced into the furnace. A. R. POWELL.

**Roasting of iron carbonate ores.** A. APOLD and H. FLEISSNER (U.S.P. 1,729,697, 1.10.29. Appl., 28.11.24).—See B.P. 253,305; B., 1926, 710.

**Metallurgical carbons** (B.P. 288,551 and 304,676).—See II.

## XI.—ELECTROTECHNICS.

**Some characteristics of photo-electric tubes.** L. R. KOLLER (J. Opt. Soc. Amer., 1929, 19, 135–145).—An account of recent work on photo-electric cells, and of some of their practical applications.

C. A. SILBERRAD.

**Pressure electrolysis of water.** R. SCHNURMANN (Z. angew. Chem., 1929, 42, 949–952).—Electrolysis of dilute sulphuric acid solution was carried out between platinum electrodes, the acid being enclosed in a sealed glass tube with a space above the liquid in which the gases, hydrogen and oxygen, were collected, thereby increasing the pressure (to about 870 atm.) until it was sufficient to rupture the vessel. The results show that the amount of current which could be passed increased with time, and therefore with gas pressure, until a

maximum was reached. A suggested use for the gas mixture prepared by this method is for the driving of an electric motor. H. T. S. BRITTON.

Testing permanent-magnet steel. OERTEL.  
Silver-plating solutions. SANIGAR. Electrodeposited chromium. PIERSOL.—See X. Electro-metric titration [of worts]. EMSLANDER.—See XVIII.

## PATENTS.

Electrolytic cells [for producing hydrogen and oxygen]. MONTECATINI SOC. GEN. PER L'IND. MIN. ED AGRIC., Assees. of G. FAUSER (B.P. 292,130, 13.6.28. Ital., 14.6.27).—Flat parallel electrodes are joined by current leads made of nickel-plated copper strip.

J. S. G. THOMAS.

[Uni-directional] electrolytic condenser. A. E. WHITE. FROM FANSTEEL PRODUCTS CO., INC. (B.P. 319,033, 15.5.28).—One or more filmed electrodes are immersed in a substantially dry electrolytic solution in an alcohol, *e.g.*, glycerin. A film of oil above the electrolyte serves to prevent absorption of moisture.

J. S. G. THOMAS.

Deoxygenation of enclosed atmospheres [of oil-immersed electric transformers etc.]. C. J. RODMAN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,725,160, 20.8.29. Appl., 23.12.22).—The atmosphere above the oil in such apparatus is deoxygenated by treatment with a deoxidising agent, the activity of which increases with rise of temperature.

A. R. POWELL.

[Construction of Faure-type] electric accumulator [positive] plates. H. G. BROWN, and D.P. BATTERY CO., LTD. (B.P. 319,388, 16.6.28).

[Unspillable vent for] electric batteries. M. WILDERMAN (B.P. 319,198, 17.5.28).

[Arrangement of parts in] photo-electric cells. E. G. O., A. E. M., and K. E. H. PRESSLER. ("OTTO PRESSLER" THÜRINGER VAKUUMRÖHRENFABR. U. FABR. WISS. APPARATE) (B.P. 302,183, 10.12.28. Ger., 9.12.27).

Aluminium (B.P. 305,458 and 318,431).—See X.

## XII.—FATS; OILS; WAXES.

The fat of barley and of its malting products. K. TÄUFEL and M. RUSCH (Z. Unters. Lebensm., 1929, 57, 422—431).—The fats extracted from barley and various products of its malting and brewing were analysed, using the Twitchell separation of solid and liquid fatty acids after removal of the unsaponifiable matter. The solid fraction was shown to be a mixture of stearic and palmitic acids; the proportions were calculated from the neutralisation value. The liquid fraction was shown to be a mixture of oleic, linoleic, and linolenic acids which was analysed by determining the linolenic acid as the hexabromostearic acid and then determining the oleic and linoleic acids indirectly from the iodine value of the liquid fraction (cf. B., 1929, 564). Barley fat and malt fat were thus found to contain, respectively, oleic acid 26.5, 16.4; linoleic acid 43.7, 49.4; linolenic acid 0.44, 0.82; palmitic acid 7.4, 8.2; stearic acid 2.6, 5.1; and unsaponifiable matter 5.4, 6.1%. The embryo fat contained 23—26% of unsaponifiable matter,

and the fat of the spent malt differed slightly from that of the malt. W. J. BOYD.

Fat from the seed husks of laurel berries. G. WALLRABE (Chem. Umschau, 1929, 36, 293—295).—The fat extracted from the (seed) husks (23.7%) of the laurel fruit was dark yellow and of buttery consistency, optically inactive, and contained no ethereal oils. It had acid value zero, saponif. value 198.2, iodine value 78.72, insoluble fatty acids 94.4%, unsaponifiable matter 1.35%,  $[\alpha]_D^{20} +0.82^\circ$ . The insoluble fatty acids had m.p. 39—41°, iodine value 85.19, mean mol. wt. 273.9; these yielded 24.15% of solid fatty acids (m.p. 60—61°, mean mol. wt. 257.9) and 74.82% of liquid fatty acids (iodine value 113.9, mean mol. wt. 284.3). The solid fatty acids consisted preponderantly of palmitic acid; the liquid fatty acids contained about 74% of oleic acid and 26% of linoleic acid. The phytosteryl acetate isolated from the unsaponifiable matter had m.p. 123—124° (from alcohol). Corresponding values for fat from the seed-free fruit are also given for comparison (cf. B., 1929, 859). E. LEWKOWITSCH.

Twitchell fat-splitting reagents. K. NISHIZAWA (Chem. Umschau, 1929, 36, 277—284).—The addition of electrolytes to solutions of fat-splitting reagents ("Idrapid," "Pfeiling," "Kontakt") has considerable influence on the emulsifying power as measured by the Donnan drop method (cf. A., 1900, ii, 102; *i.e.*, the drop number of an oil in reagent solution compared with that in water), the effect varying with the particular reagent employed. Increasing additions of a strong acid or salt, *e.g.*, hydrochloric or sulphuric acid, or sodium chloride or sulphate, up to a certain maximum (when salting-out of the reagent occurs) increases the emulsifying power (to an extraordinary extent in the case of the "Kontakt" reagent). A similar result is obtained with weak acids in much smaller degree, and no salting-out occurs. The emulsifying power of the sodium salts of the reagents increases with concentration, the effect being less if the reagent itself is admixed; the emulsifying power is greater with a mixed solution of 1% of reagent and 1% of its sodium salt than with a 2% solution of the reagent alone. The emulsifying power was found to be increased by the addition of glycerol; a marked increase was also obtained by the addition of fatty acids to the oil, the optimum concentration being at about 70% of fatty acid (1% solution of reagent).

E. LEWKOWITSCH.

Determination of sugar in soaps and soap preparations. K. BRAUN and E. WALTER (Chem.-Ztg., 1929, 53, 778).—A known weight of the sample is decomposed with excess dilute sulphuric acid, the vessel being kept at about 100° for  $\frac{1}{2}$  hr. in order to invert the sucrose; after 24 hrs. the acid liquor is filtered, neutralised with soda, and made up to standard volume (200 c.c.). Then 25 c.c. of the sugar solution are added to 50 c.c. (excess) of boiling Fehling's solution and 25 c.c. of water; the whole is boiled for 5 min. and allowed to settle (20 min.). The cuprous oxide is collected, washed with boiled-out distilled water, and dissolved in 50 c.c. of standard ferric sulphate solution (50 g. of ferric sulphate dissolved in 200 c.c. of concentrated sulphuric acid and diluted to 1 litre), and the

ferrous sulphate produced is titrated with 0.1*N*-permanganate (1 c.c. = 0.0029 g. of sucrose). The method is suitable for the determination of sugar in glycerol, and of starches in soaps etc.

E. LEWKOWITSCH.

**Polymerisation of linseed oil in stand-oil formation.** F. WILBORN and F. KITTLER (*Farben-Ztg.*, 1929, 34, 2942—2943).—Determinations of mol. wts. of stand oils in order to ascertain the degree and type of polymerisation occurring have not led to concordant results. The mol. wts. of a sample of raw linseed oil, before and after heat-thickening, and of the fatty acids derived therefrom were determined by a variety of methods. The influences of solvent used, concentration of solution, etc. on the results obtained lead to the conclusion that it is impossible to investigate the polymerisation problem by mol. wt. determination alone.

S. S. WOOLF.

**Drying of tung oil.** H. WOLFF (*Farben-Ztg.*, 1929, 34, 2941—2942).—A dried tung oil film was stored for a period of 2½ yrs., light and air being excluded. After about 1¼ yrs. syneresis occurred, droplets of oil of lower refractive index than the original oil being formed. After 2¼ yrs. three phases were present, a crystalline substance—probably  $\beta$ -elæostearin—having also separated from the film.

S. S. WOOLF.

**Determination of organically-combined sulphur in sulphonated oils.** R. HART (*Chem. Umschau*, 1929, 36, 295—297).—The author's revised volumetric method is described (cf. *J. Amer. Leather Chem. Assoc.*, 1927, 22, 588); improvements in the original procedure (cf. *B.*, 1917, 1139) consist in the use of *N*-sulphuric acid, and the addition of ether and salt to facilitate observation of the end-point.

E. LEWKOWITSCH.

**Determination of fats in drugs.** GLASER and HALBERSTAM.—See XX.

#### PATENTS.

**Extraction of fatty acids, resins, bitter substances, and mucilage from oils and fats.** K. K. WILHELM (U.S.P. 1,729,809, 1.10.29. Appl., 15.9.27. Ger., 2.5.27).—A volatile fat solvent is added to the oil or fat and the mixture is stirred (preferably warm) with an aqueous alcohol-ammonia solution; about 1% of Glauber's salt dissolved in 10 pts. of the ammonia solution is added, and the whole is re-mixed. On being kept the oil-solvent layer separates free from mucilage and is drawn off and evaporated. E. LEWKOWITSCH.

**Manufacture of sulphonated derivatives of unsaturated fatty acids.** I. G. FARBENIND. A.-G. (B.P. 296,999, 10.9.28. Ger., 10.9.27).—Unsaturated fatty acids or esters (*e.g.*, oleic acid, castor oil) are treated with fuming sulphuric acid in the presence of a halogenated unsaturated hydrocarbon (*e.g.*, trichloroethylene); the sulphonated products are neutralised, washed, etc., and the solvent is removed by distillation. The products are resistant to acids and not precipitated by calcareous water. E. LEWKOWITSCH.

**Manufacture of fatty acid peroxide.** W. B. STODARD and V. R. KOKATNUR, ASSRS. to PILOT LAB., INC. (U.S.P. 1,718,609, 25.6.29. Appl., 21.10.27).—A fatty acid chloride is treated with sodium peroxide.

C. HOLLINS.

**Extraction of oils from oleaginous materials.** R. O. BOYKIN, ASSR. to N. R. VAIL (U.S.P. 1,721,686, 23.7.29. Appl., 22.11.26).—Oily meal is dried and extracted with a water-insoluble oil solvent the sp. gr. of which is not greater than that of the meal; the mixture is then pressed and the residual material again washed with a fresh quantity of the solvent. Suitable apparatus is described. R. H. GRIFFITH.

**Process, steps, and product of reaction of cashew nut-shell oil. Modified cashew [nut-]shell liquid [varnish]. Cashew nut-shell oil condensation product.** M. T. HARVEY, ASSR. to HARVEL CORP. (U.S.P. 1,725,795—7, 27.8.29. Appl., [A] 23.12.26, [B] 17.11.27, and [C] 15.6.28).—(A) Cashew nut-shell oil (25 vols.) is dissolved in 50 vols. of light petroleum, 10 vols. of formalin and 5 vols. of concentrated hydrochloric acid are added, and the mixture is kept for 48 hrs. The oily layer is separated and used as a varnish. (B) The oil is heated rapidly to 340°, preferably under pressure, cooled to 230°, and mixed with 1% of litharge and 1% of manganese resinate. The product is suitable for varnishing fabrics and paper, and dries to a flexible film at 135°. (C) The oil is heated rapidly to 340°, mixed with 25% of its weight of raw linseed oil, cooled to 260°, mixed with driers as under (B), cooled to 150°, mixed with an equal weight of a solvent, cooled, treated with 1% of its weight of formalin, set aside overnight, and heated at 140° to expel water and complete the reaction.

A. R. POWELL.

**Soap preparation.** F. GÜNTHER and J. NÜSSLEIN, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,730,037, 1.10.29. Appl., 29.5.26. Ger., 2.6.25).—See B.P. 253,105; *B.*, 1928, 60.

**[Detergent for] washing of textile goods.** E. RIEHL and O. LIND, ASSRS. to HENKEL & Co. G.M.B.H. (U.S.P. 1,716,347, 4.6.29. Appl., 15.8.27. Ger., 17.8.26).—See B.P. 276,339; *B.*, 1928, 647.

**Packing material for soft soap** (B.P. 319,517).—See V. **Liquors for treating leather** (B.P. 318,070).—See XV. **Soaps and oils from sewage** (B.P. 318,849).—See XXIII.

