

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

SEPT. 14, 1928.

### I.—GENERAL; PLANT; MACHINERY.

**Value of inert gas as a preventive of dust explosions in grinding equipment.** H. R. BROWN (U.S. Dept. Agric. Tech. Bull. 74, June, 1928, 23 pp.).—Results of experiments in grinding oat hulls on a scale comparable with industrial conditions and in a plant so designed that the atmosphere might be controlled by the admission of carbon dioxide (boiler flue gas) showed that no ignition of dust occurred in the mill when the oxygen content of the atmosphere was below 15%, the ignition agent being an electric arc. Under varying industrial conditions of grinding a 12% oxygen content is considered completely safe. The use of an inert gas, *e.g.*, carbon dioxide, is considered valuable not only in preventing dust explosions wherever an explosion hazard exists, but also for fire-extinguishing purposes, since no injury is caused to plant or materials as is usually experienced with other methods of fire extinction. C. A. KING.

**Method of evaluation of analyses.** I. O. LIESCHE (Z. angew. Chem., 1928, 41, 748—750).—A systematic method is described for the calculation of the composition of a complicated mixture, as regards the actual simple, double, or complex salts present, from the usual analytical data giving the percentages of anions and cations or basic and acidic oxides. Examples of the application of the method to analyses of ultramarine and of a hard water are given. A. DAVIDSON.

**The unimeter, a new [optical] instrument for chemical industry.** L. BLOCH and H. G. FRÜHLING (Chem.-Ztg., 1928, 52, 488—490).—By means of a polarising photometer fixed to a movable arm, the intensity and colour of the reflected or transmitted light from solids, liquids, or solutions are compared with standards, the intensity of the light being proportional to the square of the tangent of the angle of rotation of the photometer head. The instrument may also be adapted for the examination of large or of distant objects, so that the course of a chemical or physical change may be followed and controlled by the variation in the optical behaviour of the materials. F. R. ENNOS.

**Separation of fractions and the size and shape of rectification columns.** K. V. KOSTRIN (Nef. Choz., 1927, 13, 180—187).—The fuel consumption of stills is increased by lengthening the column, but not always by increasing its cross-section. The efficiencies of rectifying columns are compared.

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Changes in construction of modern stills.

S. VUISHETRAVSKI (Azerbeidj. Nef. Choz., 1928, No. 3, 58—69).—Two new Russian stills are described.

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Tube stills. ACKERMAN.—See II.

PATENTS.

**Tunnel kilns.** A. HERMANSEN (B.P. 292,781, 7.7.27).—A tunnel kiln of the duplex type in which two lines of traffic move in opposite directions and in an intermittent manner, with heat exchange transversely, is provided with transverse partitions combined with the trucks, two such partitions and a pillar of a central row of fixed pillars being in line during the intervals of rest of the trucks. B. M. VENABLES.

**Heat-treating apparatus.** C. I. IPSEN, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,675,271, 26.6.28. Appl., 29.12.26).—The material is placed in a number of receptacles which are passed through a long furnace, and the contents of each are dumped as they pass over a discharge opening. B. M. VENABLES.

**Set pans and the like.** I. G. FARBENIND. A.-G. (B.P. 284,281, 17.1.28. Ger., 27.1.27).—A pan is constructed with Perkins heating or cooling tubes cast on the outer surface of, or buried in the middle of, the wall of a cast-iron pan which is enamelled. B. M. VENABLES.

**Grinding mills.** W. F. CARR-HILL (B.P. 293,559, 23.6.27).—A grinding mill of the centrifugal roll type is provided with resilient suspending and driving means for the roll shafts, so that they can move slightly independently of their driving gear, such movement increasing the relative rubbing between the rolls and ring. B. M. VENABLES.

**Grinding mill.** J. MURSCH, Assr. to BUFFALO HAMMER MILL CORP. (U.S.P. 1,675,658, 3.7.28. Appl., 20.6.27).—In a disintegrator provided with a driving boss and pairs of swinging links, every hammer head is arranged with several working faces, has a circular concentric hole, and is keyed positively in any one of a number of positions on a short shaft which is eccentrically and loosely pivoted between a pair of swinging links, so that one desired grinding face is always presented to the material, but not rigidly. B. M. VENABLES.

**Machine for crushing ore, stone, etc.** J. W. ERICSSON (B.P. 293,651, 18.1.28).—The machine resembles a horizontal disc crusher, but the moving disc is reciprocated axially and not gyrated; the reciprocating motion is produced by the eccentric gyration of a toggle bar, and arrangements are made to keep the crushing strains as self-contained as possible. B. M. VENABLES.

**Disintegrating, grinding, reducing to fibres, or mixing moist or dry materials.** E. STERZL

(B.P. 276,356, 19.8.27. Ger., 20.8.26).—In apparatus wherein rings of teeth on a rotating cylinder run between rings of teeth on the interior of the casing, the faces of the teeth are inclined (*e.g.*, at 23° to the axial plane) so as to produce longitudinal movement of the material under treatment, both ways from the inlet if that is at the centre, or in one direction if the inlet is at one end of the apparatus. B. M. VENABLES.

**Crushing apparatus.** E. B. SYMONS (B.P. 293,642, 23.12.27).—Further improvements are claimed in the gyratory cone crusher described in B.P. 243,926 (B., 1926, 111). B. M. VENABLES.

**Apparatus for separating materials of different density.** J. W. HARTLEY (B.P. 293,137, 4.4.27).—In apparatus of the type where a bucket elevator or raff wheel removes lighter material floating on water and an endless conveyor material which sinks, an upward current of water is maintained in front of the raff wheel by circulating pumps or propellers which draw the upward current from behind the raff wheel, enabling material which would otherwise just sink to be caught. Mechanical improvements are described. B. M. VENABLES.

**Apparatus for the continuous drying or distillation of fine granular masses.** TROCKNUNGS-, VERSCHWELUNGS-, & VERGASUNGS-GES.M.B.H., and L. HONIGMANN (B.P. 293,147, 22.4.27).—The granular material passes through a cylindrical chamber which is traversed also by a gaseous heating medium, using as heat-exchanging devices a number of rotating or oscillating perforated metal discs attached to a central driving shaft. When it is desired to collect the volatile products of distillation, the discs do not come into direct contact with the heating gases in the upper part of the container, but are heated by radiation from fixed heating bodies interposed between them. In another form of apparatus the drum itself may rotate and the heat-exchange bodies may be rigidly attached to it. A. B. MANNING.

**Effecting reactions between solid materials and air or gas.** E. HAYWARD (B.P. 292,642, 22.2.27).—An apparatus by which pulverulent, dry material may be continuously treated in a counter-current of gas at raised temperatures and pressures, say 450° and 20 atm., comprises a closed vessel with internal worm or other conveyor, and external heating. The solid is admitted and discharged in regular cycles by means of valves from or to intermediate chambers at the ends, the necessary valves being automatically operated in correct sequence. B. M. VENABLES.

**Apparatus for the production of dispersions of solids or liquids.** W. H. WHATMOUGH (B.P. 293,510, 29.4.27).—The apparatus is specially suitable for breaking down globular, amorphous aggregates produced by mills of the Plauson or Premier type and referred to in B.P. 276,400 (B., 1927, 831), and consists of a high-speed rotor covered with mesh work through which the liquid is forced. B. M. VENABLES.

**Rotary vacuum filters etc.** BRINJES & GOODWIN, LTD., R. STEWART, and C. F. N. SEAMAN (B.P. 292,644, 24.2.27).—A form of rotary drum filter is described, having provision for separate withdrawal of filtrate and washings. B. M. VENABLES.

**Method and apparatus for filtering air.** REED AIR FILTER CO., Assees. of C. P. HEGAN (B.P. 279,102, 14.10.27. U.S., 15.10.26).—The dust is caught on surfaces coated with a viscous liquid such as oil, which when fully charged with dust is renewed, without heating, by a large flush of new liquid, the flushings being filtered and re-used. The flushing is done when the air current is cut off, and automatic switches may be used which operate so that when the air fan or air current stops, the motor for the oil pump starts. B. M. VENABLES.

**Filter.** E. J. SWEETLAND (U.S.P. 1,677,503, 17.7.28. Appl., 22.1.20).—The end portion of a strip of filtering fabric lies flat upon the bottom of a one-piece casing, and the remainder of the strip is folded backwards and forwards upon itself, spacing members being placed between each fold, until the casing is full. The lateral free edges of the fabric are folded within channels on opposite sides of the casing in such a manner that solids are prevented from escaping from the folds without sewing or cutting the fabric. F. G. CLARKE.

**Discharge of liquids from centrifugal separators.** AKTIEBOLAGET SEPARATOR (B.P. 285,088, 9.2.28. Swed., 12.2.27).—In a centrifugal separator for two liquids the heavier liquid is led, prior to discharging, to a point close to the axis and preferably above the outlet for lighter liquid, and the passages are arranged so as to be easily cleaned. B. M. VENABLES.

**Liquid atomisers.** SIEMENS-SCHUCKERTWERKE A.-G., Assees. of SIEMENS-SCHUCKERTWERKE GES.M.B.H. (B.P. 287,105, 27.2.28, and 292,940, 27.3.28. Ger., 15.3. and 27.6.27).—A form of spray especially suitable for humidifying purposes comprising a specially shaped disc rotated at very high speed by an electric motor with vertical shaft. B. M. VENABLES.

**Production of liquid dispersions.** ANODE RUBBER CO., LTD. From P. KLEIN and A. SZEGVÁRI (B.P. 293,071, 1.3.27).—The mixture is subjected to the action of hard, rounded, grinding elements which are kept in motion by a stirring device, and is preferably circulated between the emulsifier and a sedimentary or centrifugal separator in which the coarser unemulsified particles are separated. The size of the grinding elements should lie between 1 mm. and 8 mm. mesh, and two sizes may be used, one being at least double the diameter of the other. The metallic parts of the apparatus may be covered with rubber, and cooling may be applied. B. M. VENABLES.

**Circulating or mixing of liquids etc.** DUNLOP RUBBER CO., LTD., R. F. MCKAY, H. WILLSHAW, W. G. GORHAM, and R. F. LEE (B.P. 293,072, 2.3.27).—A bath of liquid matrix, such as rubber ready for deposition on moulds, is kept in circulation by pumping, the delivery of the pump being upwards and fan-shaped at the centre of the bath, and the flow then along the surface to weirs at the ends. Any coagulated matter is caught in strainers at the weirs and the overflow liquid runs into a sump (where make-up is added) and returns to the pump. The weirs also prevent increase in depth of the bath due to the presence of extra large moulds. B. M. VENABLES.

**Evaporators.** G. & J. WEIR, LTD., and J. SIM (B.P. 292,842, 22.11.27).—The liquid in the evaporator

is subdivided by trays into two or more bodies each having a free evaporating surface, and each heated by steam tubes through which the steam passes in parallel. The level of the liquid in the lowest chamber, which is the bottom of the containing shell, controls by a float valve the feed of liquid, which is wholly to the uppermost chamber, the liquid overflowing downwards from tray to tray.

B. M. VENABLES.

**Continuous treatment [e.g., hydrogenation] of liquids with gases.** H. MIELCK (B.P. 272,913, 14.6.27. Ger., 15.6.26).—A long, horizontal, stationary autoclave is divided by moderately permeable partitions into a number of separate chambers, which may be separately heated or cooled, and all are provided with stirrers preferably fixed on a long common shaft. The permeability of the partitions is sufficiently low to prevent any mixing of liquids in adjacent compartments, while permitting only a slow one-way flow.

B. M. VENABLES.

**Apparatus for rarefying gases.** J. O. BOVING (B.P. 292,758, 31.5.27).—In an apparatus where liquid flows in a siphon tube, a vacuum being produced at the top of the siphon, the delivery branch is arranged on the Venturi principle with high velocity at the top and low at the bottom, thus reducing the height of the necessary liquid column. The throat is also smaller than the inlet conduit, and the gas inlet or aspirator connexion from the vessel to be exhausted is situated in the midst of the stream at or just after the highest point, and is given a stream-line form with a number of small holes.

B. M. VENABLES.

**Gas diffuser.** E. J. SWEETLAND (U.S.P. 1,677,502, 17.7.28. Appl., 19.12.19).—The porous blocks are treated with a preservative which does not obstruct the pores.

F. G. CLARKE.

**Liquefaction apparatus.** R. C. TOLMAN, W. L. DE BAUFRE, J. W. DAVIS, and M. H. ROBERTS, Asss. to S. G. ALLEN (U.S.P. 1,676,225, 3.7.28. Appl., 16.9.22).—The gaseous mixture is pre-cooled in heat exchangers and passes on to a liquefier for the greater part of the undesired constituent of the gas, and to a separating chamber whence the gas passes to a purifier cooled by a separate refrigerating cycle. The liquid from the separator and the purifier is used to cool (by counter-current) the liquefier and heat exchangers.

B. M. VENABLES.

**Production and utilisation of cold.** G. CLAUDE and P. BOUCHEROT (B.P. 278,762, 11.10.27. Fr., 11.10.26).—Cold water is drawn through a conduit, which may be heat-insulated, from the depths of the ocean, and the cold water either taken directly to the place where cold is required or used to cool and dry air in a heat interchanger or spray device. At a depth of 1000 m. the temperature of the sea is about 4° even near the equator.

B. M. VENABLES.

**Refrigerating compounds.** O. HANNACH (B.P. 293,581, 4.8.27).—A mixture, e.g., containing 2 pts. of ammonium chloride and 3 pts. of sodium carbonate, added to 6 pts. of water gives a maximum decrease of temperature (31°).

H. ROYAL-DAWSON.

**Carrying out exothermic catalytic chemical reactions.** L'AIR LIQUIDE SOC. ANON. POUR L'ÉTUDE

ET L'EXPLOIT. DES PROC. G. CLAUDE, Assees. of SOC. CHIM. DE LA GRANDE PAROISSE (AZOTE ET PROD. CHIM.) (B.P. 289,759, 18.7.27. Fr., 30.4.27).—In catalytic reactions such as removal of carbon monoxide from a mixture of nitrogen and hydrogen by combining the first with part of the last, it is usual to provide heat interchange between the catalytic material and the incoming gas. In order to obtain regulation of the heating the stream of gas is divided into two streams, separately regulable either at the inlet or outlet, which are passed over different surfaces for heat interchange.

B. M. VENABLES.

**Absorbing agent.** A. ROSENHEIM (B.P. 275,203, 12.7.27. Ger., 30.7.26).—Natural or artificial zeolites or similar substances (e.g., the reaction products of silicates and aluminates or iron compounds etc.) are treated with acids or acid salts in the liquid or gaseous state at ordinary or higher temperatures up to the b.p. of the liquids concerned, forming a mixture containing a gel of amphoteric metal compound beside the silicic acid gel. [Stat. ref.]

H. ROYAL-DAWSON.

**Colorimeters and analogous instruments.** R. A. LEGENDRE (B.P. 285,848, 21.12.27. Belg., 23.2.27).—The sighting system, which makes use of a Pulfrich prism, permits of two separate sighting lines, each of which passes through a unit consisting of three reservoirs or tanks with transparent walls. A unit may be formed by two right-angled triangular prisms placed with their hypotenuses in juxtaposition and a tank of uniform thickness placed against the longer surface of the side of the rectangular prism. It may also be constructed of two co-axial cylindrical tanks in combination with a third of cylindrical shape but not co-axial.

C. RANKEN.

**Temperature-regulating system.** SELDEN Co., Assees. of C. E. ANDREWS (B.P. 285,499, 18.2.28. U.S., 19.2.27).—See U.S.P. 1,666,251; B., 1928, 391.

**Apparatus for boiling or raising the temperature of water and other liquids.** C. F. HAMMOND and W. SHACKLETON (U.S.P. 1,675,347, 3.7.28. Appl., 4.9.26. U.K., 5.9.25).—See B.P. 265,253; B., 1927, 240.

**Straining or filtering apparatus.** F. C. FULCHER and W. R. BELDAM (U.S.P. 1,673,743, 12.6.28. Appl., 12.6.24. U.K., 22.6.23).—See B.P. 223,627; B., 1924, 1000.

**Refrigerating system.** C. W. VOLLMANN (B.P. 291,856, 7.3.27).

**Refrigerating systems.** COMSTOCK & WESCOTT, INC., L. F. WHITNEY and E. A. WEAVER (B.P. 292,812, 22.9.27).

**Rotary refrigerating apparatus.** H. NIELSEN (B.P. 291,835, 25.2.27).

**Furnace roofs.** E. STRUNK and A. REPPMAN (B.P. 294,026, 12.10.27).

**Machines for breaking coal and other friable material.** B. NORTON (B.P. 294,068, 12.1.28).

**Liquid filters [for fuel oil etc.].** SOC. ANON. DES NOUV. INVENTIONS MÉCANIQUES ET ÉLECTRIQUES (B.P. 278,349, 22.9.27. Fr., 30.9.26).

**Thermostats.** G. G. ROYER (B.P. 293,930, 3.5.27).

Chemical reactions (B.P. 293,138).—See VII. Muffle kiln (U.S.P. 1,677,818).—See VIII. Rotary kiln (B.P. 293,331).—See IX. Filtering apparatus (B.P. 293,534).—See XXIII.

## II.—FUEL; GAS; TAR; MINERAL OILS.

Agglomeration of coals by means of hydrocarbons partially dehydrogenated by sulphur. A. LÉAUTÉ (Compt. rend., 1928, 187, 227—229).—Partial dehydrogenation of the oils used increases their adhesive power, as shown by the increased resistance of the agglomerates to crushing. Briquetting without the addition of pitch is then possible. The effect is less the poorer the specimen in light oils. C. W. GIBBY.

Effects of temperature and pressure on the explosibility of coal dust-air mixtures. H. SANO (Nihon-Kogyokwaishi, 1925, No. 486, 845—852).—The lower limit of inflammability in coal dust-air mixtures descends with rising initial temperature, but is unaffected by change of pressure from 1 to 5 atm., although the ignition temperature at the limit is lowered. Dust clouds incapable of inflaming by ignition can propagate explosions. CHEMICAL ABSTRACTS.

Gas producer operation. V. WINDETT (Proc. Eng. Soc. W. Pa., 1928, 44, 11—34).—The results of a large number of records of the ordinary working of ten Wellman-Seaver-Morgan, Type L, No. 10, mechanical gas producers at a factory of the American Window Glass Co., Jeanette, Pa., are given. The gas is supplied for heating two glass furnaces, each holding 1800 tons of molten glass, and 26 annealing ovens. A curve showing the results from 430 determinations of the heat of combustion of producer gas made from 60,000 tons of coal over 14 months shows a maximum deviation of 0.24%. There is no seasonal effect on the operation of the producers. An inspection of the heat balance indicates loss of heat by radiation is only 4% of the total. The factors chiefly affecting the quantity and quality of the gas are the temperatures of the air blast and of the gas. The results of analyses, based on variations in the temperature of the air blast, show that the gas of highest calorific value is obtained at a saturation temperature of 48.9—54.4°, with a maximum at 51.7° of 174 B.Th.U. net. A tabulation based on gas temperatures shows the most advantageous range is 677—788°. Plottings of the calorific values, or heats of combustion of the gas, and of curves of the principal constituents of the gas shows the existence of a tendency towards an increase in the carbon monoxide, accompanied by a decrease of the carbon dioxide, and also a rise in temperature. The decrease in the hydrogen accounts for the lowering of the calorific value of the gas as the temperature rises. The relationship between the fusing temperature of the ash, the fineness of the coal, and the temperature of the air-blast shows the general desirability of a low percentage of fines and an ash of high fusing point. The total useful heat of gasification is 88.20%, which is the measure of the efficiency of the gas-producer operation. H. S. GARLICK.

Apparatus for determining the ignition point of coke and of technical carbon. W. SWIENTOSLAWSKI and B. ROGA (Przemysl Chem., 1928, 12, 18—31).—

An apparatus for the determination of the ignition point of coke, coal, graphite, wood charcoal, etc. is described. The ignition point is defined as being that temperature at which the given combustible substance begins to react vigorously with pure oxygen, and is shown to have a constant value for each individual substance. R. TRUSZKOWSKI.

Ignition point of wood charcoals. W. SWIENTOSLAWSKI and M. CHORAŻY (Przemysl Chem., 1928, 12, 31—37).—The ignition point of birch charcoal rises proportionately to the temperature of carbonisation previously employed. If a sample of charcoal be heated at a higher temperature than that at which it was carbonised, the new ignition point is identical with that which would have been obtained had the wood been carbonised at the higher temperature. If wood charcoal be heated in presence of oxygen at a temperature close to its ignition point, the latter rises continually with duration of heating. R. TRUSZKOWSKI.

Ignition points of active charcoals. W. SWIENTOSLAWSKI and B. ROGA (Przemysl Chem., 1928, 12, 38—39).—The ignition points of active charcoals bear no relation to their adsorptive power, but are strictly dependent on the temperature of activation. Thus charcoals activated at 450° and at 1000° have ignition points of 169° and 457° respectively. R. TRUSZKOWSKI.

Thermal disintegration of gaseous hydrocarbons. S. MANTEL (Przemysl Chem., 1928, 12, 333—342).—The hydrocarbons contained in illuminating gas and natural gas are resolved into their elements at 1600°. The resulting gas consists of hydrogen contaminated with 0.7% of methane. R. TRUSZKOWSKI.

Occurrence and detection of hydrogen in mine air. HEYER (Z. ges. Schiess- u. Sprengstoffw., 1928, 23, 78—79).—Hydrogen is frequently a constituent of mine atmospheres, although seldom in amount sufficient to cause accidents. Small amounts of it may be detected by the fact that it reduces sodium molybdate in weakly acid solution with formation of a blue coloration. To prepare the reagent 1 g. of molybdic anhydride is dissolved in caustic soda solution, 0.1 g. of colloidal palladium mixed with sodium protalbinat is added, the solution is diluted to 200 c.c., and acidified with dilute sulphuric acid till the palladium coagulates. The test may be rendered quantitative by comparison with gases of known hydrogen content. S. BINNING.

Wood-gas-producer tar. I. J. POSTOVSKI, N. A. APOLLOV, and B. P. LUGOVKIN (Ann. Inst. Polyt. Ural, 1928, 6, 241—244).—A sample of tar from a gas producer using various species of pine wood as fuel showed a mean moisture content of 50%, 14.72% of neutral products, 0.1% of bases, and 31.8% of phenols. The components separated were *p*-cresol, *p*-ethylphenol, a dimethoxy-*p*-xylene (?), an unidentified phenolic compound, an unsaturated hydrocarbon (C<sub>8</sub>H<sub>6</sub>)<sub>n</sub> (? naphthylfulvene), and 40% of resinified products insoluble in ether. The formation of the last, and the absence from the tar of the products usually found in wood tar, are ascribed to the high temperature prevailing in the producer. T. H. POPE.

**Kraemer-Sarnow method [for determining the softening point of pitch] and mercury poisoning.** H. MALLISON (*Z. angew. Chem.*, 1928, **41**, 839—840).—The danger of chronic poisoning by prolonged contact with mercury has been pointed out by Stock and others; it is therefore suggested that the mercury method of determining the softening point of pitch should be universally replaced by the American "ring and ball" method.

A. R. POWELL.

**Crude oils from the district of the Kruimskaya settlement (Kuban, Black Sea district).** A. O. YURKOV (*Neft. Choz.*, 1928, **14**, 217—219).—The wells are rich in gas, but the portion having b.p. < 100° is low; the kerosene fraction is satisfactory in yield but has high *d*. Kruimskaya oil contains less aromatic substances than Maikop crude oil.

CHEMICAL ABSTRACTS.

**Lubricating oils from Surakhani crude oil.** A. N. SACHANOV (*Azerbeid. Neft. Choz.*, 1928, No. 1, 50—52).—The light fractions consist of naphthenes and the heavy of paraffins; the fraction (mazout) available for lubricating oil is 54.2%. On distillation in a vacuum 72% was recovered as oil. Data for the cylinder stock obtained from the residue, and for the above fractions, are recorded. CHEMICAL ABSTRACTS.

**Pyro-benzene from mazout from Surakhani paraffin oils and from residue from heavy gas tars.** N. NOVIKOV-VAKULENKO (*Azerbeid. Neft. Choz.*, 1928, No. 1, 55—59).—Atomised heavy residues, blown against heated fire-brick, left at 617—774°; 33.5—54% of gas was produced, and the residual tar was distilled. Fractional yields and densities are recorded for solar oil, mazouts, and heavy gas tar. The gases from solar oil and paraffin mazout contained, respectively, CO<sub>2</sub> 0.7—1.3, 0.8; CO 0.7—0.5, 1.0; O<sub>2</sub> 1.1—1.6, 1.8; H<sub>2</sub> 9.2—25.4, 16.8; CH<sub>4</sub> 46.7—36.5, 36.1; heavy hydrocarbons, 38.8—27.6, 20.8; N<sub>2</sub> 2.8—7.1, 22.7%.

CHEMICAL ABSTRACTS.

**Cracking of mazouts from heavy crude oils.** A. N. SACHANOV and M. D. TILTSCHIEV (*Neft. Choz.*, 1928, **14**, 328—353).—On cracking at 425° and 10—60 atm., the speed at which gasoline and kerosene are formed is practically the same for all mazouts and heavy crude oils. At 425° 8—10 and 18—20% of gasoline and 12—15 and 18—22% of kerosene are formed in 30 and 60 min., respectively; the rate of cracking is doubled every 10°. Various crude oils were examined. The relation between the yields of gasoline and coke depends on the oil used, and is independent of the temperature.

CHEMICAL ABSTRACTS.

**Liquefaction of petroleum asphalt by Bergius' method.** K. SMOLEŃSKI and W. BADZYŃSKI (*Przemysł Chem.*, 1928, **12**, 117—136).—Petroleum asphalt when heated at 400° for 12 hrs. with hydrogen at above 100 atm. yields 60% by weight of liquid hydrocarbons, similar to those obtained by the distillation of Galician light petroleum, and of which 32% boils below 150° and 36.5% from 150° to 300°. The solid residue (25—30%) contains 87% C and 4% H, and is a good substitute for coke. About 15% of the original weight of asphalt is evolved as gaseous products containing about 40% CH<sub>4</sub> and 40% H<sub>2</sub>. Somewhat lower yields of liquid

hydrocarbons are obtained by the substitution of water-gas for hydrogen in the above reaction.

R. TRUSZKOWSKI.

**Extraction of high-quality cylinder oils from lubricating oil residue.** B. E. VORONOV (*Neft. Choz.*, 1928, **14**, 213—216).—Lubricating oil residues are mixed with petroleum (15%) and treated with 92.5% and fuming sulphuric acid, the petroleum then being removed, the residue treated with fuller's earth, and mixed with petroleum (15%) and naphthenic acids (0.5%). After heating at 90° and removal of water, sulphuric acid is added, the mixture agitated with air, and the acid sludge removed after 2 hrs. The treatment is repeated, sodium hydroxide solution and water being finally employed. The yield, after treatment with fuller's earth, was 60% of an oil having flash point (Brenken) 305—310°, viscosity (Engler, 100°) 6—6.5, carbon (Konradson) 2.2—2.4%, colour dark green. (Russ. Pat. 16,151 of 1927).

CHEMICAL ABSTRACTS.

**Comparison of foreign and Soviet lubricating oils.** G. ANDRÉEV (*Neft. Choz.*, 1928, **14**, 354—359).—The Russian spindle oils have the lower flash point. The cold test (−20°) and the resin content are favourable.

CHEMICAL ABSTRACTS.

**Treatment of bottoms from the lubricating oil fraction of Emba crude oil.** N. TSCHERNOSHUKOV (*Neft. Choz.*, 1927, **13**, 188).—By treatment with sulphuric acid (2 pts.) and naphthenic acids (1 pt.), 45% of oil was separated.

CHEMICAL ABSTRACTS.

**Acid sludge from oil fractions from Emba crude oil.** S. OBYRADCHIKOV (*Neft. Choz.*, 1928, **14**, 360—362).—The tar from the washed sludge which had been heated at 130—150°, when treated with air at 300° gave asphalts having m.p. 80°, 60°, and 30°. The yields are recorded.

CHEMICAL ABSTRACTS.

**Improving viscosines (cylinder stocks).** L. GUKHMAN (*Azerbeid. Neft. Choz.*, 1928, No. 2, 61—63).—"Vapours" (cylinder stocks from Emba crude oils) contain 2.1—3.1% C; "viscosines" (from Balkhanui crude oil) contain 3.0—7.0% C, and are washed with steam, treatment with alkali hydroxide being then unnecessary. The production of heavy viscosines is described, the bottoms being mixed with solar oil and treated with sulphuric acid at 20—25°.