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

**Viscosity of oil paints.** H. WOLFF (*Farben-Ztg.*, 1929, 34, 2990—2991).—Paints of identical oil content but prepared either by grinding to paste and thinning out or by grinding with the total amount of oil show differences in viscosity, the latter type being the more viscous. The difference is accentuated at low oil contents, but diminishes with increase of oil. It is shown, however, that the "second critical point" previously described (*B.*, 1929, 825) is virtually independent of the method of grinding, and may thus, for a particular oil, be regarded as a constant for the pigment.

S. S. WOOLF.

**Some optical properties of paints and pigments.** F. C. TOY (*J. Oil & Col. Chem. Assoc.*, 1929, 12, 225—239).—The term "covering power" is discussed from the aspect of obliteration rather than of spreading. The theory of measuring instruments of the "cryptometer" type is examined, and it is shown that the physiological characteristics of the eye render such instruments

unreliable, since the matching point (and hence the measured "covering power") varies with the observer and with the intensity and colour of the light used. It is suggested that the grading of paints in order of covering power could be carried out mechanically by making use of a photoelectric cell. A series of curves correlating grain size with obliterating power for light of different wave-lengths is considered. The existence of a maximum in these curves introduces the difficulty of ascertaining on which side of the maximum a determined obliteration lies, whilst the accuracy of measurement depends largely on the actual size. A method for determining grain size is discussed, in which the optical density of a dispersion is observed, undeviated light alone being measured in spite of the fact that a fraction of the scattered light comes forward in the same beam. The extinction coefficient ( $\beta$ ) for undeviated light is given by the expression  $I = I_0 e^{-\beta M}$ ,  $I_0$  and  $I$  being the intensities of incident and transmitted beams, respectively, and  $M$  the mass traversed. The quantity  $\beta\rho$ , where  $\rho$  is the density of the solid phase, is suggested as a criterion of grain size, applicable to all kinds of substances. If the reciprocal of  $\beta\rho$  be plotted, a straight-line relation between it and grain size holds over nearly all the range of sizes investigated.

S. S. WOOLF.

**Properties of current Finnish turpentine oil.**

E. PYHÄLÄ (Chem.-Ztg., 1929, 53, 758—759).—The somewhat primitive methods of commercial production of Finnish turpentine oil (from pine stumps) are described: since no refining of the crude oil or after-treatment with deodorisers or decolorisers etc. is undertaken, a pine oil of variable character and odour is obtained. The characteristics (distillation range, flash point, etc.) of typical oils (20—70% of turpentine) are given. It is shown that by suitable treatment prior to distillation a higher yield of a purer turpentine can be obtained.

E. LEWKOWITSCH.

**Oil absorption of pigments.** W. VAN WÜLLEN-SCHOLTEN (Farben-Ztg., 1929, 34, 2940—2941).—The factors on which depends the determination of oil absorption of pigments are indicated. Oil absorption values obtained by the Gardner method (modified by the use of a short glass rod in place of a flat spatula) are quoted for a range of common pigments. Oil absorptions of mixtures are, in general, additive quantities.

S. S. WOOLF.

**White pigments.** III. H. WOLFF and G. ZEIDLER (Farben-Ztg., 1929, 35, 24—25; cf. B., 1928, 274).—No appreciable difference in the extent of lead soap formation in paints made from basic carbonated and basic sulphate white lead was detected by optical or analytical methods. In exposure tests paints made from the carbonate show a greater loss of weight than those made from the sulphate. It is considered that the influence of lead soap formation on the durability of paint films is still problematical.

S. S. WOOLF.

**Determination of water-soluble arsenious acid in Schweinfürth green.** H. M. L. BRÉMOND (Bull. Soc. Pharm. Bordeaux, 1928, 66, 226—231; Chem. Zentr., 1929, i, 2085).—The finely-divided material (1 g.) is boiled for exactly 5 min. over a small flame with

25 c.c. of 12.5% sodium acetate solution, boiling water being added as the liquid evaporates. The cooled liquid is diluted to 50 c.c., filtered, and the arsenious oxide is titrated with 0.1*N*-iodine solution. The process is repeated with 2 g. and 50 c.c., respectively, of material; the difference in the volumes of iodine solution required multiplied by 0.00495 represents the quantity of arsenious oxide in 1 g. of the Schweinfürth green. The result is accurate when the quantity of water-soluble arsenious oxide does not exceed 3%. A. A. ELDRIDGE.

## PATENTS.

**Manufacture of emulsion paints.** L. KIRSCHBRAUN (U.S.P. 1,691,765, 13.11.28. Appl., 7.5.25).—Paint base, e.g., pitch, bitumen, which may contain finely-divided mineral powder, is dispersed in an aqueous medium by means of a finely-divided pigment paste.

R. BRIGHTMAN.

**Production of [bituminous] emulsions or dispersions.** L. KIRSCHBRAUN and H. L. LEVIN, Assrs. to FLINTKOTE Co. (U.S.P. 1,691,766—7, 13.11.28. Appl., [A] 17.12.25, [B] 12.10.26).—(A) Maximum dispersion of asphalt or other pitches in aqueous media is obtained by adjusting the  $p_H$  value of the system to a predetermined value depending on the nature of the pitch. Alternatively, the pitch may be treated to render it suitable for dispersion by a particular emulsifying agent. The emulsion may be subsequently treated to modify its  $p_H$  value and the properties of the final system without breaking the emulsion, particularly to increase the resistance of the dried product to water action and re-emulsification. (B) The dispersion is effected in an aqueous solution of bentonite or other insoluble mineral dispersing agent.

R. BRIGHTMAN.

**Production of [bituminous] emulsions or dispersions.** L. KIRSCHBRAUN (U.S.P. 1,691,768, 13.11.28. Appl., 16.3.28).—The dispersion of bitumen or pitch is effected in an aqueous suspension of a mixture of two or more mineral dispersing agents, previously adjusted to a definite  $p_H$  value by addition of an electrolyte as buffer salt.

R. BRIGHTMAN.

**Plasticised moulding composition.** H. M. WEBER, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,720,406, 9.7.29. Appl., 17.12.24).—Hexamethylenetetramine-triphenol is used as a flux to give greater fusibility to a mixture of a resin with a filler.

R. H. GRIFFITH.

**Manufacture of colours or the like capable of being fixed by heat.** O. VAN CUYCK (U.S.P. 1,730,178, 1.10.29. Appl., 7.5.26. Belg., 8.5.25).—See B.P. 251,866; B., 1926, 638.

**Mills for grinding paints, enamels, inks, and other viscous substances.** E. A. WHITE (B.P. 319,544, 4.12.28).

**Decohering solid substances** (B.P. 292,965).—See I. Aryl alkyl and hexyl esters (U.S.P. 1,700,960 and 1,702,180).—See III. Iron oxide pigments (U.S.P. 1,726,851—2).—See VII. Compositions for paints (B.P. 317,502).—See IX. Resins from oils and fats (U.S.P. 1,729,809). Cashew nut-shell oil products (U.S.P. 1,725,795—7).—See XII.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Increased stretching capacity of dipped rubber articles (toy balloons etc.) and decrease in number of dippings required for thick-walled dipped rubber articles resulting from the increased viscosity of rubber solutions containing sipalin. R. DITMAR and K. H. PREUSZE (Chem.-Ztg., 1929, 53, 779).—The addition of 5% of sipalin (adipic esters) to rubber solutions greatly increased the stretching capacity of articles made from them by dipping (as determined by the size of balloons at the bursting point). Sipalin MOM was the most effective when vulcanisation was brought about by sulphur chloride; with sulphur vulcanisation the action of the sipalin appeared to depend on the accelerator used, and the choice of the most suitable ester is determined by the conditions. Since the addition of sipalin greatly increases the viscosity of the solutions fewer dippings are needed to produce a given thickness of rubber. E. LEWKOWITSCH.

New microscopical methods in connexion with problems of vulcanisation. E. A. HAUSER, H. MEDEL, and M. HÜNEMÖRDER (Coll. Symp. Mon., 1928, 6, 207—217).—When the material is undercured sulphur reappears as colloidal globules which grow and form dendrites; zinc oxide retards this process. Accelerators appear to prevent separation of the sulphur except at the surface. Ether-soluble  $\alpha$ -caoutchouc vulcanises less readily than whole rubber or  $\beta$ -caoutchouc. It is considered that vulcanisation depends on minute traces of a substance present in crude rubber which adheres to the  $\alpha$ -fraction so that it can be removed only after this fraction has been isolated from its original combination in the rubber. CHEMICAL ABSTRACTS.

## PATENTS.

Preservation of rubber latex. A. J. SOMER and R. B. R. WALKER (B.P. 318,717, 30.7.28).—Rubber latex is stabilised by the addition of a boron compound, e.g., sodium pentaborate or a mixture of borax and boric acid, so as to impart a  $\eta_H$  value between 6 and 9.2; a small proportion of a germicide may also be introduced. D. F. TWISS.

Preparation of viscous dispersions of rubber and similar materials. ANODE RUBBER CO. (ENGLAND), LTD., Assees. of COMP. GÉN. D'ELECTRICITÉ SOC. ANON. (B.P. 291,805, 9.6.28. Ger., 9.6.27).—Highly viscous aqueous dispersions of rubber, gutta-percha, or balata, which may contain compounding ingredients, are produced by the addition of polysaccharides, proteins, or plant extracts which even at high concentrations form viscous solutions but not jellies. E.g., gum arabic, or partially hydrolysed starch, gelatin, or agar-agar may be used, and such hydrolysis may be effected after introduction of the materials into the latex. D. F. TWISS.

Chemical products [vulcanisation accelerators and anti-oxidants for rubber]. S. M. CALDWELL, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,724,180, 13.8.29. Appl., 2.3.28).—Alkali salts of mercaptobenzthiazoles are heated with chlorodinitrobenzenes, e.g., in alcohol solution. 2:4-Dinitrophenylbenzthiazole sulphide (yellow, m.p. 162—5°) and 4-chloro-2:6-

dinitrophenylbenzthiazole sulphide (yellow, m.p. 167°) are described. R. BRIGHTMAN.

Vulcanisation of caoutchouc. GOODYEAR TIRE & RUBBER CO., Assees. of L. B. SEBRELL (B.P. 297,051, 19.6.28. U.S., 13.9.27).—Vulcanisation is accelerated by the reaction product of a mercaptobenzthiazole, e.g., mercaptobenzthiazole or mercaptophenylthiazole, and a Schiff base, e.g., ethylidene- or crotonylidene-aniline. The reaction may be effected by heating the mixture under reflux for about 2 hrs. D. F. TWISS.

Manufacture of rubber. E. A. HAUSER, Assr. to K.D.P., LTD. (U.S.P. 1,729,651, 1.10.29. Appl., 16.12.25. U.K., 19.12.24).—See B.P. 250,639; B., 1926, 598.

## XV.—LEATHER; GLUE.

Antiseptic value of chlorine with reference to animal skin disinfection. R. C. HOWARD, G. E. ROCKWELL, and W. L. CRIST (J. Amer. Leather Chem. Assoc., 1929, 24, 456—479).—Experiments with Hay bacillus and its spores have shown that the time required for sterilisation by chlorine is increased by increasing the number of bacteria present. Treatment lasting 1 hr. was necessary to sterilise 24 millions of bacteria per c.c. and 3 hrs. for 120 millions, using 120 p.p.m. of chlorine. A longer time was required to sterilise the older spores. The germicidal actions of hypochlorites and chlorine were diminished by the presence of sodium chloride, ammonia, organic matter, and ammonia and ammonium chloride, respectively. The effect of 0.05N-hydrochloric acid was to lower the resistance of the spores to chlorine treatment, but the presence of acid in the chlorine solution lowered its germicidal action. It was shown that such a large amount of chlorine is fixed by raw skins immersed in water containing chlorine, that the concentration of chlorine necessary to effect their complete sterilisation bleaches the hair and retards its loosening. The addition of ammonia to the chlorine water diminished its fixation by the skins. The addition of lactic or formic acid to the chlorinated soak water was not advantageous. Spores were not killed by treatment with lime liquors. The amount of chlorine required to disinfect tannery effluent will depend on the amount of nitrogenous matter and the number of bacteria per c.c. present in the liquor. D. WOODROFFE.

Tanning materials and their determination. G. GRASSER and S. TAU (J. Amer. Leather Chem. Assoc., 1929, 24, 406—427).—Pieces of hide were treated with various tanning agents and the time of hydrolysis, shrinkage temperature, and resistance to boiling were determined on each piece. Iodine was better than bromine or sulphur as judged by the hydrolysis and boiling tests. Bromine, unlike iodine, formed an insoluble compound with gelatin. The salts of chromium, iron, aluminium, thallium, uranium, platinum, and zinc increased the shrinkage temperature of hide to above 65° and, with the exception of zinc, tanned the hide well. All metallic salts except those of barium increased the m.p. of gelatin. Chromium dichromate gave the best tanned leather as judged by the hydrolysis test. The effects of other metallic

dichromates on hide and gelatin respectively showed variations with these materials. No increase in the m.p. of gelatin was observed by the action of dyes, but an increase was noted when they were allowed to act on tanned gelatin; this is attributed to the tanning action of the dyes themselves. Evidently pretreatment of the gelatin with tannin is necessary to obtain the tanning effects of the dyes. The tests with hide were not so favourable. The shrinkage temperature and the hydrolysis and boiling tests showed a difference as the result of the action of gallo-tannic acid on hide, but the m.p. of gelatin was not much affected by it unless sodium sulphate, sodium acetate, or potassium carbonate was added to the solution. The shrinkage temperature of hide was raised by treating it with vegetable tanning materials; the m.p. of gelatin was, however, unaffected. The m.p. of gelatin was raised by treatment with sulphite-cellulose extract, but only when hide had been pretreated with some other tanning materials was its shrinkage temperature increased by subsequent treatment with sulphite-cellulose extract. In an appendix, the effect of ultra-violet light on tanning intensity was studied, and the results are tabulated.