CHEMICAL ABSTRACTS.

**Distillation of oil shales from the liquid phase.** E. VON PEZOLD (*Chem.-Ztg.*, 1928, **52**, 541—542, 562—564).—If oil shales are distilled with certain high-boiling solvents, *e.g.*, middle or heavy shale oils boiling at 200—300°, the yield of light oils of b.p. below 200° is considerably greater than that obtained by heating the shale and oil separately either at 400° or under pressure. At the same time there is a decrease in the total oil distillate and an increase in the amount of coke and water formed. By continued distillation of the higher-boiling oil with fresh quantities of shale the yield of light oil first rises to a maximum and then falls to an approximately constant figure. No increase in the yield of light oil is obtained by passing the shale oil vapour over shale coke.

F. R. ENNOS.

**Gasoline stripping plant.** V. DENISEVICH (*Azerbeid. Neft. Choz.*, 1928, No. 1, 59—64).—The plant

is described and figured; the heat recovery is 75% and the fuel consumption 0.7%.

## CHEMICAL ABSTRACTS.

**Tube still for distillation of crude oil.** I. N. ACKERMAN (*Neft. Choz.*, 1928, 14, 193—212).—Calculations for the construction of tube stills are given.

## CHEMICAL ABSTRACTS.

**Rapid corrosion test for gasoline.** H. P. RUE (*U.S. Bureau Mines Rep. Invest.*, 1928, No. 2862, 5 pp.).—The sample (100 c.c.) is shaken in a 4 oz. bottle for 2 min. with 1 c.c. of mercury. The gasoline is decanted and immediately filtered, the paper dried, and the intensity of the black colour indicates the corrosiveness of the gasoline.

## CHEMICAL ABSTRACTS.

**"Formolite" reaction for testing mineral oils.** M. BORODULIN (*Neft. Choz.*, 1928, 14, 363—364).—The precipitate dissolves when the ammonium sulphate is washed out; the use of methylal is recommended.

## CHEMICAL ABSTRACTS.

**Acid asphalts.** S. GASIOROWSKI (*Przemysl Chem.*, 1928, 12, 90—99).—Acid asphalts, obtained as by-products of the refining of machine oils from Galician petroleum with sulphuric acid, when extracted with water to remove free sulphuric acid, yield a very stable emulsion of water in asphalt which loses water at 110°, giving a sticky, plastic mass with a mineral ash content of 0.1%. This product has acid value 8.7, saponif. value 16.6, and contains 2% of free acids, 9.6% of asphaltenes, 15% of resins, and 65% of unchanged oil. The water-soluble products of hydrolysis consist of sulphonic and asphaltogenic acids. When acid asphalt is heated at 220—230° and subjected to a current of air it becomes progressively harder and more brittle, the asphaltene content rises from 9.6 to 30.3%, the mean mol. wt. from 476 to 790, and the free oil content falls to 45.8%, the addition of free sulphur intensifying this process. The reaction of asphaltisation of mineral oils appears to be one of polymerisation associated with oxidation and formation of asphaltenes.

## R. TRUSZKOWSKI.

**Influence of lead tetraethyl on the combustion of mixtures of air and hydrocarbons.** R. DUCHÉNE (*Compt. rend.*, 1928, 187, 200—201).—The effect of 5% of lead tetraethyl on the explosion of mixtures of hexane and air has been studied at pressures sufficiently low to prevent condensation by adiabatic compression. It is concluded that the action of the lead tetraethyl occurs in the gaseous phase, and not on the surface of drops of condensed liquid.

## C. W. GIBBY.

**Stills.** VUSHETRAVSKI. **Rectification columns.** KOSTRIN.—See I. **Argan wood.** CABRAL.—See V. **Sulphur from gas.** CUNDALL.—See VII. **Ultra-marine.** KLIMOV and LANIN.—See XIII.

## PATENTS.

**Treatment of carbonaceous and other materials.** F. FRANK, Assr. to W. W. KEMP (U.S.P. 1,677,757—8, 17.7.28. Appl., [A] 1.12.20, [B] 2.1.23).—(A) Chemical reaction between one constituent of a heat-dissociable carbonaceous material and another present in excess is effected by the direct action of a flame produced by burning a previously formed mixture of uniform com-

position in an air-tight chamber. (B) The flame is produced by burning a uniform mixture of combustible material, a supporter of combustion, and an excess of a reducing agent in an air-tight chamber.

## H. S. GARLICK.

**Reducing materials by heat. Producing combustible gas.** W. W. KEMP (U.S.P. 1,677,784—5, 17.7.28. Appl., 7.11.21).—(A) Carbonaceous material is passed through a heated retort, from which air is excluded, in which it comes into direct contact with a flame from the burning of a uniform mixture of a combustible material with a minimum quantity of a supporter of combustion. (B) Gas-producing material is charged into a gas generator from which air is excluded, and heated by a flame produced by the combustion of premixed gas and air in which the proportion of oxygen is sufficient only to effect combustion of the gas mixture.

## H. S. GARLICK.

**Treatment of coal, peat, lignite, shale, etc. with alkaline solvents.** T. D. KELLY (B.P. 293,578, 29.7.27).—The material is treated with a hot alkaline solution of sodium, potassium, or ammonium oleate and then with aluminium sulphate. The solids are separated from the liquid and, suitably prepared, can be used as fuel, whilst from the liquid can be isolated fuel oil or dyes, or it can be used as a waterproof dressing.

## H. S. GARLICK.

**Binding materials for agglomerating finely-divided fuels or other pulverulent matter. Briquetting finely-divided coal, fuel, or other pulverulent materials.** O. REYNARD and F. F. TAPPING (B.P. 293,135—6, 4.4.27).—(A) A binding agent is made by neutralising concentrated sulphite-cellulose waste liquor with alkali, adding carbonaceous material to form a paste or, alternatively, emulsifying with a petroleum or tar oil, and incorporating the product with molten bituminous material at a temperature above 100°, and under pressure. (B) The finely-divided material is mixed with 3—6% of a bituminous binder, preferably one melting at or above 120°, and with 1—4% of a water-in-oil emulsion. The latter may contain 1 pt. of fuel oil and 1—6 pts. of an aqueous solution of a suitable emulsifying agent, e.g., soda ash, sodium caseinate, sulphite-cellulose waste liquor, etc.

## A. B. MANNING.

**Apparatus for carbonising pulverised fuel.** J. N. BAILEY, and METROPOLITAN-VICKERS ELECTRICAL Co., LTD. (B.P. 293,106, 29.3.27).—The powdered fuel is introduced into a retort of inverted pear shape, having a ring of tangential gas inlets around the wall at the part of greatest diameter. The fuel is supplied from a hopper at the top of the retort to a central distributing nozzle from which it is sprayed upwards and tangentially by the aid of compressed gas. Hot gases injected through the gas inlets whirl the fuel in a vortex within the retort and at the same time carbonise it. The coked fuel and the volatile products escape through an axial outlet at the bottom of the retort, and pass to a separator.

## A. B. MANNING.

**Continuous carbonising furnace.** W. M. HEBURN, Assr. to SURFACE COMBUSTION Co. (U.S.P. 1,677,136, 17.7.28. Appl., 15.2.26).—The gases from the combustion chamber pass through ports to flues surrounding

the permeable walls of an open-top heating chamber, placed above the combustion chamber. Hot gases are also passed around the muffle, the floor and walls of which are continuous with those of the heating chamber. Spent gases leave the furnace at a point remote from the muffle.

F. G. CLARKE.

**Gas producers.** K. A. and E. H. WIDEGREN (B.P. 280,912, 14.11.27. Swed., 22.11.26).—A down-draught gas producer, comprising an upper store chamber for the fuel and a lower, preferably rectangular, combustion chamber, has all the air inlets arranged at the same horizontal level along the upper part of each of the longer sides of the combustion chamber. The distance between these two opposing groups of air inlets is small enough to ensure a practically uniform reaction across the chamber. The fire-box may be divided into two or more such chambers, having a common store chamber and a common ash-pit. The store chamber may be made wider than the combustion chamber, so that the sides of the former pass down by and provide double walls for the latter.

A. B. MANNING.

**Production of gases.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 293,413, 2.4.27. Addn. to B.P. 214,544; B., 1924, 549).—Gas is generated in a producer having a grate provided with a number of small holes through which a stream of air or steam is blown at such a rate that it prevents the finely-divided solid fuel used from falling through. The gasifying agent may in some cases be admitted through the centre of the grate only, so that the fuel bed assumes a conical form and fresh material is fed continuously to the reaction zone.

R. H. GRIFFITH.

**Treatment of gases for removal of hydrogen sulphide and carbon dioxide.** M. AURIG and G. BRÜCKLMAYR (B.P. 293,592, 24.8.27).—Gases are treated with an alkaline lye in a cyclic system in which the used lye is regenerated continuously by treatment with an iron compound and with lime.

H. S. GARLICK.

**Gas purification.** F. W. SPERR, JUN., Assr. to KOPPERS Co. (U.S.P. 1,677,304, 17.7.28. Appl., 31.12.26).—Liquids which have been used for freeing gases from acidic impurities are exposed to the action of air in the presence of hemicellulose.

H. ROYAL-DAWSON.

**Apparatus for detecting, indicating, and recording the presence of inflammable vapours or gases.** H. T. RINGROSE (B.P. 293,125, 1.4.27).—A vessel having porous walls, which are, however, capable of maintaining a gas pressure for a substantial time, is so arranged that the gas under test can pass therein by diffusion. Any inflammable constituents of the gas are burnt within the vessel by means of a heated platinum filament, and the diminution in pressure so produced operates a manometer and gives a measure of the percentage of inflammable gases present. In a second form of apparatus the change in pressure, moving a collapsible diaphragm, completes a circuit which lights a lamp when the percentage of inflammable gas reaches a predetermined value.

A. B. MANNING.

**Tar and a process of obtaining the same.** URBANA COKE CORP. (B.P. 277,955, 8.9.27. U.S., 21.9.26).

—Between 650° and 850° carbonisation of coal produces a tar which is different from any low-temperature or gas-works tar, and is rich in valuable tar acids. A plant is described in which the coal is first heated to a temperature just below that at which it becomes viscous; it is then carried rapidly through the pasty stage to the final temperature, being treated in a vertical metal retort. The condition of the coal produced in this way enables tar of specific characteristics to be formed.

R. H. GRIFFITH.

**Purification of benzol fractions.** C. STILL (B.P. 288,591, 31.8.27. Ger., 13.4.27).—The deleterious and difficultly removable sulphur compounds present in benzol are stated to be formed by oxidation of the hydrogen sulphide present in the freshly produced material. The removal of this impurity from the crude benzol is effected immediately after production by a continuous process involving an aqueous alkali wash.

C. O. HARVEY.

**Conversion of hydrocarbons of high b.p. into compounds of lower b.p.** I. G. FARBENIND. A.-G. (B.P. 268,796, 31.3.27. Ger., 1.4.26).—In the production of low-boiling hydrocarbons from tars, mineral oils, or bitumens, the losses due to carbon deposition are largely avoided by using only cobalt, chromium, nickel, manganese, tungsten, or molybdenum in construction of the apparatus. These metals exert a catalytic effect on the reaction, and may be employed further in the form of internal heating units; additional catalysts may be distributed throughout the oil or be used as granules. The raw material is treated in the liquid phase.

R. H. GRIFFITH.

**Cracking oils.** C. L. PARMELEE and E. W. ISOM, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,675,575, 3.7.28. Appl., 13.8.23).—The ordinary tubular-still cracking process is modified by introducing a heated solvent oil into the partially cracked oil at one or more intermediate stages during its passage through the coil.

C. O. HARVEY.

**Cracking oils.** G. L. PRICHARD, Assr. to GULF REFINING Co. (U.S.P. 1,676,207, 3.7.28. Appl., 22.6.15).—The oil is cracked under pressure in a vessel provided with a mechanical stirring device, the drive of which passes through a packing box enclosed in a chamber through which a liquid is circulated.

C. O. HARVEY.

**Cracking of hydrocarbon oils.** E. C. HERTHEL and T. DE C. TIEFF, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,677,772—6, 17.7.28. Appl., [A—C] 22.6.26, [D] 12.8.27, [E] 27.10.27).—(A) The oil is cracked under pressure by passing it through a heating zone and a primary vaporising chamber, the residue then passing to an externally unheated secondary vaporising chamber, wherein the pressure is less than in the still and first chamber. The vapours from this last chamber are condensed and recirculated through the heating zone before mixing with a fresh supply of the oil to be cracked. (B) The vapours are refluxed in contact with a down-flowing stream of untreated oil. (C) The fractionation of the vapours from the secondary vaporisation chamber is controlled by supplying suitable fractions thereto. (D) A process similar in principle to (A). (E) The reflux

condensate from the primary vaporising chamber is passed through an independent pressure-cracking plant.

C. O. HARVEY.

**Manufacture of hydrocarbons, especially liquid hydrocarbons.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 293,185, 9.6.27).—Oxides of carbon are reduced by hydrogen, or gases rich in hydrogen, in the presence of contact masses containing, in addition to metallic copper, silver, gold, or zinc, elements of the 8th periodic group, and, if desired, small quantities of other elements or compounds. The process may be carried out under ordinary or higher pressures, and at elevated temperatures, in the absence of carriers of low heat conductivity.

A. B. MANNING.

**Production of liquid hydrocarbons.** I. G. FARBENIND. A.-G. (B.P. 269,521, 4.4.27. Ger., 14.4.26).—Hydrocarbons are produced from mixtures of carbon monoxide and hydrogen under pressures of 50 atm. or less in the presence of catalysts consisting of metallic sulphides. The gases are not allowed to come into contact with surfaces containing iron, but the presence of sulphur compounds does not impair the activity of the catalyst.

R. H. GRIFFITH.

**Manufacture of liquid products by destructive hydrogenation of coal, tars, mineral oils, etc.** I. G. FARBENIND. A.-G. (B.P. 273,712, 27.6.27. Ger., 29.6.26).—High yields of low-boiling hydrocarbons are obtained from coal, tars, etc. by consecutive catalytic cracking and hydrogenation in the solid, liquid, or vapour phase. The cracking catalyst consists of elements of groups 3 and 4 of the periodic system, copper-iron mixtures or compounds, active charcoal or silica, and other substances. The hydrogenating catalysts are cobalt, molybdenum, tungsten, and their compounds. The process consists in treatment with hydrogen and the catalyst under pressure at 300–700°, followed by the use of a different catalyst under similar conditions; it may be carried out with a large excess of hydrogen, so that the product has only a low partial pressure.