D. WOODROFFE.

**Tannin content of dead chestnut trees.** R. M. NELSON and G. F. GRAVATT (J. Amer. Leather Chem. Assoc., 1929, 24, 479—499).—It is shown that the tannin content of chestnut trees killed by the blight is not seriously diminished even in trees which have been dead for 30 years. The loss of tannin occurring through the action of decay-producing fungi is not great in the decayed wood, but the amount of available material diminishes. There is no connexion between the sp. gr. and tannin content of green, sound, dead, or decayed chestnut wood. The colour of slightly decayed wood was not worse than that of sound wood. The tannin content of very badly decayed sapwood was low and the colour of the liquors was very dark.

D. WOODROFFE.

**Synthetic tanning materials. I. Hydroxybenzene series.** G. GRASSER (J. Fac. Agric. Hokkaido, 1929, 24, 73—92).—Various aldehydes were condensed with phenol, sodium phenoxide, or phenolsulphonic acid, and the precipitating power of soluble products towards gelatin was tested. CHEMICAL ABSTRACTS.

**South Japanese tanning materials.** G. GRASSER (J. Fac. Agric. Hokkaido, 1929, 24, 61—72).—The following values (% of tannin) for Formosan species are recorded: *Quercus castanopsisifolia* (acorn cups) 28·3, *Q. amygdalifolia* (acorns) 6·1, *Q. glandulifera* (galls) 20·4, *Areca catechu*, L., 12·3, *Castanopsis taiwaniana*, Hayata, (wood) 2·0, (bark) 9·2, *Castanopsis kawakamii*, Hayata, (wood) 3·3, (bark) 9·3, *Acacia confusa*, Merr., (bark) 12·0, *Discorea rhipogonoides*, Oliv., (root nodes) 3·0. *Disopyros kaki* tannin is unstable.

CHEMICAL ABSTRACTS.

**Solidification of gelatin.** M. BERGMANN and B. JACOBI (Kolloid-Z., 1929, 49, 46).—Some properties of stretched gelatin films have been compared with those of unstretched films. The tensile strength of the unstretched gelatin was 4·4 kg./mm.<sup>2</sup>, and it was observed

that immediately before rupture the formerly clear film became milky and opaque. The stretched gelatin had a tensile strength of 9·3 kg./mm.<sup>2</sup> in the direction of stretching. The middle portion of the strip had the greatest tensile strength, values between 10 and 12 kg./mm.<sup>2</sup> being obtained. In this case no turbidity appeared before rupture. In the direction perpendicular to that of stretching the tensile strength was only 3·3—4·2 kg./mm.<sup>2</sup> The fracture was smooth.

E. S. HEDGES.

**Purification of tannery effluents.** BESSE.—See XXIII.

PATENTS.

**Liquors for use in tanning or treating leather, and their application.** O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 318,070, 27.12.28).—An emulsion produced by agitating one or more oils or fats, e.g., fish oils, rape oil, wool fat, mineral oil, with an aqueous alkylcellulose solution, particularly the methyl or ethyl derivative, with or without the addition of a dye, salt, or other substance used in dyeing, is employed.

D. WOODROFFE.

**Treatment of skins or hides with tanning or other liquids.** E. LUCKHAUS (B.P. 290,681, 19.5.28. Ger., 19.5.27).—Hides or skins are hung up and stretched in a closed tanning vessel full of tan liquor, which is submitted alternately to a vacuum and air pressure. The vessel is provided at the lower end with an agitating device, and the suspension device fits into a journal in the upper part whereby it can be revolved in the vessel. The tan liquor is circulated by a pump outside the vessel and a filter is provided for it.

D. WOODROFFE.

**Adhesive and plastic mass and its manufacture.** M. LANDECKER, Assn. to AMER. NUPLAX CORP. (U.S.P. 1,725,805, 27.8.29. Appl., 29.5.26. Ger., 8.4.26).—A mixture of 20 pts. of powdered casein and 100 pts. of carbamide is made into a thick paste with water, and formaldehyde is added as a preservative, and to increase the adhesive power.

A. R. POWELL.

**Dyeing of leather** (B.P. 316,822).—See VI.

## XVI.—AGRICULTURE.

**Soils. IV. Classification of their acidic functions. V. Replaceable bases.** J. CLARENS and (MME.) PÉRON (Bull. Soc. chim., 1929, [iv], 45, 666—671, 671—674; cf. B., 1928, 869).—IV. In the determination of the acidity of a calcareous clay soil by means of aqueous baryta solution, those acidic constituents which form barium salts hydrolysable by water remain undetermined. They can be determined, however, by the use of alcoholic potash solution. A method for representing geometrically the different types of acidity of a soil is described. V. The different types of acidity present in a soil can also be distinguished by shaking the soil with varying known amounts of 0·1N-hydrochloric acid and determining the amount of free hydrochloric acid remaining in the solution.

O. J. WALKER.

**Reclamation of Fresno type of black alkali soil.** W. P. KELLY and E. E. THOMAS (Calif. Agric. Exp. Sta. Bull., 1928, No. 455, 37 pp.).—The unproductivity of this soil is due to an excess of soluble salts, particularly



sodium hydrogen carbonate, and the abnormal composition of the clay-like constituents; the productivity can be improved by the use of gypsum, sulphur, ferrous sulphate, or alum, which act simultaneously on the soluble carbonate and clay. Gypsum is effective by reason of its soluble calcium, whilst the other materials act by reason of their acidic nature; the effect of sulphur depends on its slow oxidation. CHEMICAL ABSTRACTS.

**Hardpans, concretionary layers, and neo-formations of the soils of the more arid parts of the Union [of South Africa].** J. V. CUTLER (Proc. Dept. Conf. S. Africa, Div. Chem., 1929, 421—425).—Extensive areas of hardpan occur in the Union of South Africa. These are of different types, including lime formations, iron formations which appear to be complex silicates and carbonates of calcium, magnesium, and iron coloured different shades of red to brown, and "double" hardpans, which consist of solid hardpan giving place to softer coherent material, readily broken by the spade, deeper in the soil horizon. Two tentative explanations of these formations are presented: (1) that complex soluble salts are washed down to, and deposited at, the pan layer in districts of low rainfall, and (2) that the weathering of parent rock, rich in material which is easily decomposed, but not leached out, gives products which are consolidated *in situ*. In alkali areas the point of maximum concentration of soluble salts does not usually coincide with the layer of maximum development of hardpan. Questions of root penetration in, and methods of breaking up, hardpan are discussed.

E. HOLMES.

**Practical sterilisation by heat of small quantities of soil.** W. F. BEWLEY (J. Min. Agric., 1929, 36, 623—634).—Various methods for steaming and baking soil are described. A uniform temperature of at least 100° throughout the soil mass is essential, and the treated soil should be in a suitable physical and chemical condition for plant growth. Steaming is the better process.

A. G. POLLARD.

**Determination of ammonia in soil and adsorptive power of soil for ammonia.** C. OLSEN (Compt. rend. Trav. Lab. Carlsberg, 1929, 17, No. 15, 1—20).—The sample is extracted by shaking with *N*-potassium chloride solution after adjustment of the  $p_H$  value of the suspension to 1.0 by the addition of hydrochloric acid. After filtering through a Berzelius filter, the ammonia in the extract is determined by distillation with magnesia in the usual manner. Peat soils have greater adsorptive capacities for ammonia than clays and loams.

A. G. POLLARD.

**Determination of the fertiliser requirement of soils.** J. KÖNIG (Naturwiss., 1929, 17, 755—758).—In determining fertiliser requirements the nature of the crop must be considered, together with its capacity to utilise nutrients present in soils and fertilisers. Easily soluble nutrients in soil are determined by extraction with 1% citric acid solution. The actual quantity of nutrient assimilable by the crop is calculated from these values by means of a predetermined "percentage utilisation coefficient" for the particular crop. The difference between the values so obtained and

the maximum plant requirement as determined by crop analysis represents the amount to be supplied to the soil artificially. The actual quantity of fertiliser needed is calculated by means of the percentage utilisation coefficient for the particular crop with reference to the nature of the fertiliser to be used.

A. G. POLLARD.

**Determination of soil nutrient deficiencies.** E. BECKER (Mezőgazdasági Kutatások, Budapest, 1928, 1, 57—94; Bied. Zentr., 1929, 58, 387—390).—Critical trials of the chemical methods of Lemmermann, König and Hasenbäumer, and 'Sigmond, the biological *Azotobacter* method of Niklas and Hirschberger, the chemico-physiological method of Neubauer and Schneider, and corresponding vegetative and field experiments for determining soil nutrient deficiencies are briefly summarised. Practical details are lacking. According to 'Sigmond's method potash may be determined in the filtrate from the phosphoric acid determination; iron, aluminium, calcium, and magnesium are removed by addition of ammonia and ammonium carbonate, sulphate by means of barium chloride, and the potash is then determined by the perchlorate method.

E. HOLMES.

**Influence of soil reaction on the conversion of different forms of nitrogen in the soil and their utilisation by plants.** K. NEHRING (Landw. Jahrb., 1929, 69, 105—148; Bied. Zentr., 1929, 58, 390—392).—Potash salts are physiologically neutral, superphosphate is certainly not acid, and slag is definitely basic. This physiological nature of fertilisers is of importance from the point of view of nitrification in the soil. Urea and cyanamide act in the first place as physiologically alkaline, but later as acid fertilisers, the net result being that urea is physiologically acid and cyanamide alkaline. Leunaphos and to a smaller degree nitrophoska are acid fertilisers. It is shown that in 1927 in humus, sandy loam pot cultures with artificially adjusted media sodium nitrate on the average gave its optimum effect at  $p_H$  4—5, urea and calcium cyanamide at  $p_H$  6, urea at  $p_H$  7, and ammonium sulphate at  $p_H$  8. The results varied with the crop; barley is acid-sensitive, and oats are acid-tolerant. Below  $p_H$  6.5 nitrate-nitrogen is a better plant food than ammoniacal nitrogen; above that figure they are of equal value. E. HOLMES.

**Peat as a source of nitrogen.** LOGVINOVA.—See II.  
**Micro-determination of phosphate ions.** DENIGÈS.—See VII.  
**Soil corrosion studies.** LOGAN.—See X.

#### PATENTS.

**Insecticides.** O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 317,525, 18.5.28).—The use is claimed of solutions etc. containing mono- or polythiocyanates other than aliphatic monothiocyanates, together with other constituents. L. A. COLES.

**Insect repellent.** W. MOORE and H. E. BUC, ASSTS. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,727,305, 3.9.29. Appl., 20.4.28).—The product comprises a volatile solvent and an alkyl phthalate.

L. A. COLES.

## XVII.—SUGARS; STARCHES; GUMS.

## Special case of corrosion in cane-sugar factory.

T. VAN DER LINDEN (Rec. trav. chim., 1929, 48, 965—968).—Corrosion of brass evaporator tubes, probably initiated by hydrogen sulphide present in the juice entering the evaporator, has been observed. The hydrogen sulphide is presumably formed by microbiological action.

H. BURTON.

Velocity of crystallisation of sucrose from its pure solutions. B. G. SAVINOV (Nauch. Zapiski, 1929, 7, 416—429).—The velocity increased with the speed of revolution of the agitator up to 400 r.p.m., thereafter becoming constant. CHEMICAL ABSTRACTS.

Organic acids of sugar-cane molasses. E. K. NELSON (J. Amer. Chem. Soc., 1929, 51, 2808—2810).—The volatile acids present are formic (about 0.1%) and acetic (0.2%), whilst the non-volatile acids consist of aconitic (0.8%), lactic (0.05%), and small amounts of malic and citric acids.

H. BURTON.

Determination of sugar in dry [beet] slices. N. FRIZ (Zentr. Zuckerind., 1929, 37, 42; Chem. Zentr., 1929, i, 2482).—The material is soaked for 30 min. in cold water, treated with lead acetate and calcium carbonate, diluted, heated for 0.75—1 hr. at 100°, cooled to 20°, made slightly alkaline to phenolphthalein with sodium carbonate, and the invert sugar determined before and after inversion.

A. A. ELDRIDGE.

Improvement of beet-sugar products treated with activated carbon. I. N. KAGANOV and B. E. KRASILSCHIKOV (Nauch. Zapiski, 1928, 6, 344—351).—Not only was the colour decreased, but also the speed of crystallisation and filtration was increased.

CHEMICAL ABSTRACTS.

Boiling of [sugar] thin juice, and determination of natural alkalinity. H. DÜWELL and K. SOLON (Deut. Zuckerind., 1929, 54, 237—240; Chem. Zentr., 1929, i, 2595).—For the determination of residual alkalinity carbon dioxide, purified with potassium permanganate, is passed into 205 c.c. of hot juice containing phenolphthalein until the red colour just disappears. After addition of water (50 c.c.) the liquid is boiled for 6 min., filtered rapidly, and the alkalinity of the cooled filtrate and washings is determined by titration with 0.2*N*-hydrochloric acid. The calcium oxide is determined in 50 c.c. of neutralised juice with soap solution.

A. A. ELDRIDGE.

Sugar titration. N. SCHOORL (Z. Unters. Lebensm., 1929, 57, 566—576).—Luff's solution (cupric sodium citrate, 0.1*N* in cupric salt), the preparation of which is described (cf. B., 1899, 78), is of such concentration that 1 c.c. of the reagent heated with 25 c.c. of 0.1*N*-hydrochloric acid for 1 hr. on the water-bath should neutralise 19 c.c. of the latter. For the sugar titration the sugar solution is added to 25 c.c. of the reagent and the volume is made up to 50 c.c. The liquid is heated to boiling over a naked flame, quickly attached to a reflux, boiled for exactly 10 min., cooled, and after 5 min. titrated by one of the following methods. (i) The excess of cupric salt may be determined by adding potassium iodide followed either by 25% sulphuric acid, or by a mixture of 25% hydrochloric acid and 20% of thiocyanate solution,

and titrating the iodine in the usual way against 0.1*N*-thiosulphate. (ii) The cuprous compound may be titrated by adding to the cold solution 50 c.c. of 0.4*N*-acetic acid, followed, after shaking, by 25 c.c. of 0.1*N*-iodine; after further shaking, 55 c.c. of 0.75*N*-hydrochloric acid are poured down the side of the flask on to the surface of the liquid, which is gently agitated till all the cuprous compound is dissolved, and the excess of iodine is then titrated with 0.1*N*-thiosulphate. A table is given showing the quantity of various sugars (dextrose, laevulose, lactose, and maltose) corresponding with a given amount of reduced copper measured in c.c. of 0.1*N*-thiosulphate under the above conditions. The influence of time of boiling and of the relative proportion of reagent and sugar has been studied for various sugars. Advantages of Luff's over Fehling's solution are indicated, e.g., the former is more stable on storage and on boiling.

W. J. BOYD.

Sources of waste in the manufacture of starch. SPROCKHOFF (Z. Spiritusind., 1929, 52, 306).—The amount of starch left in the water draining from the tanks in which the raw starch settles may be on an average as much as 0.6—0.75% of the total yield, but should never exceed 0.75%. This figure corresponds to 0.08 g. of dry starch per litre of wash-water. The loss of starch during the washing process for the purification of the sludge of raw starch is usually slightly higher, but should not exceed 1% of the final yield. At this stage considerable loss may occur by fermentative action causing a decrease in the size of many of the starch grains so that the filter fabric is unable to retain them. The loss of starch in the form of starch dust during the drying process is unimportant, and in most cases is less than 0.1% of the starch output.

C. RANKEN.

Amylases in dry potato starch. SPROCKHOFF (Z. Spiritusind., 1929, 52, 312—313).—In many cases the decrease in the viscosity of starch paste which cannot be attributed to the action of acid produced by bacteria is due to the presence of amylases in the original starch. That the amylases are present can be best proved from the greater viscosity of starch paste made by direct heating with water to 100°, compared with that of the paste made by first heating to 68°, cooling to 45° and retaining at that temperature for 3 hrs., and subsequently heating to 100°. If sodium chloride is present, the liquefying action of the amylases is increased.

C. RANKEN.

Influence of temperature of refinery massecuite on hardness and destruction of sugar. M. I. NAKHMANOVICH and I. F. ZELIKMAN (Nauch. Zapiski, 1928, 6, 394—412).

*Erratum*.—Page 449, col. 1, line 22 from bottom, for "nitrite" read "nitrile."

Determination of sugar in soaps etc. BRAUN and WALTER.—See XII. Determination of lactose. SCHULZE. Melezitose in honey. NOTTBOHM and LUCIUS.—See XIX.

## XVIII.—FERMENTATION INDUSTRIES.

Course of fermentation in the fermenting vessel. [Condition of yeast during primary fermentation.] F. WINDISCH (Woch. Brau., 1929, 46, 379—388).—Two

yeasts, "S" and "V," were pitched in a number of 10-litre portions of wort, and at daily intervals the proportion of suspended and sedimentary yeasts and their reproductive and fermentative powers were determined. By the end of the first day the suspended yeast was only in slight excess, and so continued until the fourth day. From the fifth day the proportion of suspended yeast gradually diminished, showing no sign of stronger sedimentation (break) at any particular reaction. Both at the beginning and end of the fermentation period of 8 days the sediment yeast showed a better biological condition than did the suspended yeast; from the third to the fifth day the difference was insignificant (cf. B., 1929, 618, 792).

F. E. DAY.

**Fermentation and the surface of vessels.** F. EMSLANDER (Woch. Brau., 1929, 46, 401—403).—The local acceleration of fermentation by the cooling coils, and checking by the enamel walls of brewery vessels is associated with negative and positive electrostatic charges, respectively. The possible effects of different lining materials on the colloids of the beer are discussed.

F. E. DAY.

**Coagulation of proteins during boiling of unhopped wort.** W. WINDISCH, P. KOLBACH, and C. VOGL (Woch. Brau., 1929, 46, 403—406, 417—419).—The amount of protein precipitated from wort by boiling is strongly influenced by the intensity and duration of heating, even in flasks connected to a reflux condenser. The most consistent results were obtained by heating in a bath of boiling salt solution (b.p. 108.8°), but heating for 4 hrs. was insufficient to obtain maximum precipitation. Comparison of boiling a wort to half volume in a beaker (70 min.), for 2 hrs. in salt solution at 108.8°, and for 2 hrs. over a burner with a layer of asbestos paper, gave 3.27—3.92 (mean 3.54), 5.94—6.36 (mean 6.23), and 7.60—9.33 (mean 8.11) mg./100 c.c., respectively, of coagulable nitrogen. Heating at 1.5 atm. for 1 hr. and 2 hrs. and for 1 hr. at 2 atm. in an autoclave gave consistent results, but below those obtained by boiling at ordinary pressure for 4 hrs. The absence of actual ebullition may explain this, as wort heated on the boiling water-bath, though only about 1° lower in temperature than that boiling on the salt-bath, yielded 7.4 mg./100 c.c. against 12.9 mg./100 c.c. of coagulable nitrogen. The optimum hydrogen-ion concentration varies with the duration of boiling from  $p_H$  6.4 or above at 1—2 hrs. to  $p_H$  5.4—6.2 at 5 hrs. in the case of laboratory worts. In an unhopped brewery wort maximum precipitation with boiling for 3 hrs. was found at about  $p_H$  6.5. In the autoclave at 1.5 atm. for 1 hr. practically identical results were obtained at  $p_H$  5.74 and 6.37, though less nitrogen was precipitated than by 4—5 hrs. boiling.

F. E. DAY.

**Chemical composition and taste of potato spirit.** T. CHRZASZCZ, A. KŁODNICKI, and J. SUCHODOLSKI (Przemysł Chem., 1929, 13, 257—268).—The chemical composition, smell, and flavour of potato spirit are unaffected by the substitution of iron for copper in the distilling and rectifying apparatus. The type of distilling apparatus used appears not to affect the composition and quality of the distillate where the

continuous process is applied, and the quality of the spirit obtained with two-column stills is no better than that using one column only. Installations designed for periodic production yield spirit of inferior quality. Spirit of higher quality is obtained from stills giving more highly concentrated distillates. The use of fermentation vats possessing the smallest practicable surface, which should be as smooth as possible, is advised, as minimising the danger of undesirable bacterial infection. Rotten potatoes yield inferior spirit, often possessing a repulsive taste and odour, these faults depending on the nature of the factors causing decay, and being the greater the more the potatoes are decayed. Potatoes attacked by potato cancer yield spirit of poor quality. The appearance of large quantities of foam during fermentation, as well as hyperacidity of the mash, lead to the production of inferior spirit; the evolution of hydrogen sulphide during fermentation does not involve its presence in the spirit, nor are the taste and odour of the latter to any great extent affected by this type of fermentation. Increase in the acidity of the mash does not involve the production of a more acid spirit, and the potability of the spirit is not adversely affected by its acidity, or aldehyde and ether content. The unpalatability of spirit increases with its fusel oil content, as well as with the velocity with which it decolorises permanganate solution, although no close parallel can be drawn between chemical composition and palatability.

R. TRUSZKOWSKI.

**Tyramine as constituent of a Japanese vinegar.** K. MIYAJI (Z. physiol. Chem., 1929, 184, 157—162).—From rice vinegar succinic and *l-p*-hydroxyphenyllactic acids were isolated, but no tyramine. All three substances were obtained from sakékasu vinegar. The latter yielded two strains of *B. megatherium*, de Bary, capable of almost quantitative conversion of tyrosine into tyramine.

J. H. BIRKINSHAW.

**Relation of temperature of fermentation to quality of sauerkraut.** E. A. MARTEN, W. H. PETERSON, E. B. FRED, and W. E. VAUGHN (J. Agric. Res., 1929, 39, 285—292).—The quality of sauerkraut, which depends chiefly on a proper degree of acidity, was highest when the temperature of the fermentation lay between 15° and 18°. Sauerkraut produced at 18—24° was soft and of a pink colour, whilst that fermented at 4—8° possessed a bitter taste, poor texture, and a dark colour. A rise of 1.5—3°, which occurred during the first 8 days in fermentation vats kept at temperatures above that of the surrounding air, was coincident with, and was probably due to, the greatest activity of the bacteria.

C. RANKEN.

**[Examination of] foreign wines under the quartz lamp.** P. BERG and L. STOCKERT (Z. Unters. Lebensm., 1929, 57, 448—460. Cf. Werder and Zäch, Mitt. Lebensm. Hyg., 1928, 19, 60, 147).—As found by Werder and Zäch (*loc. cit.*) the quartz lamp can be used to determine whether a wine has been made from dried grapes or contains a considerable quantity of such wine, provided it is known by analysis that a concentrated must has not been used. The behaviour of Samos wine in ultra-violet light confirms the view of Günther

("Der Wein," Leipzig, 1918, 660) that it is prepared from fresh grapes. In the case of dessert wines of high extract content and high luminescence it is not at present possible to draw definite conclusions from luminescence phenomena. It appears probable that alcoholic extracts of all sugar-containing dried fruits show strong luminescence. W. J. BOYD.

**Behaviour of alcoholic distillates in ultra-violet light.** M. RÜDIGER and E. MAYR (*Z. Unters. Lebensm.*, 1929, 57, 561—566).—Various brandies derived from different raw materials, *e.g.*, apples, grapes, rye, maize, potatoes, were brought to an alcohol content of 33 vol.-%, and 100 c.c. of each were distilled into 9 equal fractions and 10 c.c. of residue, which were examined under the quartz lamp. The luminescence was most marked in the case of the fourth fraction and the residue. The brandies differed so little from one another that the method cannot be used for detecting adulteration of brandy. On diluting the liquors the luminescence appeared in an earlier fraction. From various brandies oily droplets of a fatty acid nature were obtained in the fraction distilling between 94° and 97°, which droplets showed strong luminescence. The luminescence of the fourth fraction was removed by precipitation with silver nitrate and restored by decomposition of the precipitate by sodium hydroxide solution. Lauric acid separated as a solid from the fractions poorer in alcohol and does not luminesce (*cf.* Grossfeld and Miermeister, *B.*, 1929, 145). When alcoholic solutions of octoic and decaic acids are distilled these acids separate as oily droplets which are highly and feebly luminescent respectively. Hexoic and butyric acids give luminescent solutions without separation of droplets. Luminescence in later fractions than the fourth indicates the addition of an artificial essence to the brandy.

W. J. BOYD.

**Is the volume of a saccharine liquid altered in fermentation?** C. LUCKOW (*Deut. Destillateur-Ztg.*, 1929, 50, 169; *Chem. Zentr.*, 1929, i, 2596).—Theoretically 62.5 c.c. of dissolved sugar (100 g.) should produce 64 c.c. of alcohol; a 2.5% deficiency is usually observed, but the volume is increased by the secondary products. In practice the volume is unchanged.

A. A. ELDRIDGE.

**isoValeric acid from fusel oil.** PIOTROWSKI.—See III. **Removal of fermentation-restraining humic substances from hydrolysates of hemicelluloses.** SCHMIDT and others.—See V. **Micro-determination of phosphate ions.** DENIGÈS. **Determination of nitrogen.** WIENINGER and LINDEMANN.—See VII. **Barley fat and malt fat.** TÄUFEL and RUSCH.—See XII. **Amylases in dry potato starch.** SPROCKHOFF.—See XVII. **Effect of flour on yeast.** MOHS and KÜHL.—See XIX.

#### PATENTS.

**Drying apparatus for hops and like materials.** R. D. BATCHELOR (B.P. 319,394, 20.6.28).—The hot gases in their journey from the fire to the hops pass through filter screens which are hinged and readily movable. Each screen consists of a layer of horse hair, woven asbestos, or other fibrous material, supported,

if desired, on wire netting. Spent iron oxide, lime, or other chemical reagent may be present on the screens.