R. H. GRIFFITH.

**Coking hydrocarbon oils.** H. WADE. From STANDARD OIL Co. (B.P. 293,231, 9.9.27).—The oil is supplied in a thin layer to a surface made of a refractory alloy containing nickel or chromium, *e.g.*, monel metal, iron-chromium alloys, etc., and heated to a coking temperature, *e.g.*, 500°. The coke residue is removed mechanically by suitable scraping means. An apparatus for carrying out the process continuously is described.

A. B. MANNING.

**Treatment of petroleum.** W. W. GARY, Assr. to C. O. MIDDLETON (U.S.P. 1,677,440, 17.7.28. Appl., 7.6.26).—Dry, gaseous sulphuric anhydride is passed through the petroleum.

C. O. HARVEY.

**Refining petroleum fractions.** F. C. AXTELL, Assr. to AXTELL RESEARCH LABORATORIES, INC. (U.S.P. 1,677,425, 17.7.28. Appl., 24.9.25).—After washings with acid and alkali, the fraction is agitated with a solution containing Caro's acid, and then washed successively with concentrated sulphuric acid, water, alkali, and again with water.

F. G. CLARKE.

**Oil still.** E. W. ISOM and A. C. VOBACH, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,676,204, 3.7.28. Appl.,

12.2.27).—The oil is circulated under pressure from a cylindrical container through a tubular still heated by flue gases from a furnace, and thence back to the container. The tubes of the still are provided with stationary solid cores, formed from twisted rods, which thus provide a continuous annular oil passage through the tubes.

C. O. HARVEY.

**Process of treating methane.** G. OLIVIER, Assr. to Soc. ANON. LE PÉTROLE SYNTHÉTIQUE (U.S.P. 1,677,363, 17.7.28. Appl., 3.6.26. Fr., 3.2.26).—Methane is converted into ethylene and ethylenic compounds by heating in a thin stream to 950° max., subjecting to a vacuum, and suddenly cooling to atmospheric temperature.

H. S. GARLICK.

**Production of gaseous hydrocarbons from gas mixtures containing hydrogen and oxides of carbon.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 293,572, 13.7.27).—Gaseous olefines and paraffins are obtained by interaction of hydrogen and oxides of carbon in the presence of catalysts at elevated pressures and temperatures. The catalyst consists of iron or cobalt together with a noble metal or with a difficultly-reducible oxide of a metal, particularly one of the 6th periodic group. The gas mixture is circulated at a substantially lower velocity than that required for the production of liquid hydrocarbons.

R. H. GRIFFITH.

**Polymerisation of ethylene, propylene, and butylene.** F. HOFMANN and M. OTTO (B.P. 293,487, 8.4.27).—Ethylene, propylene, and butylene, either as individuals, mixtures, or in solution, are treated under pressure with boron fluoride or its double compounds or solutions, and in some cases with the addition of finely-divided metals. Good yields of liquid hydrocarbons are produced.

R. H. GRIFFITH.

**Pulverised fuel furnaces.** WITKOWITZER BERGBAU- U. EISENHÜTTEN-GEWERKSCHAFT, and C. SALAT (B.P. 285,042, 9.2.28. Ger., 9.2.27).

[Apparatus for] burning pulverised solid fuel or atomised liquid fuel. F. L. DUFFIELD (B.P. 293,330, 31.1.27).

**Transportation of coke from vertical chamber ovens or from vertical retorts.** C. M. CROFT and R. H. BUXTON (B.P. 293,916, 21.4.27).

**Apparatus for carbonisation.** C. B. WINZER (U.S.P. 1,678,687, 31.7.28. Appl., 21.12.25. U.K., 14.1.25).—See B.P. 250,661; B., 1926, 523.

**Recovering light hydrocarbons from carbonaceous material.** M. J. TRUMBLE (U.S.P. 1,676,675, 10.7.28. Appl., 31.7.22).—See B.P. 221,052; B., 1924, 1004.

**Refining oils and waxes.** W. A. PATRICK and E. B. MILLER, Assrs. to SILICA GEL CORP. (U.S.P. 1,678,298, 24.7.28. Appl., 16.3.22).—See B.P. 195,055; B., 1924, 1005.

**Apparatus for continuous distillation etc.** (B.P. 293,147).—See I.

### III.—ORGANIC INTERMEDIATES.

**Manufacture of vanillin.** A. WAGNER (Chem.-Ztg., 1928, 52, 525–526, 542–543).—A mixture of oil of cloves and potassium hydroxide solution is raised to



b.p. with constant stirring, the heating being continued for a further 37 min. with careful regulation of the temperature to a maximum of 232°. After cooling, the mass is dissolved in water, extracted with benzene after careful acidification, and the benzene recovered. The isoeugenol so obtained is acetylated and afterwards oxidised with sodium dichromate, sulphanilic acid, and sulphuric acid at 67–68°, and extracted with benzene. The extracts from several operations, after removal of 80% of the benzene, are treated with the calculated amount of sodium bisulphite solution at 22°; the bisulphite compound is separated, decomposed with sulphuric acid, and the impure vanillin is centrifuged, distilled *in vacuo*, and crystallised from water containing carbon dioxide. Suitable apparatus is described.

F. R. ENNOS.

**p-Cymene.** WHEELER and BOST.—See IV.

## PATENTS.

**Preparation of polymerised vinyl alcohol and its derivatives.** W. O. HERRMANN and W. HAEHNEL, Assrs. to CONSORT. F. ELEKTROCHEM. IND. (U.S.P. 1,672,156, 5.6.28. Appl., 17.7.25).—A polymerised vinyl ester (acetate, propionate) is hydrolysed with alcoholic alkali to form a polymeride of vinyl alcohol, from which various polymeric esters may be obtained by the usual methods; the butyrate is a rubber-like product.

C. HOLLINS.

**Production of hexamethylenetetramine.** SCHIEFERWERKE AUSDAUER A.-G. (B.P. 286,730, 9.3.28. Ger., 10.3.27).—A solution of ammonium chloride and sodium hydrogen carbonate in contact with the latter substance in solid form (Solvay liquors are suitable) is treated with formaldehyde solution.

C. HOLLINS.

**Manufacture of carbazole-2-carboxylic acid.** H. GRIMMEL, Assr. to I. G. FARBENIND. A.-G. (G.P. 442,609, 9.10.24).—Carbazole and potassium hydroxide are stirred in an iron vessel first at 250°, then in a stream of dry carbon dioxide at 285–300°. The products are carbazole-2-carboxylic acid, m.p. 320° (ethyl ester, m.p. 184°), and a little of the more soluble 4-carboxylic acid. Below 270° the latter is the main product; above 300° a dicarboxylic acid is formed.

C. HOLLINS.

**Manufacture of hydroaromatic dicarboxylic acids.** J. D. RIEDEL A.-G. (B.P. 273,244, 28.2.27. Ger., 22.6.26. Addn. to B.P. 265,959; B., 1928, 117).—The process of the prior patent is extended to the oxidation of alicyclic alcohols and ketones, such as tetrahydro- and decahydro-naphthols, hydrindone, etc., with production of hydroaromatic dicarboxylic acids, e.g., trans-cyclohexylene-1:2-diacetic acid, m.p. 151°, the cis-isomeride, m.p. 164°, and  $\beta$ -2-carboxycyclohexylpropionic acid, from decahydro- $\beta$ -naphthol.

C. HOLLINS.

**Manufacture of sulphonic acids [wetting-out agents].** R. SAJITZ and E. POTT (CHEM. FABR. POTT & Co.) (B.P. 263,873, 31.12.26).—Wetting-out agents are obtained by condensing naphthalene or sulphonated naphthalene with aliphatic or cyclic olefines, sulphonic groups being introduced if not already present. Naphthalene- $\alpha$ - and - $\beta$ -sulphonic acids are heated, e.g., in concentrated sulphuric acid containing propylene and higher olefines below 100°; or naphthalene is sul-

phonated and the crude reaction product is treated at 85–90° with a mixture of tetrahydrobenzene and sulphuric acid.

C. HOLLINS.

**Preparation of  $\beta$ -hydroxyethylnaphthylamines and their substitution products.** I. G. FARBENIND. A.-G., Asses. of W. DUISBERG, W. HENTRICH, and L. ZEH (G.P. 442,310, 18.11.24).—Naphthols or naphthylamines are boiled with  $\beta$ -aminoethyl alcohol and aqueous bisulphite as in the usual Bucherer reaction. The following products are described:  $\beta$ -hydroxyethyl- $\beta$ -naphthylamine, m.p. 51° (and hydrochloride); 2- $\beta$ -hydroxyethylnaphthylamine-7-sulphonic acid; *N*- $\beta$ -hydroxyethyl- $\gamma$ -acid; 1- $\beta$ -hydroxyethylnaphthylamine-4-sulphonic acid; *N*-methyl-*N*- $\beta$ -hydroxyethyl-2-naphthylamine-6-sulphonic acid; and 2- $\beta$ -hydroxyethylamino-8-methoxynaphthalene-6-sulphonic acid (and nitroso-compound).

C. HOLLINS.

**Manufacture of condensation products [naphthindanones] from aromatic hydrocarbons or derivatives thereof.** I. G. FARBENIND. A.-G. (B.P. 267,940, 16.3.27. Ger., 16.3.26).—Aromatic hydrocarbons (particularly  $\alpha$ -methyl-naphthalene) are condensed with a  $\beta$ -halogenated aliphatic acid halide in presence of aluminium chloride, and the products are cyclised by first removing hydrogen halide to give unsaturated ketones and then treating them with aluminium chloride or sulphuric acid. The process may be carried out in one or several stages. 1-Methyl-4-naphthyl  $\beta$ -chloroethyl ketone, m.p. 60°, from  $\alpha$ -methyl-naphthalene and  $\beta$ -chloropropionyl chloride, is converted by concentrated sulphuric acid at 100° into 5-methyl- $\alpha$ -naphthindan-9-one. Distillation of 1-methyl-4-naphthyl- $\beta$ -chloropropyl ketone, m.p. 44–45°, yields the corresponding propenyl ketone, b.p. 204–206°/12 mm., which is similarly cyclised to 5:7-dimethyl- $\alpha$ -naphthindan-9-one, m.p. 67–68°, also obtainable by the action of sulphuric acid on the chloroketone.

C. HOLLINS.

**Manufacture of 1-anthraquinonyl ketones.** I. G. FARBENIND. A.-G. (B.P. 271,884, 25.5.27. Ger., 25.5.26, and Addn. 272,225, 2.6.27. Ger., 7.6.26).—(A) A 3-aryl- or (B) a 3-arylbenzanthrone is oxidised with chromic-acetic acid. Examples are: phenyl 1-anthraquinonyl ketone, m.p. 229°, from 3-phenylbenzanthrone; 1:1'-dianthraquinonylglyoxal, R·CO·CO·R, m.p. 330–331°, from 3:3'-dibenzanthronyl;  $\alpha$ -phenyl- $\beta$ -anthraquinonyl-glyoxal, m.p. 181°, from 3-hydroxy-2-phenylbenzanthrone (less drastic oxidation gives in this case a substance, m.p. 237°) or from phenyl 3-benzanthronyl ketone (3-benzoylbenzanthrone).

C. HOLLINS.

**Preparation of *N*-nitrosoamines of the anthraquinone series.** I. G. FARBENIND. A.-G., Asses. of A. JOB and H. TESCHE (G.P. 442,312, 25.10.24).—The *N*-nitroso-derivatives of 1- and 2-methylamino-, 4-bromo-1-methylamino-, 1-*p*-toluidino-, 1- and 2-benzylamino-, and 2-anilino-anthraquinones are obtained by action of sodium nitrite or amyl nitrite on the appropriate amines in acetic acid solution. (Cf. G.P. 443,126, following.)

C. HOLLINS.

**Preparation of nitro-compounds of arylamino-anthraquinones, their derivatives and substitution products.** I. G. FARBENIND. A.-G., Asses. of

H. TESCHE and A. JOB (G.P. 443,126, 7.11.24).—The *N*-nitroso-derivatives of arylaminoanthraquinones (preceding abstract) are converted into nitro-compounds when heated in acetic acid, oxidation and migration of the nitroso-group to the aryl nucleus taking place. The same result is obtained by nitrosating the arylaminoanthraquinones in boiling acetic acid. Nitroarylaminanthraquinones are obtained by these methods from 1- and 2-anilino-, 1- and 2-*p*-toluidino-, 1:4- and 1:5-di-*p*-toluidino-, and 1-*p*-toluidino-4-hydroxyanthraquinones, and from 4-*p*-toluidino-*N*-methylpyridoneanthrone. C. HOLLINS.

**Obtaining absolute alcohol.** D. B. KEYES, Assr. to U.S. INDUSTRIAL ALCOHOL Co. (U.S.P. 1,676,735, 10.7.28. Appl., 27.12.22).—See B.P. 206,747; B., 1924, 193.

**Process of making acetic acid.** H. W. MATHESON, Assr. to CANADIAN ELECTRO PRODUCTS Co., LTD. (U.S.P. 1,676,454, 10.7.28. Appl., 31.3.21).—See B.P. 132,558; B., 1919, 846.