C. RANKEN.

**Valuable products from organised substances.** R. GRIESSBACH and O. AMBROS, Assrs. to WINTHROP CHEM. Co., Inc. (U.S.P. 1,724,706, 13.8.29. Appl., 31.3.28. Ger., 6.4.27).—In the production of compounds from organised substances the cell walls are subjected to enzymic degradation before the usual extraction and pressing, so that the products can be readily separated without decomposition. The yield of ergosterol from yeast is increased by adding ethyl acetate, to initiate autolysis, before extracting with alcohol.

F. G. CLARKE.

**Production of butyl alcohol and acetone by fermentation.** DISTILLERS Co., LTD., and H. B. HUTCHINSON (B.P. 319,079, 25.6.28).—Starchy materials, such as manioc and the like, which have a low content of protein, are fermented in the presence of the non-protein nutrient ammonium phosphate. The process is applicable to every type of organism capable of producing butyl alcohol and acetone from starchy materials.

C. RANKEN.

**Cereal beverage and liquid.** H. HEUSER, Assr. to UNITED STATES PROCESS CORP. (U.S.P. 1,724,021, 13.8.29. Appl., 1.8.27).—Cereal liquids are stabilised after alcoholic fermentation by the addition of a quantity of pyrocatechol tannic acid not less than that required to precipitate the readily precipitable proteins.

L. A. COLES.

**Briquettes** (B.P. 294,879).—See II.

#### XIX.—FOODS.

**Bleaching of flour.** A. HEIDUSCHKA and A. WARLMONT (*Z. Unters. Lebensm.*, 1929, 57, 587—592).—The bran of flour is not sufficiently bleached by benzoyl peroxide and Novadelox to remove its undesirable effect on the appearance of the flour. Bleaching with benzoyl peroxide improves the baking qualities of the flour. There is a definite proportion of the agent which gives optimum results for a given flour: excess imparts a bluish tint to the bread. Bleaching at higher temperatures gives no better results. The action of benzoyl peroxide increases the water-soluble material of the flour in proportion to the quantity of the agent used. At the same time the total water-soluble carbohydrate decreases; water-soluble nitrogenous material and acid are unchanged.

W. J. BOYD.

**Test for bleaching of flour.** J. KULMAN (*Chem. Listy*, 1929, 23, 333—336, 375—382).—Flour bleached with chlorine or with nitrogen peroxide gives darker-coloured benzene extracts than does unbleached flour. Aged flour, however, also yields highly coloured extracts, so that in order definitely to establish whether the flour has been bleached, extraction with benzene-toluene or -xylene mixtures is applied, when brown or red chloro- or nitro-aromatic derivatives are obtained, unbleached flour giving light yellow extracts under these conditions.

R. TRUSZKOWSKI.

**Effect of flour on the fermentative power of yeast.** K. MOHS and H. KÜHL (*Z. Unters. Lebensm.*, 1929, 57, 443—448).—The toxicity of normal flour

towards yeast is changed to a favourable effect on increasing the degree of milling, owing to the increased proportion of bran, or by the addition of soya-bean flour. This is probably due to the phosphates contained in bran and bean flour. The diastase of germinating wheat has a favourable effect on yeast fermentation, counteracting the toxicity of the flour. High acidity of spoiled flour is not invariably accompanied by a correspondingly increased toxicity such as might be expected if the toxamine were a protein decomposition product. A high toxicity towards yeast does not unfavourably influence the baking qualities of the flour.

W. J. BOYD.

**Utilisation of skim milk.** A. BICKEL (Z. Unters. Lebensm., 1929, 57, 437—443).—The high nutritive value of "plasmon" (casein) in comparison with vegetable proteins is shown by the results of feeding tests on rats.

W. J. BOYD.

**Secretion of lipolytically active "rancid" milk.** G. KOESTLER, C. L. ROADHOUSE, and W. LÖRTSCHER (Landw. Jahrb. Schweiz, 1928, 42, 937—966; Chem. Zentr., 1929, i, 2116).—The occurrence of milk naturally containing lipase is discussed; a sample was high in caseinogen, lactose, and calcium, but low in citric acid and chlorine.

A. A. ELDRIDGE.

**Determination of lactose [in milk] from the refraction of the calcium chloride serum.** G. SCHULZE (Z. Unters. Lebensm., 1929, 57, 460—465).—The observation of Drost (B., 1925, 822) that Ackermann's table (B., 1917, 1025) for finding the lactose content of milk from the refraction of the serum gives results considerably lower than those found by gravimetric methods is confirmed. A new table, prepared by determinations of the refraction and of the lactose by Allihn's method, is provided which gives the true lactose content corresponding to the refraction if the milk is normal. If the content of lactose gravimetrically determined is lower than that given by the table, the presence of milk from diseased udders is indicated. A method of distinguishing diluted milk and milk defective through disease is thus available. The natural variations at the beginning and end of lactation must be considered.

W. J. BOYD.

**Melezitose in honey-dew honey from the lime.** F. E. NOTTBOHM and F. LUCIUS (Z. Unters. Lebensm., 1929, 57, 549—558).—The trisaccharide melezitose was found in honey-dew honey from the lime as a grey sediment consisting of anhydrous lamellæ (m.p. 153—156°) and rhombic prisms containing water of crystallisation which was completely driven off by heating in the water-oven for 2 hrs. The sugar was much less soluble in water than sucrose. It had a faintly sweet taste and reduced Fehling's solution very slowly. Hydrolysis with 1% sulphuric acid reduced the specific rotation from 89.5° to 66°. More concentrated acid reduced it further to 26.0°, but after destruction of the lævulose formed it rose to 36.0°, or to two thirds that of dextrose. The intermediate products were turanose and dextrose, identified by their osazones. Turanose was shown to consist of lævulose and dextrose. The insolubility of melezitose in water and its great resistance to the action of the ordinary sugar-splitting

enzymes are suggested as the cause of the toxic action on bees which such a honey possesses. Yeast, invertase, and diastase have no effect on it. (Cf. Kuhn and von Grundherr, A., 1926, 1127.)

W. J. BOYD.

**Detection of [hydr]oxymethylfurfuraldehyde in honey and artificial honey.** J. FIEHE and W. KORDATZKI (Z. Unters. Lebensm., 1929, 57, 468—470).—The honey (5 g.) is stirred in a mortar with 10 c.c. of ether, and the ether is poured off and gently evaporated. The residue is dissolved in 0.5 c.c. of water, and to the solution 0.5 c.c. of 32% hydrochloric acid is added followed by 1 c.c. of 0.625% solution of phloroglucinol in 16% hydrochloric acid. A reddish coloration and a red-brown precipitate are formed in the presence of hydroxymethylfurfuraldehyde. Neither coloration nor precipitate occurs when pure honey is used. By comparison with synthetic mixtures of natural and artificial honey the height of the layer of sediment can be used as a measure of the adulteration of any sample. Genuine honey heated at 80° for 2 hrs. and then warmed for 2 hrs. on the boiling water-bath gives no precipitate, but with Fiehe's resorcinol reagent an evanescent red coloration is obtained and with phloroglucinol a strong lemon-yellow coloration which gradually fades. The latter reaction is characteristic for heated honey and is not due to hydroxymethylfurfuraldehyde, for the production of which the presence of acid is necessary.

W. J. BOYD.

**Determination and content of water in dried fruit.** J. PRITZKER and R. JUNGKUNZ (Z. Unters. Lebensm., 1929, 57, 520—524).—The apparatus described is suitable for the determination of water in dried fruits by distillation with tetrachloroethane, xylene, or other volatile liquid lighter or heavier than water. It can also be used for the determination of water in coal or other commodities. With 75 c.c. of tetrachloroethane and 25 g. of dried fruit a determination can be completed in 20 min. Good agreement was found between the tetrachloroethane and the xylene methods. The drying-oven method requires 10—14 hrs. and gives less uniform results. Moisture contents found for various dried fruits are given. Dried apples, apricots, and peaches should contain not more than 30% of water.

W. J. BOYD.

**Determination of husk in cocoa and cacao products.** M. WAGENAAR (Z. Unters. Lebensm., 1929, 57, 525—537).—The sclereide tissue in 1 g. of fat-free cocoa powder is isolated by heating with 20 c.c. of 5% potassium chlorate solution and 10 c.c. of 4*N*-hydrochloric acid for 10 min. on the water-bath. Then 25 c.c. of 4*N*-sodium hydroxide are added, and the heating is continued for 10 min. more. After settling, the clear liquid is drawn off, the sediment washed repeatedly by decantation, and recovered by centrifuging. The whole or an aliquot portion is suspended in a weighed quantity of glycerin, 6 drops of known total weight are placed on slides under calibrated cover-slips, and the total area of sclereide tissue in them is measured, using polarised light (1 g. of fat-free powdered husk contains 2100 mm.<sup>2</sup> of sclereide tissue.) The content of husk in 100 g. of fat-free cocoa is obtained by dividing the area of sclereide tissue in 1 g. by 21. W. J. BOYD.

**Chemical and physical examination of flesh and flesh fluid of various animals: the Feder ratio.** A. GRONOVER and E. WOHLICH (*Z. Unters. Lebensm.*, 1929, **57**, 592—604).—Efforts to establish chemical and physical constants (*e.g.*, f.p. depression, sp. conductivity, sp. gr., and chlorine content) for the flesh sera of different animals were unsuccessful. Not only different individuals but different portions of the same individual gave widely varying values. Analyses are given for flesh from different parts of various animals. That of calves only a few weeks old has usually a Feder value above 4, whereas that of normally-fed cattle is usually below 4 or only slightly above. Variations in the water content of different parts were inconsistent and sometimes even reversed in different individuals. Nevertheless, contrary to the opinion of Bongert and Muchlinsky (*ibid.*, 1929, **57**, 248), determinations of the Feder value are of great use in detecting addition of water to minced flesh, but suspicions founded thereon must be confirmed otherwise. Naturally-occurring moisture in fat has little influence on the Feder value. W. J. BOYD.

**Spectroscopy of food dyes.** HEATH.—See IV.  
**Micro-determination of phosphate ions.** DENIGÈS.  
**Determination of nitrogen.** WIENINGER and LINDEMANN.—See VII.  
**Metals in dairy equipment.** HUNZIKER and others.—See X.  
**Quality of sauerkraut.** MARTEN and others.—See XVIII.

#### PATENTS.

**Reduction of grain and its subsequent conversion into bread or like product.** E. RABINOWITSCH (B.P. 317,571, 8.6.28).—The cleaned and dried grain is washed for  $\frac{3}{4}$ — $\frac{3}{4}$  hr. in 8% sulphuric acid, which is then removed thoroughly by running water. The grain is allowed to steep for about 24 hrs., when germination begins and lasts for 24—36 hrs., during which the grain is exposed on a perforated tray to a current of air and a stream of water. When the decomposition of the gluten reaches the requisite stage, 2.5% of salt, 0.3% of lime (added as 10% lime water), and 0.5% of glycerin are added and the mixture is kneaded to a coarse dough, which is fermented in a closed space at 86—95° for 12—14 hrs., the pressure rising to about  $4\frac{1}{2}$  atm. When fermentation ceases the pressure is suddenly released, yeast is kneaded in, and the dough baked into wholemeal bread. E. B. HUGHES.

**Removing caffeine and unsavoury substances from roasted coffee.** J. PÄFFGEN (B.P. 302,332, 16.8.28).—Coffee liquor as prepared for drinking is filtered through absorbent material such as active carbon, whereby it is claimed that caffeine, pyridine, etc. may be removed, the carbon being preferably treated with fumes from the roasting of coffee to render it a non-absorbent of the volatile (aroma) constituents of the liquor. In another example an absorbent, obtained by treating suitable organic substances with zinc chloride, is treated with smoke gases of aromatic resins. Suitable apparatus is described. E. B. HUGHES.

**Production of edible paste from the liver of cod fish and the like.** H. J. EGGE (B.P. 319,529,

29.10.28).—The liver is treated with dilute acid to remove surface (oxidised) oil, then heated to 70°, converted into a paste with casein etc. as binder, canned, and sterilised. E. B. HUGHES.

**Cereal beverage (U.S.P. 1,724,021).**—See XVIII.

#### XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Purification and preservation of ether for anaesthetic use.** S. PALKIN and H. R. WATKINS (*Ind. Eng. Chem.*, 1929, **21**, 863—867).—The tendency of ether to develop peroxide and aldehyde during storage is entirely stopped by the addition to the container of a few g. of a preservative consisting of fibrous asbestos moistened with (a) a saturated solution of potassium permanganate in 30% caustic soda or potash, or (b) 1 g. of pyrogallol in 15 c.c. of 30% caustic potash. Comparative tests, lasting over a year, showed that deterioration apparent in the untreated samples was thereby completely inhibited, even when the ether was exposed to direct sunlight and elevated temperatures, and no contamination of the ether resulted by such addition. T. H. MORTON.

**Purification of ethyl acetate for the evaluation of opium and opium preparations.** G. VON MIKÓ (*Ber. Ungar. pharm. Ges.*, 1928, **4**, 392—396; *Chem. Zentr.*, 1929, **i**, 1846).—Ethyl acetate which has suffered decomposition is shaken with 0.15% calcium hydroxide solution; it is preferably preserved over neutral potassium tartrate. A. A. ELDRIDGE.