**Quinoline derivatives** (G.P. 440,008). **Acridine compounds** (B.P. 293,617).—See XX.

#### IV.—DYESTUFFS.

***p*-Cymene. X. 2:5-Diamino-*p*-cymene and certain new dyes.** A. S. WHEELER and R. W. BOST (J. Amer. Chem. Soc., 1928, 50, 2000—2005).—2:5-Diamino-*p*-cymene hydrochloride (cf. Liebermann and Ilinsky, A., 1886, 239) is most conveniently prepared by reducing 5-*p*-sulphobenzeneazocarvacrylamine hydrochloride (A., 1928, 55) with stannous chloride and hydrochloric acid. An apparatus is described suitable for the isolation of the pale yellow free amine, which is rapidly oxidised by air and gives *p*-cymoquinone with cold ferric chloride. It yields a *dihydrobromide*, m.p. 330° (decomp.); *dinitrate*, m.p. 196° (decomp.); *monochloroacetate*, m.p. 144°; *bisdichloroacetate*, m.p. 165°; *bistrichloroacetate*, m.p. 171°; *monobromoacetate*, m.p. 148° (decomp.); *dibenzene-sulphonate*, decomp.; *monobenzoate*, m.p. 145°; *mono-*o*-chlorobenzoate*, m.p. 161°; *mono-3:5-dinitrobenzoate*, m.p. 199° (decomp.); *mono-2:4:6-trinitrobenzoate*, m.p. 130° (decomp.); *dipicrate*, m.p. 207° (decomp.); and *dibenzoyl*, m.p. 280°, *dithiocarbamyl*, m.p. 235—237°, and *dicarbamyl*, m.p. above 350°, derivatives. With anhydrous oxalic acid in ether it forms *p*-aminocymyl-oxamic acid, m.p. 210—211°. Disazo dyestuffs of the following colours and m.p. have been prepared by tetrazotising the amine at 0° and coupling with 2 mols. of the component named: resorcinol, brown, m.p. 168—170°; β-naphthol, violet-purple, m.p. 295—298° (decomp.); sulphanilic acid, orange-yellow, m.p. 100° (decomp.); 2-naphthol-3:6-disulphonic acid, blue, m.p. above 340°; naphthionic acid, red, m.p. 270—272° (decomp.); β-thionaphthol, buff, m.p. 138—140°. When boiled with *p*-nitrosodimethylaniline or *p*-nitrosophenol in dilute alcohol, it yields the Eurhodine (aminophenazine) dyes, C<sub>18</sub>H<sub>22</sub>N<sub>4</sub>, purplish-black, m.p. 300° (decomp.), and C<sub>16</sub>H<sub>17</sub>ON<sub>3</sub>, brown, m.p. 200—206° (decomp.), respectively. The colours given by the above dyes on silk, wool, and cotton are tabulated.

H. E. F. NOTTON.

#### PATENTS.

**Manufacture of dyes [preservation in finely-divided condition].** I. G. FARBENIND. A.-G. (B.P. 269,918, 22.4.27. Ger., 22.4.26).—The finely-divided dye is mixed with up to 10% of a cellulose alkyl (methyl ether and then retains indefinitely its fine division. The mixture may be used as paste or may be dried without coagulation or crystallisation. C. HOLLINS.

**Manufacture of benzantrones.** I. G. FARBENIND. A.-G. (B.P. 268,830, 4.4.27. Ger., 3.4.26).—Anthrone or oxanthrol is condensed in sulphuric acid with ketones or esters of the general formula, R·CO·CR'·CHR'', where R and R' may be alkyl, aryl, or alkoxy, R'' may also be hydrogen, and R' may be any radical. The unsaturated ketone may be generated *in situ* from corresponding hydrates or hydrogen halides. The preparation of the following benzantrones is described: 3-methyl- (from vinyl methyl ketone, methyl β-hydroxyethyl ketone, hydroxymethyleneacetone, ethoxymethyleneacetylacetone, or ethyl ethoxymethyleneacetoacetate), m.p. 164°; 3-phenyl- (from phenyl vinyl ketone or β-chloropropiophenone), m.p. 183°; 2:3-dimethyl- (from methyl hydroxymethylene-ethyl ketone), m.p. 207°; 2-methyl-3-ethyl- (from hydroxymethylenediethyl ketone), m.p. 142°; 1:3-diphenyl- (from phenyl styryl ketone), m.p. 195—196°; 1:3-dimethyl- (from propenyl methyl ketone), m.p. 114—115°; 1-phenyl-3-methyl- (from styryl methyl ketone), m.p. 175—176°; 3-phenyl-1-methyl- (from phenyl propenyl ketone), m.p. 174—175°; 1-*p*-anisyl- (from *p*-anisyl β-chloroethyl ketone, m.p. 64°, by way of an intermediate product, m.p. 181°, m.p. 186°; 3-hydroxy-2-acetyl- (from ethyl hydroxymethyleneacetoacetate, m.p. 295°; 3-hydroxy-2-carbethoxy- (from ethyl ethoxymethylenemalonate), m.p. 206°; 3-hydroxy-2-phenyl- (from ethyl phenyl-hydroxymethyleneacetate), m.p. 230°. C. HOLLINS.

**Production of dyes [for wool and silk].** L. B. HOLLIDAY & Co., LTD., J. KITSON, and C. SHAW (B.P. 293,110, 30.3.27).—Chloranil or bromanil is hydrolysed with dilute sodium hydroxide or carbonate, the 3:6-dichloro(or dibromo)-2:5-dihydroxy-*p*-benzoquinone is filtered off, and the filtrate treated with acid or salt to precipitate a brown dye for wool or silk. Similar products are obtained by condensing chlor- or brom-anil with its hydrolysed product in alkaline solution.

C. HOLLINS.

**Manufacture of chloroperylenequinones.** F. BENSÄ (B.P. 281,281, 17.11.27. Austr., 26.11.26).—Highly chlorinated perylenes, containing 8—12 (especially 12) atoms of chlorine per molecule, are converted by 25% oleum at 150—160° into octachloroperylenequinone, an olive-green vat dye. C. HOLLINS.

**Manufacture of vat dyes.** O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 292,741, 5.5.27).—Halogenated perylenetetracarboxylic dianhydrides containing less than 4 (preferably 2) halogen atoms are condensed with *o*-phenylenediamine. The vat dyes so obtained give more easily soluble leuco-compounds than the non-halogenated product (B.P. 201,786; B., 1923, 986 a) and the dyeings are faster to soap and sodium carbonate than those of the tetrachloro-compound (B.P. 248,519; B., 1926, 355). C. HOLLINS.

**Manufacture of [black] vat dyes.** I. G. FARBENIND. A.-G. (B.P. 280,846, 6.4.27. Ger., 18.11.26).—Diazobenzanthrone, prepared from the reduced nitro-compound of Example 2 of B.P. 12,518 of 1906 (cf. B., 1906, 844), is treated with potassium xanthate or other sulphurising agent, and the product is fused with alkali (alcoholic potassium hydroxide at 280°) to give an intense black vat dye. C. HOLLINS.

**Manufacture of vat dyes of the anthraquinone series.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 290,408, 24.3.27).—2 : 6(or 7)-Dithiolanthraquinone is condensed with 2 mols. of 1-chloroanthraquinone-2-carboxylic acid or 1-diazoanthraquinone-2-carboxylic acid, or 2 : 6(or 7)-tetrazoanthraquinone is condensed with 2 mols. of 1-thiolanthraquinone, and the resulting 2 : 6(or 7)-bis-(2-carboxyanthraquinonylthiol)anthraquinone is cyclised to an anthraquinonebisthioxanthone by heating in concentrated sulphuric acid or boiling benzotrichloride. The products are yellow vat dyes of high tinctorial power and good fastness. C. HOLLINS.

**Manufacture of orange vat dyes of the anthraquinone series.** I. G. FARBENIND. A.-G. (B.P. 280,492, 25.4.27. Ger., 15.12.26).—Orange vat dyes are produced by converting 1-acylamino-4-hydroxyanthraquinones into their ethers by treatment with hydroxyalkylating agents, e.g., *p*-toluenesulphonic esters of ethylene glycol or  $\beta$ -chloroethyl alcohol, glycerol, etc. From 1-benzamido-4-hydroxyanthraquinone and  $\beta$ -chloroethyl *p*-toluenesulphonate are obtained (a) a mixture of  $\beta$ -chloro- and  $\beta$ -hydroxy-ethyl ethers and (b) the ethylene ether of the hydroxy-compound. The ethylene ether also results when ethylene di-*p*-toluenesulphonate is used. 1-Anisamido-4-hydroxyanthraquinone gives a similar yellow-orange vat dye, which does not change shade on soaping. C. HOLLINS.

**Manufacture of dyes of the anthraquinone series.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 292,896—7, 18.3.27).—(A) The 1 : 1'-dihydroxy-2 : 2'-dianthraquinonyl of G.P. 167,461 (cf. Scholl and others, A., 1920, i, 169) is obtained more smoothly by fusing the  $\alpha$ -hydroxyanthraquinone with alkali in presence of an organic medium (alcohol, aniline) and with the exclusion of oxygen. Similar products are prepared from substituted  $\alpha$ -hydroxyanthraquinones. That from 4-amino-1-hydroxyanthraquinone gives a grey, that from 1-hydroxy-4-methoxyanthraquinone a brown, on wool from a faintly alkaline vat. (B) The crude alkaline fusion products of the preceding patent may be purified by treatment with oxidising agents (hypochlorite, permanganate) and/or fractional precipitation from sulphuric acid. C. HOLLINS.

**[Anthraquinone] dyestuffs and intermediates.** H. W. HERWARD, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 293,328, 29.12.26).—Hydroxyanthraquinones, particularly alizarin, are produced by heating 2-methylanthraquinone or anthraquinone-2-carboxylic acid with caustic alkali at 180° in presence or absence of oxidants such as sodium chlorate. C. HOLLINS.

**Manufacture of copper compounds of substantive azo dyes.** W. CARPMAEL. From I. G. FARBENIND.

A.-G. (B.P. 292,660, 18.3.27).—Copper compounds fast to alkali and ironing are obtained from azo couplings of *o*-aminophenol ethers (excluding *o*-anisidine containing a 3-hydroxyl or 3-carboxyl group). Examples of suitable azo dyes are: *o*-anisidine (2 mols.)  $\rightleftharpoons$  di-J-acid (reddish-violet); 4-nitro-*o*-anisidine (2 mols.)  $\rightleftharpoons$  di-J-acid (bluish-violet); 5-nitro-*o*-anisidine-4-sulphonic acid (2 mols.)  $\rightleftharpoons$  phenyl-J-acid (violet); *o*-aminophenyl benzyl ether (2 mols.)  $\rightleftharpoons$  carbonyl-J-acid (bordeaux); *o*-phenetidine (2 mols.)  $\rightleftharpoons$  di-J-acid (reddish-violet); *o*-anisidine  $\rightarrow$  carbonyl-J-acid  $\leftarrow$  aniline-*o*-sulphonic acid (red); *o*-aminophenoxyacetic acid (2 mols.)  $\rightleftharpoons$  di-J-acid (violet); *o*-aminophenoxyethyl alcohol (2 mols.)  $\rightleftharpoons$  di-J-acid (reddish-violet); aniline-2 : 5-disulphonic acid  $\rightarrow$  2 : 5-dimethoxyaniline  $\rightarrow$  phenyl-J-acid (blue). Numerous variations of these examples are mentioned. C. HOLLINS.

**Metallic compounds of *o*-hydroxyazo dyes.** I. G. FARBENIND. A.-G. (B.P. 268,754, 17.3.27. Ger., 31.3.26).—Metal compounds of *o*-hydroxyazo dyes which contain nitro- or amino-groups may be modified by reduction of nitro-groups or acylation of amino-groups without destroying the metal complex. Examples are the chromium compounds of: 4-nitro-2-aminophenol-6-sulphonic acid  $\rightarrow$   $\beta$ -naphthol, reduced with sodium sulphide (bluish-grey); *p*-chloro-*o*-aminophenol  $\rightarrow$  H-acid, acetylated (reddish-blue); nitro-1 : 2 : 4-aminonaphtholsulphonic acid  $\rightarrow$   $\beta$ -naphthol, reduced with alkali and dextrose (dark blue); also the copper compound of picramic acid  $\rightarrow$  H-acid, acetylated (redder shade). C. HOLLINS.

**Manufacture of intermediate products and vat dyestuffs derived from diaroylperylene.** COMP. NAT. DE MAT. COL. ET MANUF. DE PROD. CHIM. DU NORD RÉUNIS, ÉTABL. KUHLMANN (B.P. 267,121, 1.3.27. Fr., 5.3.26).—See F.P. 612,338; B., 1927, 698.

Coloured masses etc. (B.P. 293,067).—See XIII.

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

**Degumming of silk.** III. Action of hydrochloric acid on the sericin and effect of formaldehyde on the action. IV. Gelatinisation of the sericin. T. TAKAHASHI (J. Soc. Chem. Ind. Japan, 1928, 31, 34—39, 39—45; cf. B., 1928, 184).—III. Raw silk was boiled for 30 min. with hydrochloric acid of various concentrations ranging from  $5 \times 10^{-3}$  to  $95 \times 10^{-3}N$ , and the amount of acid consumed and the degree of degumming (i.e., loss of weight of the fibre mass) were determined. The effect of formaldehyde on these values was also examined. The acidity of the bath,  $2-3 \times 10^{-3}N$ , was supposed to be the isoelectric point of sericin. The degree of degumming decreased as the acidity of the bath was increased up to this acidity, reaching a minimum at this point, and then increased; the amount of hydrochloric acid consumed, however, increased up to this point and then remained stationary even after further increase of the acidity. In the presence of formaldehyde the degree of degumming showed no minimum as the acidity of the bath increased. The curves for the degree of degumming in the presence and absence of formaldehyde, respectively, coincided at the minimum point of the latter and again further on. The amount of acid

consumed, however, in the presence of formaldehyde at first increased as the acidity of the bath increased, reached a maximum, and then decreased. It is concluded that when the acidity of the hydrochloric acid bath is less than  $2-3 \times 10^{-3}N$ , the sericin adsorbs hydrogen ions and becomes gelatinised; above this point it does not gelatinise, but reacts with the acid to form a soluble hydrochloride.

IV. The viscosity of the silk boiling-off liquor produced from the degumming baths of various  $p_H$  values in presence or absence of formaldehyde was examined. As the  $p_H$  value of the bath increased, the viscosity of the liquor increased and reached a maximum at a value corresponding to  $3 \times 10^{-4}N$  of sodium hydroxide, and then decreased. This point agreed with the maximum point of gelatinisation. In the presence of formaldehyde, liquors of low viscosity were obtained indicating the destruction thereby of the gelatinising properties of sericin. It is concluded that degumming is mainly due to gelatinisation of sericin in the alkaline liquor, and to swelling in the acid liquor. S. OKA.

#### Composition and use [distillation] of argan wood.

A. DA C. CABRAL (Bull. Inst. Pin, 1927, 289; Chem. Zentr., 1928, i, 1295).—The dry wood of *Argania sideroxylon* (Morocco) yields: lignin 32.36,  $\alpha$ -cellulose 50.90,  $\beta + \gamma$ -cellulose 5.10, ether extract 0.27%. Dry distillation (8 hrs. at  $500^\circ$ ) yields carbon 28.1, anhydrous tar 4.9, soluble tar 2.0, acetic acid 2.2, methyl alcohol 0.7%. The tar affords oil 40.1, residue 51.7, gas 8%. A. A. ELDRIDGE.