**Determination of chlorine and cyanogen in chloral cyanohydrin.** G. FAVREL (*Bull. Soc. Pharm. Bordeaux*, 1928, **66**, 139—142; *Chem. Zentr.*, 1929, **i**, 2088).—The chloral cyanohydrin (0.5 g.; cf. Carré, *Prod. pharm. ind.*, 1909, **2**, 37) is boiled for 0.5 hr. under reflux with amyl-alcoholic sodium hydroxide; the cold liquid is extracted several times with water. The extracts are diluted to 200 c.c.; in 100 c.c. the cyanide is determined with silver nitrate by Denigès' method, whilst the remainder is boiled with nitric acid and silver nitrate to determine the chlorine gravimetrically or volumetrically. The method is inapplicable to nitriles. A. A. ELDRIDGE.

**Synthesis of camphor. Purity of isobornyl acetate.** M. T. LACRUE (*Bull. Inst. Pin.*, 1929, Special No., 15—30; *Chem. Zentr.*, 1929, **i**, 2303).—Losses occur in the "borneol oil," the amount of which is diminished when purer (97—99%) acetate is employed. The fractionation of the 90% product is discussed. A. A. ELDRIDGE.

**Preparation of a solution of iron albuminate.** I. A. OBERHARD (*Farm. Zhur.*, 1928, 702—705).—Dilute solutions of ferric chloride and albumin at 50° are mixed and the solution is neutralised with 15% sodium hydroxide solution at 50°. The precipitate is washed free from chlorine, dissolved by the addition of alkali, and the remaining pharmacopœial components are added.

CHEMICAL ABSTRACTS.

**Behaviour of some colloidal silver preparations in aqueous solution.** O. TOMIČEK (*Časopis Českoslov. Lék.*, 1928, **8**, 196—202; *Chem. Zentr.*, 1929, **i**, 2443).—

The proportion of ionised silver in commercial preparations has been determined, usually potentiometrically.

A. A. ELDRIDGE.

**Titration of mercurial preparations used in pharmacy.** A. WÖHLK (Dansk Tidsskr. Farm., 1929, 3, 225—244).—(a) In the analysis of tablets of mercuric chloride containing sodium chloride, 20 c.c. of an aqueous solution of the tablets (containing about 2.5%  $\text{HgCl}_2$ ), 50 c.c. of 0.1N-caustic soda, and 5—6 c.c. of 3% hydrogen peroxide are boiled until the precipitated mercury has agglomerated. The solution is filtered and the excess caustic soda in the filtrate titrated against methyl-red with 0.1N-hydrochloric acid. (1 c.c. of  $\text{N-NaOH} \equiv 0.01356$  g. of  $\text{HgCl}_2$ .) (b) Rupp's method (cf. B., 1908, 1179) gives satisfactory results with solutions of mercuric chloride (from 1% upwards). (c) In the examination of the solution of mercuric chloride (1 pt.) in 5% acetic acid (300 pts.), 50 g. of the solution, 15 g. of Liq. kalii arsenicosi, and 30 c.c. of 2N-caustic soda are boiled for 5 min., cooled, and diluted with 100 c.c. of water. After addition of 8 c.c. of 30% acetic acid and 3 g. of sodium bicarbonate, the solution is titrated with 0.1N-iodine, using starch indicator. (d) For solutions containing not less than 1% of mercuric chloride the following method is recommended. Mercury is precipitated by potassium iodide, the end-point being indicated by the liberation of iodine by hydrogen peroxide. A mixture of 20 c.c. of the mercurial solution, 0.5—1.0 c.c. of 3% hydrogen peroxide, 5 c.c. of dilute sulphuric acid, and 3—5 c.c. of starch solution is titrated with 0.1N-potassium iodide until the mercuric chloride turns brick-red. Blue rings form above the precipitate and, on shaking, the whole liquid turns blue. (e) Mercuric oxide can be determined (i) by dissolving in hydrochloric acid and, after adding sodium chloride, titrating residual acid with caustic soda; or (ii) by dissolving the oxide in water containing excess of sodium thiosulphate (10 times the weight of oxide), maintaining the temperature below 35° to prevent decomposition of the complex, and titrating the resultant sodium hydroxide with hydrochloric acid. (f) For the determination of calomel iodometric methods are recommended. An indirect method is to boil the material with hydrogen peroxide and caustic soda (cf. a). (g) Mercuric amidochloride may be determined by decomposition with potassium iodide or sodium thiosulphate followed by titration of the liberated base, or by titration with hydrochloric acid in the presence of sodium chloride. (h) Mercuric cyanide and oxycyanide may be determined by decomposition with sodium thiosulphate or potassium iodide. The alkali cyanide is titrated with acid against methyl-orange. (i) For the determination of mercuric oxide or mercuric amidochloride in ointments, the ointment base is removed by dissolving in benzene or ether-acetone, the residue being then dissolved in acid and the excess titrated back after addition of sodium chloride.

H. J. DOWDEN.

**The tannin of the purple digitalis.** P. BOURCET and A. FOURTON (Bull. Soc. chim., 1929, [iv], 45, 776—778).—The green coloration given by the digitalis extracts from the fresh purple digitalis is due to caffeic acid and not to the presence of gallic acid or tannins in the soluble glucoside complex.

R. BRIGHTMAN.

**Determination of fats in drugs.** E. GLASER and A. HALBERSTAM (Arch. Pharm., 1929, 267, 526—532).—Sulphuric acid (*d* 1.81; 10 c.c.) is introduced into a Gerber butyrometer, a small amount of water added cautiously, then 0.4—1.0 g. of the powdered drug, 1 c.c. of amyl alcohol, and sufficient water to make a total volume of 22 c.c. The mixture is agitated, heated at 60—70° for a short time, centrifuged, and re-warmed. The volume of fat which separates is converted into weight by means of a factor which varies somewhat for the substances used. This factor is determined by the usual ether extraction method. If the fat layer is contaminated with the powder used, it is advisable to carry out the determination with a smaller quantity (7—8 c.c.) of sulphuric acid.

H. BURTON.

**Application of "critical solution temperature" to pharmaceutical investigations.** F. WRATSCHKO (Pharm. Presse, 1929, 34, 143—145).—Values for a number of pairs of organic substances are recorded.

CHEMICAL ABSTRACTS.

**Application of various micro-methods in qualitative toxicological and pharmaceutical analysis.** L. FUCHS and A. MAYRHOFFER (Mikrochem., 1929, Pregl Fest., 106—126).—The application of microchemical methods, especially sublimation and determination of the refractive index of the crystalline sublimate, to the identification of various pharmaceutical products has been studied, and the results obtained are critically discussed.

H. F. HARWOOD.

**Transparent emulsions of some essential oils.** W. F. WHITMORE and R. E. LINEHAN (Ind. Eng. Chem., 1929, 21, 878—880).—The properties of transparent emulsions prepared by dispersing the oil in an aqueous solution of invert sugar having a refractive index equal to that of the oil are described. Of gelatin, gum-arabic, agar-agar, and tragacanth, the first was the best peptising agent and the last two were unsatisfactory. Transparent emulsions of orange, peppermint, and rose oils were easily made by this method, but for the preparation of emulsions of wintergreen, anise, and cinnamon oils the refractive indices of the oils themselves had to be lowered by the addition of coconut oil ethyl esters to a value low enough to permit the use of an aqueous sugar solution of sufficiently low concentration to pass through the colloid mill without difficulty. These emulsions developed a coconut oil taste on keeping. Graining of certain of the emulsions was obviated by using a mixture of 2 pts. of invert sugar and 1 pt. of sucrose instead of invert sugar alone.

E. H. SHARPLES.

**Constituents of East Indian lemon-grass oil.** F. ELZE (Riechstoffind., 1929, 4, 23—24; Chem. Zentr., 1929, i, 2248).—Methylheptenol, methylheptenone, geraniol, nerol, and farnesol were isolated.

A. A. ELDRIDGE.

**Essential oil of *Juniperus excelsa*.** G. V. FIGULEVSKI and Y. N. LOVYAGIN (Trans. Sci. Chem. Pharm. Inst. Moscow, 1927, No. 17, 151—158).—The b.p. and yields of fractions are tabulated. Pinene and cedrol were isolated.

CHEMICAL ABSTRACTS.

**Essential oils from Irish-grown plants.** V. Oil of dill. J. REILLY, P. J. DRUMM, and C. BOYLE (Econ.

Proc. Roy. Dublin Soc., 1929, 2, 415—418).—Following a review of the cultivation of *Anethum graveolens* and the chemical properties of dill oil produced in Europe, India, and the East Indies, it is shown that *A. graveolens* grown near Cork gave in 1926—27, 7450 lb. of fruit and 258 lb. of redistilled oil per acre, a 3½% yield of oil. It had  $d_{20} 0.9135$ ,  $n_D 1.48513$ ,  $\alpha_{70} 54'$ , percentage of carvone 37.8, and solubility in 70% alcohol 1:6. Similar values were obtained in 1927—28, except that the percentage of carvone was 43—44. E. HOLMES.

**Modified determination of total geraniol content in citronella oil.** M. VAN DER SLIK and J. VERMEULEN (Chem. Weekblad, 1929, 26, 482—483).—The use of anhydrous sodium acetate involves in a damp climate repeated preparation of fresh material, and is laborious and slow. Anhydrous sodium carbonate is found to give equally satisfactory results. The data obtained with 97 samples, using both acetate and carbonate, are tabulated. S. I. LEVY.

**Nerol and its esters.** A. RECLAIRE (Deut. Parfüm. Ztg., 1929, 15, 71—73; Chem. Zentr., 1929, i, 2249).—The following limiting data for neryl esters are recorded: formate,  $d_{15} 0.9163$ ,  $0.9169$ ,  $n_D^{20} 1.4558$ ,  $1.4578$ ; acetate  $d_{15} 0.903$ ,  $0.906$ ,  $n_D^{20} 1.451$ ,  $1.454$ ; propionate  $d_{15} 0.9044$ ,  $n_D^{20} 1.4550$ ; isobutyrate  $d_{15} 0.8915$ ,  $0.8936$ ,  $n_D^{20} 1.4508$ ,  $1.4527$ ; butyrate  $d_{15} 0.8968$ ,  $0.8986$ ,  $n_D^{20} 1.4539$ ,  $1.4536$ ; isovalerate  $d_{15} 0.8898$ ,  $n_D^{20} 1.4531$ .

A. A. ELDRIDGE.

**Evaluation of Liquor cresoli saponatus according to D.A.B. VI.** K. KRAFFT and G. ZEITLER (Süddeut. Apoth.-Ztg., 1928, 68, 613—615; Chem. Zentr., 1929, i, 2212).

**Determination of iodoform.** JÁNSKÝ.—See III. **Determination of hydrogen peroxide.** FRERIGHS. **Determination of phosphorus in "Phosphor solutus."** BÖTTGER.—See VII. **Vitamins in canned foods.** KRAMER and others.—See XIX.

#### PATENTS.

**Manufacture of pharmaceutical products.** I. G. FARBENIND. A.-G. (B.P. 282,453, 19.12.27. Ger., 20.12.25. Addn. to B.P. 267,169; B., 1927, 379).—Amino-derivatives of the quinoline, di- and tri-arylmethane, azine, oxazine, thiazine, acridine, and xanthen series are prepared in which the amino-group carries a substituent comprising a nitrogenous alicyclic or heterocyclic residue linked to the nitrogen directly or through an aliphatic carbon chain. From 8-aminoquinoline and 1- $\beta$ -chloroethylpiperidine is obtained 8-( $\beta$ -1-piperidylethyl)aminoquinoline, m.p. 59—60°, b.p. 180—182°/1 mm. (hydrochloride, m.p. 190—191°). 2-Bromocyclohexyldimethylamine, prepared from the alcohol and hydrogen bromide, gives with 8-amino-6-methoxyquinoline 8-(2-dimethylamino-1-cyclohexyl)amino-6-methoxyquinoline, b.p. 192—195°/1 mm. Methyltriacetonalcamine is converted by hydrogen bromide into 4-bromo-1:2:2:6:6-pentamethylpiperidine, which reacts with 8-amino-6-methoxyquinoline to yield 8-(1:2:2:6:6-pentamethyl-4-piperidyl)amino-6-methoxyquinoline, b.p. 215—218°/0.5 mm. 1- $\beta$ -(*N*-Methylaniline)ethylpiperidine, obtained from methylaniline and 1- $\beta$ -chloroethylpiperidine, is converted

by means of nitrosodimethylaniline and sodium thio-sulphate into the thiazine. C. HOLLINS.