Investigation and purification of sulphate-turpentine. I. J. POSTOVSKI and V. G. PLJUSNIN (Ann. Inst. Polyt. Ural, 1927, 6, 245—258).—The turpentine oil obtained in abundance in the preparation of cellulose by the sulphate process may be readily purified from compounds which give precipitates with mercury salts (mercaptans and sulphides) by distillation in a current of steam. The first 15% of the distillate contains 86% of these sulphur compounds, and if 50% of the oil is distilled only 2% of the sulphur compounds remains. The purified sulphate-turpentine is a colourless liquid of characteristic, pleasant odour, of initial b.p.  $153^\circ$ ,  $d_{15}^{20}$  0.8645,  $n_D^{20}$  1.46728,  $[\alpha]_D^{20} +14.47^\circ$ . It contains 90% of  $\alpha$ - (and  $\beta$ -) pinene and not more than 1% of dipentene, and is suitable for the preparation of paints and varnishes, and for pharmaceutical purposes. T. H. POPE.

Cellulose ester solvents. II. H. WOLFF [with G. ZEIDLER and W. TOELDT] (Farben-Ztg., 1928, 33, 2668—2672).—Solvents of the glycol type (cf. B., 1928, 519) were further investigated from the practical point of view. The conditions under which a tendency to irregularity appears in the films of lacquers containing these solvents and varying amounts of diluents, and the correcting of the irregularity by the addition of butyl alcohol and plasticisers are described. The use of the glycol solvents, particularly "ethyl glycol," in lacquers induces good "insulation" from oil undercoats, whilst improving the rubbing qualities and elasticity of the lacquer film. The whitening effect produced by "methyl glycol" may be counteracted by suitable solvent mixtures, or alternatively may be made use of

to obtain opacity with a smaller concentration of pigment than would otherwise be necessary. The mechanical properties of such films are in no way inferior to those of corresponding films containing butyl acetate as solvent. S. S. WOOLF.

Detergent experiments on cotton. CHAPIN.—See XII.

#### PATENTS.

Obtaining textile fibres of high quality from the skins of fish, e.g., *Chondropterygii selachii*. A. EHRENREICH (B.P. 284,297, 27.1.28. U.S., 27.1.27).—The fleshed skins of fish, particularly those of the plagiotomi such as sharks, are treated for a short period with lime, then subjected to the diastatic action of enzymes such as an extract of trypsin or of shark glands for 24—30 hrs. at  $35-40^\circ$  whereby the intercellular substances are disintegrated and the adhesion between the fibres of the skin is reduced; the skins are then rinsed and tanned, and the fibres separated by hand or mechanically. It is advantageous to treat the skins, immediately after their treatment with enzymes, with a colloid such as gum tragacanth or tragasol. The resulting fibres are durable, have a high lustre, and are capable of being printed, dyed, and finished. A. J. HALL.

Saccharification of cellulose-bearing material. J. PERL, Assr. to M. M. CORY (U.S.P. 1,677,046, 17.7.28. Appl., 3.7.25).—Cellulosic material is disintegrated by treatment with a counter-current of anhydrous hydrogen chloride diluted with a cooled inert gas. A. J. HALL.

Manufacture of coloured cellulose plastics and solutions thereof. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 293,485, 8.4.27. Addn. to B.P. 247,288; B., 1926, 315).—Large amounts of colouring substances (up to 25%) are incorporated in the plastic masses prepared by methods described in the chief patent, so that the resulting products when diluted with solvents or other substances do not require the further addition of colouring substances. A. J. HALL.

Process of making paper. PLASTIC, INC., Assees. of A. L. KENNEDY (B.P. 287,538, 8.6.27. U.S., 25.3.27).—Paper having superior resistance to electricity, heat, cold, oils, and moisture is manufactured by incorporating alginic acid in a soluble form in a fibrous pulp, then precipitating the alginic acid by the addition of a salt (e.g., zinc sulphate or calcium acetate) or lime or a mineral acid, washing the product, and converting it into paper by the usual methods. A. J. HALL.

Production of high- $\alpha$ -cellulose fibre. G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,678,230, 24.7.28. Appl., 28.4.25).—See B.P. 278,767; B., 1927, 935.

Filters for the fluid material for production of artificial textile threads or fibres. H. KINDERMANN (B.P. 293,816, 12.4.27).

Spinning machines for artificial silk. C. HAMEL A.-G. (B.P. 284,986, 14.9.27. Ger., 7.2.27).

Drying apparatus for textile goods etc. E. J. ROWE (B.P. 293,954, 9.6.27).

Binding materials (B.P. 293,135—6).—See II. Tanning materials (F.P. 620,394).—See XV. Nitration of cellulose (B.P. 293,190).—See XXII.

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

## PATENTS.

**Dyeing of mixed textile goods.** I. G. FARBENIND. A.-G. (B.P. 267,985, 22.3.27. Ger., 27.3.26).—Level shades on silk-wool mixtures are obtained by using sulphonated mono- or dis-azo couplings of phenyl-pyrazolones containing in either or both components one or more ether groups. Examples are: 1-*p*-sulpho-phenyl-3-methyl-5-pyrazolone coupled with *o*-anisidine, *o*-aminodiphenyl ether, 3-aminotolyl benzyl ether; aniline-*o*-sulphonic acid  $\rightarrow$  1-*p*-anisyl-3-methyl-5-pyrazolone; 4-amino-2-sulphodiphenyl ether  $\rightarrow$  1-phenyl-3-methyl-5-pyrazolone. C. HOLLINS.

**Production of fast dyeings from dyes of the anthraquinone series.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 292,658, 18.3.27).—Wool, cotton, and artificial fibres are dyed with 1:1'-dihydroxy-2:2'-dianthraquinonyl (from alkaline fusion of  $\alpha$ -hydroxyanthraquinone; G.P. 167,461) from a faintly alkaline vat in fast yellow shades. The products of B.P. 292,896—7 (B., 1928, 667) may be used with advantage in this way. C. HOLLINS.

**Dyeing mixed textile goods.** H. WAGNER, H. EICHWEDE, and E. FISCHER, AssTs. to GRASELLI DYESTUFF CORP. (U.S.P. 1,678,611, 24.7.28. Appl., 28.1.26. Ger., 7.2.25).—See B.P. 247,224; B., 1927, 520.

**Dyeing with vat and azo dyes.** W. WINTERHALDER, AssT. to GRASELLI DYESTUFF CORP. (U.S.P. 1,678,580, 24.7.28. Appl., 15.5.26. Ger., 20.5.25).—See B.P. 252,384; B., 1927, 775.

**Dyeing apparatus [for hat bodies etc.].** V. BÖHM (B.P. 270,758, 9.5.27. Austria, 7.5.26).

**Sulphonic acids** (B.P. 263,873).—See III. **Printing colours etc.** (B.P. 292,665 and 292,894).—See XIII.

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

**Preparation of mixed acid.** VON BEZOLD (Z. ges. Schiess- u. Sprengstoffw., 1928, 23, 190—193).—Methods for preparing mixed acids are described. Continuous mixing may be carried out in the turbine apparatus described by Neumann (G.P. 274,854). This apparatus was designed for continuous nitration of toluene, but it is equally suitable for acid mixing. Another type of mixer is described which has no moving parts. The mixing takes place in a cooled azidur coil into which the nitric and sulphuric acids are fed together with previously prepared mixed acid. By using two sets of measuring vessels the mixing can be made practically continuous. S. BINNING.

**Catalytic oxidation of ammonia.** VII. L. ANDRUSSOV (Z. angew. Chem., 1928, 41, 205—206; cf. A., 1927, 1039).—A reply to criticism by Raschig of previous work (cf. B., 1927, 906). The author re-states his reasons for believing that nitroxyl, HNO, is an intermediate product of the catalytic oxidation of ammonia. W. A. RICHARDSON.

**Catalytic oxidation of ammonia.** F. RASCHIG (Z. angew. Chem., 1928, 41, 207).—A reply to Andrussov (see preceding abstract).

**Regulating the density of milk of lime.** L. DYACHUK (Nauch. Zapiski, 1928, 5, 285—287).—A new type of automatic regulator is described.

CHEMICAL ABSTRACTS.

**Electrolytic removal of iron from aluminium salts.** L. WASILEWSKI and S. MANTEL (Przemysl Chem., 1928, 12, 48—51).—Iron present as an impurity in aluminium salts can be separated electrolytically using a mercury cathode; in this way 3 mg. of ferric oxide per 100 g. of aluminium oxide can be separated. The iron is present in colloidal solution in the mercury cathode, from which it can be separated by filtration through chamois leather. The residue after distillation of the mercury contains 1.27% of aluminium.

R. TRUSZKOWSKI.

**Removal of iron from aluminium salts by recrystallisation.** S. ZABICKI (Przemysl Chem., 1928, 12, 77—90).—Ammonium alum containing only 0.002% of ferric oxide is obtained in 86% yield by three successive recrystallisations, using concentrations of not above 68% of alum; above this concentration supercooling is liable to occur. Rapid stirring favours the formation of small crystals, thereby yielding a purer product.

R. TRUSZKOWSKI.

**Making pure tungstic oxide [from wolframite].** W. B. GERO and C. V. IREDELL (Chem. Met. Eng., 1928, 35, 412—417).—Wolframite concentrates containing 65—75% WO<sub>3</sub> are ground and digested with concentrated boiling caustic soda, calcium chloride is added to the resulting solution, and the calcium tungstate digested with boiling hydrochloric acid. The tungstic acid is converted into ammonium paratungstate and the latter ignited. The lay-out of a plant to produce tungstic oxide of high purity by this process is discussed. Combined mechanical and steam agitation is employed in the caustic soda treatment of the ore, which is very heavy. The hydrochloric acid treatment of the calcium tungstate is performed in a concrete tank with internal lining of acid-proof brick backed by asphalt. A rubber-covered agitator and steam pipe for heating are used, together with a stoneware outlet closed by a hard rubber plug for tungstic acid removal. The clear solution is siphoned off. The dissolution of tungstic acid in ammonia and the subsequent filtration are carried out in wooden vessels, and all pipe lines in this stage are of hard rubber; the precipitation of the ammonium paratungstate is carried out in a rubber-lined steel tank. The necessary hydrochloric acid is added through 24 fine jets up to neutrality, the precipitate washed by decantation, drained on wooden vacuum filters, and dried on glass trays. The ignition is carried out on fused silica trays. The finished product contains over 99.98% WO<sub>3</sub>. C. IRWIN.

**Drying sulphur recovered from manufactured gas.** K. N. CUNDALL (Chem. Met. Eng., 1928, 35, 407—411).—Sulphur is now being recovered as a paste by the liquid purification process at many gas works in Western America. It is very finely divided and contains 55% of moisture. If this paste is treated with steam in a digester at about 119° the sulphur is flocculated and partial drying results. Addition of alkali is necessary to neutralise sulphuric acid formed. The partly melted

sulphur is dark in colour and contains 25% of moisture, but is suitable for the manufacture of lime-sulphur preparations. A rotary gas-fired dryer for complete drying was abandoned owing to the discovery that excessive heating detracted from the toxicity of the sulphur dust. A steam-heated tunnel dryer in which the sulphur was treated in the form of press cakes was found satisfactory. The quantity dried was 0.2 lb. per sq. ft. of tray space per hour and the loss was small. The sulphur can also be coagulated by boiling, after which it may be partially dried by centrifuging. This sulphur is much more toxic than other forms and more finely divided, most of the particles being below 15  $\mu$ .

C. IRWIN.

**Iron carbonyl.** MITTASCH.—See X. **Electrolysis of water.** NOEGGERATH.—See XI. **Tannery investigations.** GRASSER.—See XV.

#### PATENTS.

**Manufacture of arsenic acid.** F. ULLMANN and G. TREWENDT, Assrs. to J. MICHAEL & Co. (U.S.P. 1,677,251, 17.7.28. Appl., 26.6.23. Ger., 6.3.23).—A mixture of 10 pts. of arsenic trioxide, 20 pts. of water, and 3.8 pts. of potassium chlorate is heated to boiling, and a small amount of hydrochloric acid is added.

H. ROYAL-DAWSON.

**Synthetic manufacture of ammonia.** I. G. FARBENIND. A.-G. (B.P. 267,554, 12.3.27. Ger., 13.3.26).—The gases are brought into contact with a catalyst of pure iron produced by thermal decomposition of iron carbonyl, and a small percentage of alumina, magnesia, zirconium oxide, or potassium oxide as activating agent.

W. G. CAREY.

**Manufacture of alkali nitrates.** W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 293,495, 12.4.27).—An alkali nitrite is treated with oxygen or air at 300–500° in presence of a catalyst, *e.g.*, an alkali hydroxide or an alkaline-earth oxide, intimate contact being obtained by using an easily liquefiable catalyst or by melting the nitrite; the action below 360° can be facilitated by using an eutectic mixture, *e.g.*, caustic potash and caustic soda, melting at 180°.

W. G. CAREY.

**Manufacture of chromium oxide and chromium hydroxide.** W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 293,494, 12.4.27).—An aqueous solution of an alkali chromate is heated above 110°, under pressure, with an organic reducing agent, *e.g.*, a carbohydrate soluble in water, or sawdust, and the resulting hydroxide is washed, dried, and ignited.

W. G. CAREY.

**Production of anhydrous chlorides free from oxides.** A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 293,410, 6.4.27).—Oxides, substances containing oxides, or chlorides which on heating decompose into oxides are mixed with reactive carbon, made into a suspension in fused chlorides and caused to flow down a heated irrigation tower in counter-current to hydrogen chloride, chlorine, phosgene, etc. The coke filling of the tower serves as resistance for the electric heating.

W. G. CAREY.

**Carrying out chemical reactions in which reaction gases are circulated.** SYNTHETIC AMMONIA & NITRATES, LTD., and J. HUGHES (B.P. 293,138, 6.4.27).—The

invention is illustrated by the following example. Gas removed from a synthetic ammonia plant containing an undue accumulation (5%) of useless argon and methane is scrubbed with kerosene at the working pressure of 200 atm., the quantity of oil used being 10 cub. m. per 1000–5000 cub. m. of gas, and the temperature normal or lower. The scrubbed gas is returned to the ammonia plant. The pressure of the liquid is let down in a first stage to 3 atm., in a Pelton wheel if desired, and the gas released will contain roughly 25% each of methane and nitrogen and 40–50% of hydrogen. In the second stage to atmospheric pressure the gas evolved will contain 50–60% of argon, the rest nitrogen; this fraction may be of value as a source of argon.