**Manufacture of unilaterally acylated diamines of therapeutic activity.** O. Y. IMRAY. From Soc. CHEM. IND. IN BASLE (B.P. 307,719, 6.12.27).—Monoacylated diamines having depressant or other therapeutic action are obtained from alkylendiamines, especially *NN*-diethylethylenediamine, and heterocyclic acids. The following are described: *N*- $\beta$ -diethylaminoethylamides of 2-phenylquinoline-4-carboxylic acid [m.p. 70—75°; dihydrochloride, m.p. 185—186° (decomp.); isomeric 1:2:3:4-tetrahydro-derivatives, m.p. 132° and 97.5°, respectively (dihydrochlorides, m.p. 192° and 225°, respectively, both decomp.)], of 2-ethoxyquinoline-4-carboxylic acid (m.p. 97°), of 3- $\beta$ -hexyl-2-naphthaquinoline-1-carboxylic acid (dihydrochloride, m.p. 274—275°), of acridine-*ms*-carboxylic acid (m.p. 105—106°; 1:2:3:4-tetrahydro-derivative, m.p. 97—98°), of 1-phenyl-5-pyrazolone-3-carboxylic acid (hydrochloride, m.p. 220°), of 5:6-methylenedioxy-8-phenyl-2:3-dihydroisoquinoline-2-carboxylic acid (oxalate, m.p. 206—207°), of 6-ethoxy-2-phenylquinoline-4-carboxylic acid (m.p. 127—128°), of quinoline-3-carboxylic acid (b.p. 245°/4 mm.), of 2-methoxyquinoline-4-carboxylic acid (m.p. 94°), of 2:3-diphenylquinoline-4-carboxylic acid (m.p. 146—147°), of 2- $\beta$ -phenylethylquinoline-4-carboxylic acid (m.p. 98—99°), and of 2-styrylquinoline-4-carboxylic acid (m.p. 98—99°; hydrochloride m.p. 122°); *N*- $\beta$ -diethylaminoethyl-*N*-ethylamides,  $\text{NEt}_2\text{CH}_2\text{CH}_2\text{NEt}\cdot\text{COR}$  of 2-phenyl-1:2:3:4-tetrahydroquinoline-4-carboxylic acid, of 1:2:3:4-tetrahydroacridine-*ms*-carboxylic acid (b.p. 189—191°/0.015 mm.), and of 2- $\beta$ -phenylethylquinoline-4-carboxylic acid (b.p. 187—188°/0.007 mm.); 6-aminoethylamide of 2-phenylquinoline-4-carboxylic acid (m.p. 124—125°); 3-*N*-piperidinoethylamides of 2-phenylquinoline-4-carboxylic acid (hydrobromide, m.p. 102°), of pyridine-3-carboxylic acid (hydrochloride, m.p. 182—183°), and of quinoline-4-carboxylic acid; and acridine-*ms*-carboxylic *N*- $\epsilon$ -diethylamino-*n*-amylamide, m.p. 84°. *NNN*'-Triethylethylenediamine has b.p. 160—165°; 2- $\beta$ -phenylethylquinoline-4-carboxylic acid melts at 221—222°. C. HOLLINS.

**Anæsthetic compound.** R. ADAMS and O. A. BARNES, Assrs. to ABBOTT LABS. (U.S.P. 1,724,248, 13.8.29. Appl., 13.1.28).— $\omega$ -Piperidylalkyl *p*-aminobenzoates, prepared as already described (A., 1927, 672), are claimed. R. BRIGHTMAN.

**Manufacture of 4-amino-3-hydroxyphenylarsinic acid.** I. E. BALABAN, and MAY & BAKER, LTD. (B.P. 309,249, 24.1.28. Cf. Balaban, A., 1928, 655).—4-Nitro-3-hydroxybenzenearsinic acid, obtained by hydrolysis of the 3-chloro-compound with alkali, is reduced with alkaline glucose, hyposulphite, or ferrous sulphate solution. C. HOLLINS.

**Manufacture of pyridines.** I. OSTROMISLENSKY, Assr. to PYRIDUM CORP. (U.S.P. 1,724,305, 13.8.29. Appl., 23.9.27).—Benzenediazonium salts are coupled in weakly acid medium with 2:6-diaminopyridines, and the *py*-diazoamino-compound, e.g., 2-phenyldiazoamino-6-aminopyridine, m.p. 117°, yellowish-brown, is converted quantitatively into, e.g., 4-benzeneazo-2:6-



diaminopyridine, m.p. 137°, by boiling its hydrochloride with water. B. BRIGHTMAN.

**Obtaining and separating physiologically active substances from male generative organs.** Soc. CHEM. IND. IN BASLE (B.P. 298,092, 1.10.28. Switz., 30.9.27).—The male generative organs, *e.g.*, bull testicles, are extracted with water-soluble solvents, *e.g.*, alcohol, and with water-insoluble solvents, *e.g.*, ether, in either order, the two solutions are mixed, preferably after concentration of the alcoholic extract, the mixture with water is shaken, and, after settling, the aqueous and ethereal layers are worked up separately into water-soluble and lipid-soluble extracts, respectively. L. A. COLES.

**Extracting nicotine and ammonia from tobacco and its products.** F. R. ABLETT (B.P. 317,564, 5.6.28).—The tobacco etc. is heated at 60–205° on perforated trays in an oven provided at the top with an outlet pipe conveying vapours etc. to a "scrubber" filled with substances, *e.g.*, charcoal, lime, tannic acid, phosphotungstic acid, gypsum, etc., having an affinity for the deleterious constituents in the vapour, and the residual gases are pumped from the scrubber into the lower end of the oven. At the end of the treatment the tobacco is cooled in the oven after the addition of sufficient water to restore its normal moisture content. L. A. COLES.

**Manufacture of a chlorinised and ozonised topical remedy.** B. E. CLARKE (U.S.P. 1,724,562, 13.8.29. Appl., 10.12.25).—A vehicle, *e.g.*, vaseline or cacao butter, is impregnated with chlorine and ozone by passing the latter mixed with pure dry air into it in melted condition. On contact with the body nascent oxygen and chlorine are liberated. F. G. CLARKE.

**Pharmaceutical products.** H. HAHN and L. SCHÜTZ, Assrs. to WINTHROP CHEM. Co., INC. (U.S.P. 1,723,695—6, 6.8.29. Appl., [A] 27.8.23, [B] 24.3.27. Ger., [A, B] 6.11.22).—See G.P. 406,151; B., 1925, 426.

**Manufacture of arseno-compounds of the pyridine series.** A. BINZ and C. RÄTH (U.S.P. 1,702,334, 19.2.29. Appl., 8.11.24. Ger., 19.11.23).—See B.P. 250,287; B., 1926, 512.

**Removing caffeine from coffee** (B.P. 302,332).—See XIX.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Photography and the wave-theory of light. Clash between ray and wave theories of image formation.** T. SMITH (Proc. 7th Internat. Congr. Phot., 1928, 276—292).—The ray and wave theories of image formation are discussed. J. W. GLASSETT.

**Photographic method of investigating the colour of light sources, and the reflecting power of coloured fabric and other substances.** P. W. CUNLIFFE and F. D. FARROW (Proc. 7th Internat. Congr. Phot., 1928, 327—333).—The light from the source to be investigated is divided into a series of spectral bands by transmission through a number

of colour-filter strips arranged side by side, and the intensity in each wave-length band is compared by the normal photographic wedge method with the intensities in the corresponding bands of a standard light source. J. W. GLASSETT.

**Function of gelatin in photographic emulsions.** S. E. SHEPPARD (Phot. J., 1929, 69, 330—336).—It is suggested that the protective action exerted by gelatin on the silver halides depends on the formation of an oriented adsorption layer on the surface of the halide of certain groups in the gelatin containing active nitrogen atoms. Evidence is given for the existence of the :C:NH and  $\cdot N \left\langle \begin{array}{c} C \cdot C \\ C \cdot C \end{array} \right\rangle N \cdot$  groups in gelatin, and

an attempt is made to correlate the reversible effect on the sensitivity of emulsions caused by changes in  $p_H$  with the modification of this adsorption layer produced by the tautomeric changes taking place under the influence of  $p_H$  changes in compounds possessing such groups. J. W. GLASSETT.

**Photographic sensitivity of photographic layers.** F. WEIGERT (Proc. 7th Internat. Congr. Phot., 1928, 87—91).—It is suggested that the changes which produce photodichroism in photochloride layers also cause developability of the silver halide. The silver halide crystals are not homogeneous, but contain complexes of gelatin, primary silver, and silver halide, the structure of which is altered by the action of the light in such a way that the action of the reducing agents during development is promoted by the resultant change in absorption properties. J. W. GLASSETT.

**Theory of photographic light sensitivity.** A. STEIGMANN (Proc. 7th Internat. Congr. Phot., 1928, 258—262).—The sensitiveness of high-speed emulsions is not explained merely by the presence of ripening nuclei, for it has been shown that there are a large number of ripening nuclei in a high state of dispersion upon the surface of the fine grains of emulsions of low sensitivity. The high sensitivity of the rapid emulsion is due to the fact that the relatively few active ripening nuclei strongly orient the crystallisation of the silver formed by the decomposition of the silver halide, thereby causing a rapid growth of the nuclei to a size sufficient to induce development. J. W. GLASSETT.

**Preparation of a paper film for negatives and positives.** L. HERVÉ (Bull. Soc. Franç. Phot., 1929, 16, 182—188).—A linen-rag paper of medium strength is bathed in a solution composed of 60 g. of soft gum copal dissolved in 500 g. of benzene, dried, and mounted on a flat glass plate. A coating of a 10% gelatin solution is then applied, allowed to dry slowly, and the emulsion poured on. When dry the paper film is stripped from the glass support. J. W. GLASSETT.

**Contrast of photographic printing paper.** L. A. JONES (Proc. 7th Internat. Congr. Phot., 1928, 216—235).—The contrast of the photographic paper is dependent on (a) the rate factor, which is given by the value of the gradient of  $dD/d \log E$  of the characteristic curve ( $D - \log E$ ), and (b) on the extent factor, which is the difference between the maximum and minimum

densities. A contrast value, which comprises both these factors, is given by the area under the curve obtained by plotting the density ( $D$ ) against the gradient ( $dD/d \log E$ ) at those densities.

J. W. GLASSETT.

**Expression of [photographic] plate speed in terms of minimum useful gradient.** L. A. JONES and M. E. RUSSELL (Proc. 7th Internat. Cong. Phot., 1928, 130—143).—The specification of speed in terms of inertia is not altogether satisfactory, and fails particularly in those cases where the foot of the characteristic curve alone is being used. A more useful expression of speed, especially from the point of view of tone reproduction requirements, may be derived from the exposure ( $E$ ) at which a fixed minimum useful gradient ( $dD/d \log E$  of the  $D$ - $\log E$  curve, where  $D$  is the measured density) is obtained, measured at a definite degree of development. The most suitable value of the minimum useful gradient to cover all classes of work is the subject of further experiments.

J. W. GLASSETT.

**Dependence of the resolving power of photographic materials on the contrast in the test object.** O. SANDVIK (Z. wiss. Phot., 1929, 27, 60—76, and Proc. 7th Internat. Congr. Phot., 1928, 243—256).—With low contrast values the resolving power was found to be very small, but it rapidly increased with increasing contrast. The relation between the resolving power ( $R$ ) and the density ( $D$ ) of the opaque parts of the test object, *i.e.*, the measure of the contrast, can be expressed by means of the exponential equation  $R = C(1 - e^{-\alpha D})$ , where  $C$  and  $\alpha$  are constants. The resolving power varied widely with the exposure, but was almost independent of the degree of development.

J. W. GLASSETT.

**Relation between time and intensity in photographic exposure.** L. A. JONES and V. C. HALL (Proc. 7th Internat. Congr. Phot., 1928, 115—128).—With rapid emulsions the density developable for a fixed exposure varies very little over a wide range of intensities, whilst for slow emulsions the density diminishes rapidly at low intensities. The optimal intensity, at which the density produced is greatest, tends towards higher values for slow emulsions. The behaviour of emulsions above and below the region of optimal intensity may be represented by the equation  $\log E = \log (It_0/2)[(I/I_0)^a + (I/I_0)^{-a}]$ , which expresses the relation between the exposure ( $E$ ) required to produce a constant density at any intensity  $I$ , and  $I_0$  and  $t_0$ , the values of intensity and time at optimal intensity for the density in question.

J. W. GLASSETT.

**Mechanism of latent image formation.** F. C. TOY (Proc. 7th Internat. Congr. Phot., 1928, 14—34).—Existing literature dealing with the photo-conductivity effects exhibited by silver halides and similar substances is reviewed. The similarity of the position of maximum photo-conductivity effects shown by the same halides of different metals seems to suggest that the anions are the light-sensitive ions. The results obtained by Coblenz with silver bromide layers, which showed a maximum effect approximately at  $\lambda$  4600 with very low effects in the violet and ultra-violet regions, are shown to

be due to the thick silver bromide layer used, which favoured a much stronger absorption of blue light than of light of shorter wave-length. A series of experiments, using layers the thickness of which varied from about  $660\mu$  to  $20\mu$ , showed that as the thickness decreased so the effects in the violet and ultra-violet regions increased relatively to those in the blue, until the maximum in the blue finally disappeared. At the same time an increase in the magnitude of the effects was observed. With such very thin layers the three curves representing (i) the relative photo-conductivity effects, (ii) the relative photographic effects as measured by a determination of the number of centres formed by exposure of single-layer plates of a silver bromide emulsion, and (iii) the relative light absorption, each plotted against the wave-length for equal incident energy, are very nearly the same. These facts indicate that in all probability the primary stage of the photographic mechanism is intimately connected with that producing conductivity changes on illumination, consisting in the liberation of an electron from the anion of the silver halide crystal and the formation of neutral silver and free halogen.