B. M. VENABLES.

**Process of making colloidal iodine.** R. W. JAMES. From MERCK & Co. (B.P. 293,504, 20.4.27).—Iodine is dissolved in alkali hydroxide solution, gum arabic (2% of the iodine present) is added as a protective colloid, and colloidal iodine is precipitated by adding hydrochloric acid.

W. G. CAREY.

**Converting hydrogen sulphide into sulphur dioxide.** H. BÄHR, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,678,630, 31.7.28. Appl., 4.5.25. Ger., 6.5.24).—See F.P. 609,931; B., 1927, 252.

**Process of making a mixture of nitrogen and hydrogen.** F. G. LILJENROTH (U.S.P. 1,678,518, 24.7.28. Appl., 17.10.22. Swed., 3.12.21).—See B.P. 189,789; B., 1923, 1071.

**Catalytic reactions** (B.P. 289,759).—See I. **Rotary kiln** (B.P. 293,331).—See IX. **Phosphate fertilisers** (B.P. 282,619).—See XVI.

#### VIII.—GLASS; CERAMICS.

**Ancient glasses.** III. B. NEUMANN (Z. angew. Chem., 1928, 41, 203–204).—Analysis of glasses from the Babylonian city of Nippur, and dating from about 250 B.C., shows that the samples fall into three groups, distinguished by the colours blue, green, and rose. The chemical compositions of members of the same group are similar, but there are considerable differences between the groups. All contain the sulphur characteristic of ancient glass, and high proportions of iron and alumina. The alkali content is exceptionally low (9–13% Na<sub>2</sub>O) and all contain copper and manganese. The rose colour is ascribed to manganese, the blue to copper and manganese, and the green to interaction between alkali sulphide (the content of which is high in these glasses) and copper or manganese oxide.

W. A. RICHARDSON.

**Casing of clear by opal glass. Importance of annealing.** F. WINKS and W. E. S. TURNER (J. Soc. Glass Tech., 1928, 12, 161–163).—The coefficients of expansion of a clear glass and of an opal glass which had shown a tendency to crack off when used for flashing this were in close agreement over the normal range. On the other hand, the lower and upper annealing temperatures of the opal were 30° and 53° respectively above that of the colourless glass.

A. COUSEN.

**Melting of glass.** R. HEMINGWAY (J. Soc. Glass Tech., 1928, 12, 131–134).—In a cross-flame regenera-

tive oil-fired tank furnace, good colourless glass had been obtained at the rate of 1 ton per 9 sq. ft. of melting area per 24 hrs., with a maximum safe peak-load rate of 1 ton per 8 sq. ft. The corresponding rates for an amber glass were 6 and 5 sq. ft. respectively, and the increased melting rate in this case was found to be due to the sulphur employed in the batch. A. COUSEN.

**Acceleration of glass melting by the use of volatile constituents in the glass batch mixture.** W. E. S. TURNER (*J. Soc. Glass Tech.*, 1928, 12, 134—138).—The batch tested was of a simple soda-lime type, and the volatile agents added were ammonium chloride, nitrate, or sulphate. All accelerated the melting rate in pot furnaces, whilst the chloride and sulphate also accelerated fining. Results were less effective in tank furnaces. A. COUSEN.

**Design and operation of glass furnaces.** W. W. WARREN (*J. Soc. Glass Tech.*, 1928, 12, 128—131).—Means of improving the efficiency of glass-melting furnaces, as by insulation and utilisation of waste heat, the provision of measuring and regulating instruments in the furnace system, etc., are advocated. The regenerative pot furnace is more efficient than the recuperative type, and, in pot melting, the plunging of glass during founding with arsenious oxide improves the fining rate. A. COUSEN.

**Control and distribution of temperature in lehrs.** A. COUSEN, H. W. HOWES, and F. WINKS (*J. Soc. Glass Tech.*, 1928, 12, 146—158).—Essential temperature conditions within a lehr for the successful and economical annealing of glass are discussed. Temperature conditions along lehr belts have been explored by the use of a nichrome-kromore traveller thermocouple, by means of which the temperature distribution generally holding in direct-fired, semi-muffle and muffle lehrs has been found. A. COUSEN.

**Physicochemical examination of the Borovitcha refractory clays.** G. G. URAZOV and N. I. VLODAVTZA (*Ann. Inst. Anal. Phys. Chem.*, 1927, 3, 725—745).—Plastic clays have a higher water content than friable clays, as well as a higher titanium, iron, and alkaline-earth content. Three breaks may be observed in the temperature-time curves obtained by heating the clays to 1100°. The first two, indicating endothermic effects, occur at 100—110° and 400°, and are due to the loss of adsorbed and constitutional water respectively. The third, due to an exothermic reaction, takes place at 950° and is less marked for friable than for other clays. Curves connecting degree of dehydration of air-dry refractory clays with temperature show that at 400° 90% of the water originally present is given up; the remainder disappears at 520—600°. It is suggested that these clays consist of kaolinite, and that the air-dry clay ( $\text{Al}_2\text{Si}_2\text{O}_7 \cdot n\text{H}_2\text{O}$ ) between 100° and 400° continually loses water, the resulting substance being hydrated leverrierite,  $\text{Al}_2\text{Si}_2\text{O}_7 \cdot m\text{H}_2\text{O}$ , which at about 600° is completely dehydrated. X-Ray measurements of the angles of the crystal lattices of various Russian clays show that these are very close to one another and to those of nacrite. R. TRUSZKOWSKI.

**Decomposition of clays.** L. WASILEWSKI (*Przemysl Chem.*, 1928, 12, 40—48).—The decomposition of clays

and kaolin by ammonium sulphate proceeds as if ammonium hydrogen sulphate and ammonium alum are first produced (up to 357°), then ammonia and ammonium sulphate are driven off from the alum (420—430° to 450°). Up to 360° about 0.5% of the ammonia is oxidised, and ammonium alum is leached out of the products, whilst at 450° 9% of ammonia is oxidised and aluminium sulphate is obtained. Clays with a high iron content release ammonia more readily, whilst those free from iron resist conversion into aluminium sulphate. CHEMICAL ABSTRACTS.

**Firing of clays in the presence of water vapour and sulphur dioxide.** J. KONARZEWSKI and B. KRYŃSKI (*Przemysl Chem.*, 1928, 12, 176—184).—Below 550° the dehydration of china clay is retarded by the presence of water vapour; above this temperature the velocity of dehydration is unaffected by this factor. The presence of sulphur dioxide has no influence on the velocity of dehydration; the fired clay, however, contains in this case sulphates of calcium, magnesium, aluminium, and iron. The sulphur can be entirely eliminated by refiring in an oxidising atmosphere. Alumina and ferric oxide can to a certain extent be extracted from fired clay by hydrochloric acid. R. TRUSZKOWSKI.

**Adsorption phenomena with clays in non-aqueous media.** B. NEUMANN and S. KOBER (*Kolloidchem. Beih.*, 1928, 27, 1—43).—Measurements of the adsorption of a silver xylosol have shown that the peculiar relations of heated clay towards the adsorption of non-aqueous dye solutions and vegetable oils are to be traced to the different degrees of dispersion. A differentiation is made between the adsorption of crystalloidal and colloidal matter by clays. Investigation of the adsorption of a large number of substances showed a continuous progression from crystalloidal to colloidal behaviour. Silver xylosol and a gold-resin sol furnish examples of purely colloidal adsorption. Curves relating the adsorption of gold sol to the temperature of heating are given for some clays, and irregularities are traced to gas adsorption. It is concluded that the essential alteration taking place in clays on heating is a reversible change in capillary structure. E. S. HEDGES.

**Analysis of silicates by decomposition with alkali hydroxides in a nickel crucible.** C. J. VAN NIEUWENBURG and H. H. DINGEMANS (*Chem. Weekblad*, 1928, 25, 266—268).—Either sodium or potassium hydroxide in the molten condition effects practically complete decomposition of most silicates at temperatures of about 400°, at which nickel is very little attacked if it be kept out of direct contact with the flame. The nickel crucible may therefore be used for silicate analyses by this method, and the fused mass boiled with water in the crucible itself. S. I. LEVY.

**Dolomite for steel works.** JACOBS.—See X.

#### PATENTS.

**Manufacture of compound glass.** E. GEISEL (*B.P.* 293,671, 27.3.28).—The products of polymerisation of methyl or ethyl acrylate are used as a uniting layer between sheets of glass, or as a coating to a single sheet. A. COUSEN.

**Clay product, ceramic composition, and associated processes.** VITREFRAX Co., Assees. of T. S. CURTIS (B.P. 273,258, 16.5.27. U.S., 22.6.26).—A natural aluminium silicate, such as cyanite, the crystals of which expand in the direction of their major axes, at the temperature of firing, is mixed with the ceramic mass before moulding. The product is strengthened by the resulting network of elongated crystals, and no shrinkage takes place. To prepare the cyanite, the crystals are heated at 927–982°, subjected to table-concentration with water, and then milled. Alternatively, the red-hot crystals are quenched in cold water, whereby the bulk of the impurities are separated without breaking the crystals transversely.

F. G. CLARKE.

**Up-draught process of and muffle kiln for firing.** T. DOWNS (U.S.P. 1,677,818, 17.7.28. Appl., 25.1.27).—The air for drying passes upwards around the sides of the kiln and over its domed roof. The air also passes beneath the kiln and up through a central vertical flue. The various air currents can be regulated.

F. G. CLARKE.

**Manufacture of splinterless, reinforced glass.** H. LE V. LAWRENCE, and KENILWORTH MANUF. CO., LTD. (B.P. 293,666, 1.3.28).

## IX.—BUILDING MATERIALS.

**Mechanism of setting and hardening of cement.** T. MAEDA (Sci. Papers Inst. Phys. Chem. Res. Tokyo, 1928, 8, Suppl., 5–7).—Four processes are involved in the setting and hardening of cement. These are the dissolution of an unstable solid phase in a liquid phase, the formation of a solution supersaturated with regard to the stable solid phase, the separation of a new stable solid phase, in particles of colloidal dimensions, which may adsorb certain constituents of the liquid phase, and the dissolution of further quantities of the unstable solid phase in the solution saturated with regard to the stable phase. The tensile strength of set cement depends to a large extent on a film of solution adsorbed upon the precipitated particles of the stable solid phase.

R. TRUSZKOWSKI.

**Gypsum.** P. P. BUDNIKOV (Pit and Quarry, 1928, 16, 72–76).—Sulphuric, nitric, hydrochloric, and hydrofluoric acids are accelerators, whilst phosphoric, lactic, boric, formic, acetic, and citric acids are retarders of the setting of plaster of Paris. Salts of the univalent metals are positive catalysts; bivalent metal sulphates differ but slightly in catalytic activity. The mechanism of the effects is discussed.

CHEMICAL ABSTRACTS.

**Methods for obtaining high tensile strength of gypsum cement.** T. MAEDA (Sci. Papers Inst. Phys. Chem. Res. Tokyo, 1928, 8, Suppl., 8–9).—The tensile strength of gypsum cements is doubled by the addition of ethyl or methyl alcohol to the mixture of gypsum and water. Gypsum warmed to 120° in an oil-bath so that the resulting product has the composition  $\text{CaSO}_4 \cdot 0.24\text{H}_2\text{O}$ , yields a cement of greater tensile strength than without preheating, whilst a product of about the same water content obtained by heating to a higher temperature yields a product of inferior strength.

R. TRUSZKOWSKI.

## PATENTS.

**Manufacture of cement.** AMME-LUTHER WERKE BRAUNSCHWEIG DER "MIAG" MÜHLENBAU U. IND. A.-G. (B.P. 284,294, 27.1.28. Ger., 27.1.27).—Coloured (e.g., ferruginous) raw ground materials are mixed with suitable amounts of decolorising substances, such as borax, together with a flux (but excluding alkali or alkaline-earth chlorides), and then sintered, or melted and granulated, and further treated to form cement. By the further addition of a reducing agent, such as coke, to the raw materials, or by calcining in a reducing atmosphere, iron compounds are reduced and the mass is magnetically treated, after coarse and fine grinding, to remove the metal. The raw materials may be treated individually by the process before mixing.

A. COUSEN.

**Manufacture of cements rich in alumina.** A. HASSELBACH, Assr. to G. POLYSIUS (U.S.P. 1,677,842, 17.7.28. Appl., 27.9.24. Ger., 19.2.24).—The raw material, together with a small quantity of fluorspar, is burned in a rotary kiln at a temperature below the m.p. now used without fluorspar.

H. ROYAL-DAWSON.

**Rotary [cement etc.] kiln.** F. HURLBUTT (B.P. 293,331, 24.3.27).—The tapered blocks or bricks forming the lining of a rotary kiln for burning Portland cement, lime, soda, etc. are provided with a rebate along one of the edges of the wider end, and a corresponding projection at the opposite edge of the same end. The bricks thus lock together at the outside, and are prevented from falling into the kiln even when badly eaten away.

A. COUSEN.

**Cold bituminous paving composition.** O. H. BERGER, Assr. to W. P. McDONALD CONSTRUCTION CO. (U.S.P. 1,672,361, 5.6.28. Appl., 21.8.25).—A hard asphalt having a penetration not greater than 4 (46.1°, 100 g., 5 sec.) is heated with an equal quantity of a petroleum oil (e.g., topped crude oil) and mixed with the usual aggregates.

F. G. CLARKE.

**Mixing metallic salt [copper sulphate] with asphaltic compounds.** F. L. CARSON (U.S.P. 1,677,272, 17.7.28. Appl., 13.5.26).—Copper sulphate solution in excess is mixed with a small quantity of bitumen, and after evaporating off the water the mixture is incorporated with excess of hot bitumen.

H. ROYAL-DAWSON.

**Cementitious material of cellular texture.** G. B. HINTON (B.P. 294,041, 24.11.27).—See U.S.P. 1,657,716; B., 1928, 266.

**Preserving and protecting vegetable substances [wood].** L. P. CURTIN (B.P. 293,908, 19.4.27).—See U.S.P. 1,659,135; B., 1928, 299.