J. W. GLASSETT.

**Principles and practice of [photographic] fixation.** F. F. RENWICK (Phot. J., 1929, 69, 310—314).—The theory of fixation and the factors influencing the rate of fixation in sodium thiosulphate solutions are outlined.

J. W. GLASSETT.

#### PATENTS.

**Photographic films.** MIMOSA A.-G., and W. NAEWIGER (B.P. 316,811, 29.9.28).—Material suitable for the production of positives, which may be viewed both by transmitted and reflected light, is prepared by coating both sides of a semi-opaque film base, cast, for example, from a mixture of cellulose nitrate and tinted barium sulphate, with sensitive emulsion, the rear coating being the more sensitive. Chloro-bromide and bromide emulsions may be used for the front and rear sides, respectively. The image produced on the rear side gives the increased density required when the print is used as a transparency.

J. W. GLASSETT.

**Light-sensitive films or carriers therefor. Subtractive colour photography.** (A) W. T. L. BECKER and L. W. OLIVER; (B) W. T. L. BECKER, L. W. OLIVER, H. D. MURRAY, and COLOUR PHOTOGRAPHS (BRITISH & FOREIGN), LTD. (B.P. 317,909 and 317,911, [A] 26.5.28, [B] 26.5.28 and 18.1.29).—(A) To facilitate registration in the preparation of composite prints on cellophane or similar materials, the dried sheets are brought to a known state of expansion before use by either of the following methods. (i) The materials immersed in the different sensitising baths are brought to the same known state of expansion and are then mounted on a stiff support, such as celluloid, and dried. (ii) The solid contents of the different baths are adjusted by the addition of experimentally determined amounts of a neutral substance, *e.g.*, sugar, such that each bath will then cause the same swelling and contraction of the material during immersion and drying. (B) Water-permeable, homogeneous, transparent substances which retain their mechanical properties while wet, as, *e.g.*, cello-

phane or specially treated cellulose derivatives, are used in the form of films as the support and medium for the light-sensitive substances, which are introduced into the body of the film by absorption from aqueous solutions of the sensitizers. J. W. GLASSETT.

**Providing photographic raw film with visible reproducible inscriptions.** I. G. FARBENIND. A.-G. (B.P. 295,383, 27.7.28. Ger., 12.8.27. Addn. to B.P. 287,124; B., 1928, 914).—A colloid, preferably a cellulose derivative, which is insoluble in any reagent used in the subsequent treatment of the film, is admixed with the dye solution or pigment used for the printing. For printing on nitrocellulose film, a 2:1 mixture of cold saturated solution of cerasine-red in glycol acetate and a 5% solution of acetylcellulose in the same solvent may be used. J. W. GLASSETT.

**Photographic and like screens.** T. C. ERWIN (B.P. 315,681, 13.4.28).—Reticulated gelatin screens are prepared by coating a transparent support (glass, celluloid) with a gelatin solution hardened, to render it insoluble in hot water, by treatment, either before or after coating, with chrome alum, formalin, or similar tanning agents. After drying at room temperature the film is treated with a warm acid bath; preferably a bath consisting of 1 c.c. of hydrochloric acid (*d* 1.27) in 1150 c.c. of water is used at 57—60°. J. W. GLASSETT.

**Producing even coatings on transparent plates, films, etc. to serve as colour screens and for other photographic purposes.** F. H. HAUSLEITER (B.P. 316,719, 23.5.28).—The base is coated with a layer of a colloid mixture, such as dichromated gelatin, which is rendered insoluble by the action of light, given a uniform exposure through the rear side of the base, and treated to remove the soluble portion. The even coatings so formed are free from surface skin formation, but this may be produced where necessary by treatment with a tanning vapour, such as bromine, or by the precipitation of a film of lead or copper ferrocyanide on the surface. J. W. GLASSETT.

**Production of screen negatives for producing photomechanical printing surfaces.** H. A. GILL. From AMIRA TRUST (B.P. 316,364, 14.5.28).—A silver halide emulsion on a transparent support is exposed through the rear side behind a screen, and is developed in a hardening developer or treated after development by any known method for hardening the gelatin in proportion to the silver deposit. Without fixation, the negative is washed in hot water and the silver dot image intensified or reduced by the usual methods. High dot formation may be prevented by lowering the penetration of the light by the addition of a suitable dye to the emulsions or by destruction of the surface latent image by means of copper chloride, bromine-iodine tincture, etc. J. W. GLASSETT.

**Colour photography.** R. RUTH (B.P. 317,712, 20.2.28).—A multi-coloured gelatin screen, supplied with a detachable transparent support, is coated with sensitive emulsion. The exposure is made through the support and the screen is hardened at those parts in contact with the light-affected silver halide by the use of a

hardening developer such as pyrocatechol. The image is reversed, using a non-hardening developer, transferred together with the screen to a suitable base, and the original support is then stripped, the unhardened parts of the screen being removed by hot water. J. W. GLASSETT.

**Development and fixing of photographs.** ETABL. E. BELIN (B.P. 299,466, 25.10.28. Fr., 28.10.27).—The developing and fixing solutions and the wash-water are sprayed in turn through atomising jets on to the surface of the sensitive material. The solutions may be used at 50—80° for rapid work, and although this temperature is considerably lowered by the cold emulsion surface, the working temperature obtainable is much higher than that possible by the ordinary bathing methods. J. W. GLASSETT.

**Manufacture of photographic prints.** LUXOR-FILM GES.M.B.H. (B.P. 289,858, 3.5.28. Ger., 4.5.27).—A gelatin film, which has been sensitised by bathing first in a strong dichromate solution (up to 10%) followed while still wet by a short treatment in a weak solution (up to 2%), is exposed behind the negative and then left in a warm atmosphere supersaturated with water vapour and containing a small quantity of volatile organic acid (acetic acid) or carbon dioxide. During this treatment the plate is given a uniform general exposure and later removed, washed with cold water, and dyed. J. W. GLASSETT.

**N-Hydroxyethyl derivatives of [*p*]-aminophenols [photographic developers].** G. REDDELEIN and W. MUELLER, ASSTS. to AGFA ANSCO CORP. (U.S.P. 1,712,716, 14.5.29. Appl., 2.11.27. Ger., 19.11.26).—See B.P. 280,873; B., 1929, 38.

**[Registering devices for] production of [multi-colour] kinematograph film positives.** J. E. THORNTON (B.P. 316,338—9, 25.4.28).

**[Registering devices for] manufacture of two-colour kinematograph positive films.** J. E. THORNTON (B.P. 316,367, 18.5.28).

## XXII.—EXPLOSIVES; MATCHES.

**Explosive properties of "Chloratit 3" containing various amounts of petroleum.** A. HAID and H. SELLE (Z. ges. Schiess- u. Sprengstoffw., 1929, 24, 251—252).—"Chloratit 3" with 8—12% of petroleum is the most sensitive in the fall-hammer test. When "Chloratit 3" contains 3% of woodmeal and 1—2% of petroleum, it is insensitive to friction in the porcelain mortar test, a content of 3—5% of petroleum causes it to crackle, whilst 7—10% causes an explosion. Sensitiveness to initiation is greatest when the petroleum content is 3—5%. The values in the lead-block test increase linearly with the petroleum content until the latter reaches 10%, and then decrease. The maximum detonation velocity (3500 m./sec.) occurs with a content of 7% of petroleum. On storage, a cartridge of "Chloratit 3" with 3% of woodmeal and 9% of petroleum loses 1% in weight at 20° in 5 months. S. BINNING.

**Testing the chemical stability of smokeless powders and explosives by measuring their hydrogen-ion concentrations.** L. METZ (*Z. ges. Schiess- u. Sprengstoffw.*, 1929, 24, 245—250, 291—296, 335—340).—Hansen has proposed to test the stability of smokeless powders by measuring the change in their  $p_H$  when they are heated at 110° for 8—10 hrs. His data were checked by determinations on various types of nitrocelluloses and nitrocellulose powders, and his method was then extended to nitroglycerin powders, and also to explosives such as trinitrotoluene, tetryl, nitroglycerin, dinitroglycol, and gelatine-dynamite. The test was found to have decided advantages in giving in a few hours results that were in agreement with the present tests, which require many hours for performance. The relatively low temperature at which the test is carried out is also in its favour. The apparatus is easy to work, even by relatively unskilled users.

S. BINNING.

**Influence of gel structure on the technology of smokeless powder manufacture.** F. OLSEN (*Coll. Symp. Mon.*, 1928, 6, 253—264).

### XXIII.—SANITATION; WATER PURIFICATION.

**Determination of hydrogen cyanide in fumigation experiments.** C. O. EDDY and E. N. GEDDINGS (*J. Econ. Entomol.*, 1929, 22, 366—378).—Samples of the air are passed through 3.5% sodium hydroxide solution and the cyanide is determined turbidimetrically with silver iodide.

CHEMICAL ABSTRACTS.

**Equipment for the treatment of feed water for modern steam boilers.** J. D. YODER (*Ind. Eng. Chem.*, 1929, 21, 829—834).—Descriptions are given of hot-process lime-soda softeners, sodium phosphate feeding equipment, sulphuric acid feeding equipment in connexion with zeolite softeners to maintain the sulphate ratio, and continuous blow-off apparatus.

C. IRWIN.

**Examples and precepts of water conditioning.** R. E. HALL (*Ind. Eng. Chem.*, 1929, 21, 824—829).—A number of analyses of boiler-feed waters are given and softening methods used are described. For boiler pressure over 200 lb. "direct" treatment with phosphate with or without a partial preliminary lime-soda treatment is preferred. In one case where water polluted with organic matter was used ammonia was eliminated from the steam by treatment of the water with chlorine. Analyses of boiler scales include examples where the chief component was (a) calcium hydroxide, (b) sodium sulphate. If alkalinity falls too low, phosphate in solution ceases to be effective in preventing the formation of calcium sulphate scale. Some superheater deposits contained sulphides. Experiments with unsaturated asphaltic hydrocarbons as anti-foaming material, whilst successful in some cases, did not indicate that its use is generally desirable.

C. IRWIN.

**Zeolite-deconcentrator combination for boiler-water purification.** E. W. SCARRITT (*Ind. Eng. Chem.*, 1929, 21, 821—823).—In the case reported the untreated water contained 320 p.p.m. of carbonate hardness and 45 p.p.m. of permanent hardness. After 14 days,

using zeolite-softened water alone for make-up, the alkalinity had reached 2050 p.p.m. This necessitated excessive blowing down, and the low ratio of sulphate to total alkalinity involved danger of embrittlement. The make-up was therefore altered to 4 pts. of hard water with 1 pt. of softened water, and a "deconcentrator" or settling tank outside the boiler and connected with it for the removal of sludge was fitted. With this arrangement the alkali formed was sufficient to remove the permanent hardness, no scaling took place, the blow-down was reduced from 6.9% to 1.46%, and the alkali content and sulphate:alkali ratio in the boiler water remained satisfactory.

C. IRWIN.

**Purification of tannery effluents with argillaceous colloids.** J. BESSE (*J. Soc. Leather Trades' Chem.*, 1929, 13, 503—507).—Brick clay is agitated with a dilute solution of sodium carbonate (0.2 g./litre) to disperse it, the heavier particles and sand are allowed to settle out, and the milky liquor is decanted. The tannery effluent is acidified with sulphuric acid, mixed with four times its volume of the colloidal solution of clay, and the mixture is agitated; the clay thereby flocculates and carries down the impurities. The dry residue in the supernatant liquor is considerably less than that in the untreated effluent, and the treated liquor does not froth on shaking.

D. WOODROFFE.

**Antiseptic value of chlorine.** HOWARD and others.—See XV.

PATENTS.

**Liquid preparation for sanitary purposes, eliminating the smell, and decomposition of offensive matter.** S. S. PALEY (B.P. 319,113, 24.8.28).—A liquid containing chromic acid (1 pt.), sulphuric acid, *d* 1.84 (1 pt.), water (16 pts.), and as much zinc as will dissolve therein, is claimed.

C. JEPSON.

**Obtaining soaps and oils from sewage.** E. VON SPRINGBORN (B.P. 318,849, 6.6.28).—The vapours produced by heating sewage sludge either alone or in conjunction with combustible filtering material, *e.g.*, coal, coke, peat (*cf.* B.P. 307,582; B., 1929, 418), are deodorised by potassium permanganate, chloride of lime, or charcoal; after passing through a suitable alkali for the production of soaps, the remaining oils are fractionally cooled to separate them into various grades.

C. JEPSON.

**Softening of water and removing iron and manganese therefrom by base-interchange, and preparing the requisite agent therefor.** A. ROSENHEIM (B.P. 286,307, 2.3.28. Ger., 4.3.27).—The water is brought into contact with a metal oxide gel, silica gel, or mixtures thereof, into which have been incorporated alkali oxides, alkali metals, or ammonium ions which enable base-interchange to take place. The incorporation may be accomplished by treatment of the gels with aqueous solutions of (i) caustic alkalis and ammonia, (ii) alkaline reacting alkali-metal or ammonium compounds, (iii) basic reacting substances the cations of which are not those of an alkali metal or ammonium, with subsequent treatment by a solution of an alkali compound, *e.g.*, common salt.

C. JEPSON.