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

**Electric furnace cast iron.** C. E. WILLIAMS and C. E. SIMS (U.S. Bur. Mines Tech. Paper 418, 1928, 48 pp.).—Iron having about twice the strength of cupola iron can be made in the electric furnace. A tensile strength of more than 40,000 lb./sq. in. is common together with transverse strengths of 5000–6000 lb. on standard bars of grey iron. The advantages in



strength and toughness are due largely to the density and fine-grained structure of the metal, which, although whiter than cupola iron of equal carbon content, is soft and is easily machined. Virtually any kind of ferrous scrap may be used in the electric furnace, and when producing synthetic cast iron from steel scrap the power consumed ranges from 500 to 700 kw.-hours per ton, little variation occurring between the basic and acid processes. Considerable value attaches to being able to maintain the molten metal in the furnace until proper adjustments in composition are made and the metal is cast accurately as desired.

C. A. KING.

**Cementation of steels by special manganese alloys.** J. COURNOT (Compt. rend., 1928, 187, 298—300).—The characteristics of the cementation obtained from hard, semi-hard, and soft steels with ordinary spiegel, ferromanganese (76.19% Mn, 6.52% C), and ferromangano-silico-aluminium (19.25% Mn, 20.64% Si, 10.34% Al) are described, and hardness values are given. The best cementations were obtained when about 2.5% each of ammonium chloride and of alumina were mixed with the cement, and the heating carried out for 4 hrs. at 900—1050°, but the penetration was progressively less marked for successive cementations. Soft steel cemented with ferromangano-silico-aluminium is readily attacked by concentrated hydrochloric acid and dilute (10%) mineral acids, but only slightly by concentrated sulphuric and nitric acids.

J. GRANT.

**Creep in five steels at different temperatures.** H. J. FRENCH, H. C. CROSS, and A. A. PETERSON (U.S. Bur. Mines, Tech. Papers No. 362, 1928, 22, 235—267; cf. B., 1926, 57).—Creep tests were made on a low-carbon structural steel, a high-chromium steel, a chromium-molybdenum structural steel, high-speed steel, and a high-chromium, high-nickel austenitic steel, and the results are summarised in creep charts in which are shown the relations between stress, temperature, elongation, and time for each steel at temperatures ranging from 20° to 730°. By means of these charts the approximate determination of the stresses which permit life of different durations with different total elongations may be determined. In the temperature range within which strain-hardening takes place, the relation is shown between temperature and the stresses producing different initial elongations which are followed by a practical cessation of creep. At temperatures above those at which strain-hardening occurs the relation is shown between temperature and the per cent. elongation in 1000 hrs. Correlation of creep tests with short-time tension tests showed that, when using accurate equipment, the proportional limit was in the range of stresses which could be sustained for long periods with small amounts of deformation; as the temperature was raised, however, the proportional limit showed a tendency to become higher than the allowable creep. Steels which withstand the highest stresses at atmospheric or slightly elevated temperatures do not necessarily show superiority at higher temperatures. The load-carrying ability begins to drop rapidly at 315—425°. Between 595° and 730° the high-speed steel and the high-chromium, high-nickel steel gave the best results, whilst the chromium-molybdenum steel gave the

poorest; this is probably due to the fact that it was not structurally stable at 650°, oxidation being accompanied by decarburisation and grain growth. The best resistance to oxidation in air was shown by the high-chromium, high-nickel steel and the high-chromium steel.

M. E. NOTTAGE.

**Internal structure of chromium steels.** E. MAURER and H. NIENHAUS (Stahl u. Eisen, 1928, 48, 996—1005).—The work of earlier investigators on the composition of residues obtained by dissolving chromium steels in acids or by electrolysis is summarised and critically reviewed; it is concluded that the existence of the carbides  $\text{Cr}_3\text{C}_2$ ,  $\text{Cr}_4\text{C}_2$ , and  $\text{Cr}_5\text{C}_2$  is definitely established, but that  $\text{Cr}_4\text{C}$  is doubtful. Measurements of the electrical resistance of two series of chromium steels provided evidence of the presence of chromium carbides but gave no indication of their composition. The transformation points in a series of 22 chromium steels containing up to 35% Cr have been determined by thermal and dilatometric analysis and by magnetic measurements. The limits of the  $\gamma$  field at high temperatures are 17% Cr with 0% C, 25% Cr with 0.55% C, and 28% Cr with 1% C. In alloys with 0.55% C the Ac1 and Ac2 lines intersect at 2% Cr and in alloys with 1% C at 2.5% Cr. The slope of the Ac1 line is steeper the higher the carbon content for alloys containing up to 10% Cr. The Ac2 line falls in a smooth curve from 770° with 10% Cr to 600° with 32% Cr in alloys with 0.55% C, and to 600° with 29% Cr in alloys with 1% C. No indications of transformations due to chromium carbides were found.

A. R. POWELL.

**Self-hardening property of chromium steel.** I. OBINATA (Ryojun Coll. of Eng., 1928, 1, 145—166).—This investigation deals with the thermal data and microstructure of a series of chromium steels (Cr 7—18%) in which the ratio Cr/C varied from 50 to 75. In the annealed condition, the Ac1 carbide change point was raised and the Ar1 point was lowered by the addition of chromium. The temperature of the A2 magnetic change point was lowered by about 7° for each 1% Cr added, and was not affected by annealing or quenching. In the annealed specimens the stable structure, which was only attainable with a very slow rate of cooling, was pearlitic, the two phases probably consisting of a solid solution of iron and chromium and a carbide of chromium containing a little chromium in solid solution, the temperature of the A1 point varying with the composition of the solid solution. Within certain ranges of temperature, the  $\gamma$ -phase exists throughout the whole series of quenched specimens. The Ac1 point was lower and the quantity of heat absorbed was less in the quenched than in the annealed specimens, the difference increasing with the quantity of chromium, and both being unaffected by the time of soaking. A theory of the self-hardening property based on the experimental results is discussed in detail. It is suggested that these steels contain a carbide of chromium of unknown formula which dissociates above the Ac1 point. The dissociation and re-formation of this carbide is comparatively rapid at high temperatures, but the temperature coefficient of the reaction velocity is so great that the carbide cannot re-form below the normal A1 point; hence it may be

suppressed by quenching. The depression of the carbide change point on cooling which imparts the self-hardening property is due to the increase in viscosity of the steel produced by the addition of chromium which retards the deposition of the re-formed carbide from the supersaturated solid solution. M. E. NOTTAGE.

**Separation of molybdenum from vanadium in steel.** J. KASSLER (*Z. anal. Chem.*, 1928, 74, 276—289).—The method depends on the fact that in the presence of ferrous chloride vanadium is precipitated completely with the iron by the addition of an excess of sodium hydroxide, whereas the molybdenum dissolves as sodium molybdate. For steels with less than 3% Mo 4 g. are dissolved in hydrochloric acid and potassium chlorate; the solution is boiled to expel chlorine, diluted, filtered, treated with 25 c.c. of 20% sodium sulphite solution to reduce part of the iron to ferrous chloride, and poured in a thin stream into 120 c.c. of hot 24% sodium hydroxide solution. An aliquot part is filtered and the molybdenum determined as lead molybdate in the usual way. For steels with more than 3% Mo 1 g. is dissolved in 50 c.c. of hydrochloric acid (*d* 1.12) and 25 c.c. of 5% potassium chlorate solution and the solution is evaporated to a syrup. After dilution and filtration, 3 g. of ferrous chloride are added and precipitation of the iron is effected as before. Tungsten interferes in both procedures. A. R. POWELL.

**Corrosion by acids and rusting of iron containing copper.** S. S. STEINBERG (*Ann. Inst. Polyt. Ural*, 1927, 6, 73—82).—Tests were made with sheets containing from 0.13 to 1.21% Cu and with 10 and 20% solutions of sulphuric, hydrochloric, and nitric acids. Iron with 0.25—5% Cu dissolves in sulphuric acid solutions 8—10 times as slowly as ordinary iron, and in hydrochloric acid solutions twice as slowly. These effects are regarded as due to the fact that the acid dissolves the iron first, so that the copper becomes concentrated in an atomic-disperse state in the surface layers of the metal. Removal of the surface layer is followed by rapid initial attack of the iron by the acid, the metal gradually increasing in its resistivity. Experiments on rusting in air gave widely varying results, but the mean figures give for the ratio between the resistances of iron containing and free from copper the value 1.5:1. With metal immersed in water or in 1% sodium chloride solution the presence of copper does not increase the resistance. When plates of copper-free and copper-containing iron are immersed in water after removal of the scale by treatment with dilute sulphuric acid, the potential of the former is about 0.020 volt lower than that of the latter. If iron containing copper is heated in oxidising gases, copper is observed beneath the surface scale, the iron being oxidised in preference to the copper. T. H. POPE.

**Influence of the mould and the degree of deoxidation on the crystallisation of [steel] ingots cooled without disturbance.** F. BADENHEUER (*Stahl u. Eisen*, 1928, 48, 713—718, 762—766).—The shape of the mould used in making chill castings of steel has much more effect on the soundness of the ingot than has the thickness of the walls. The tendency to the formation of secondary pipes is reduced by using a mould tapering

towards the top end or by casting with a large waste-head. In the centre of all cylindrical castings there is always a more or less extensive zone with a characteristic globular structure. This zone extends practically throughout the ingot when a small quantity of deoxidiser is added to the steel just prior to casting, but with larger amounts of deoxidiser, especially with aluminium, the zone becomes more confined. Just below its apex V-shaped segregations of phosphides, sulphides, and non-metallic impurities are generally visible. The remainder of the ingot has a dendritic structure with a definite crystallite orientation; in this zone normal segregation of impurities takes place, whereas in the globular zone, inverse segregation is usual. These phenomena are attributed to variations in the gas content of the steel due to the use of deoxidisers. A. R. POWELL.

**Influence of phosphorus on strength properties of cast iron pipe.** J. T. MACKENZIE (*J. Amer. Water Works' Assoc.*, 1928, 19, 747—759).—Up to 0.8% P improves the quality of cast iron pipe by increasing the fluidity of the molten metal, but beyond 1% its stiffening and embrittling effects outweigh this advantage, and strength and resilience decrease rapidly. C. JEPSON.

**Effect of temperature on the solubility of iron oxide in iron.** C. H. HERTY, JUN., and J. M. GAINES, JUN. (*Amer. Inst. Min. Met. Eng. Tech. Bull.*, 1928, No. 88, 13 pp.).—At 1600°, the normal open-hearth tapping temperature, the solubility of ferrous oxide in iron is 1.36%, at 1900° 2.02%. Thus the oxygen is present in molten iron as a compound, and not as a gas; the constancy of the distribution coefficient between the metal and slag (ferrous oxide, with or without lime) indicates that this compound is ferrous oxide. The rate at which carbon disappears in open-hearth elimination depends on the rate of diffusion of ferrous oxide from the slag into the metal, and this depends on the saturation value of ferrous oxide under the slag in question.

#### CHEMICAL ABSTRACTS.

**Behaviour and necessary properties of dolomite for steel works.** O. JACOBS (*Stahl u. Eisen*, 1928, 48, 993—995).—For the lining of basic converters for the Thomas process the dolomite used should be free from carbon dioxide, and should be so burnt that the particles are sufficiently porous to absorb completely the tar oils from the binder during the operation of lining the furnace. A dolomite with 5—8% SiO<sub>2</sub> + Fe<sub>2</sub>O<sub>3</sub> + Al<sub>2</sub>O<sub>3</sub> after burning is more suitable than a purer product as its sintering temperature is lower, and therefore an efficient agglomeration of the grains takes place during the heating of the first charge. Other things being equal, an amorphous dolomite gives better results than a crystalline variety. The lime used for slagging the phosphorus in the iron should be as free as possible from magnesia, which produces a viscous slag and causes a more rapid wear of the lining. For the basic open-hearth furnace a dolomite low in silica and high in magnesia gives the best results in the fire-bridge, and for the hearth better results are obtained with burnt dolomite containing 28—40% MgO, 3—7% SiO<sub>2</sub>, and 4—8% Fe<sub>2</sub>O<sub>3</sub> + Al<sub>2</sub>O<sub>3</sub>. The hearth should be tempered with a slag rich in fluxes previous to charging in the iron. Alternatively,

the top layer of the hearth may be made of a sintered mixture of dolomite and old slag in the ratio 4 : 1.

A. R. POWELL.

**Iron carbonyl and carbonyl iron.** A. MITTASCH (Z. angew. Chem., 1928, 41, 827—833).—Iron pentacarbonyl is produced on the large scale by circulating carbon monoxide under pressure over sponge iron at 150—200° and cooling the gases evolved. It has  $d_4^{20}$  1.453, a surface tension of 22 dynes/cm. and a viscosity of 0.0075 c.g.s. unit at 20°. Its heat of formation (liquid) is 54.2 kg.-cal., latent heat of fusion 3.25 kg.-cal./mol., and heat of combustion 384.5 kg.-cal./mol. In alkaline solution it behaves as a powerful reducing and dechlorinating agent for organic compounds. In pentane solution it combines with bromine forming a yellow unstable compound which rapidly decomposes with the evolution of carbon monoxide and the formation of the reddish-brown compound,  $\text{Fe}(\text{CO})_4\text{Br}_2$ . Iron pentacarbonyl decomposes at 250° in a hollow vessel heated by radiation yielding a finely-divided iron containing about 1% C. If this is melted in a vacuum high-frequency furnace together with the requisite quantity of pure iron oxide, obtained by combustion of the carbonyl, an exceedingly pure iron of high permeability, low hysteresis, and small wattage loss is obtained.

A. R. POWELL.

**Determination of zinc in presence of large percentages of arsenic.** L. R. RAYMOND (Chemist-Analyst, 1928, 17, 6).—The ore (0.5 g.) is heated with sodium sulphate (2 g.), concentrated sulphuric acid (7—10 c.c.), and a small piece of filter paper until all the acid has evaporated and the paper is completely oxidised. The cool mass is disintegrated with warm water (10 c.c.) and boiled to a paste with concentrated hydrochloric acid (20 c.c.); a second evaporation with hydrochloric acid may be necessary to volatilise all the arsenic. Finally concentrated hydrochloric acid (10 c.c.) and ammonium chloride (10 g.) are added, the mixture is heated and diluted to 150 c.c., excess of ammonia and 10 c.c. of hydrogen peroxide are added, the liquid is filtered, and the ferric hydroxide washed with hot ammonium chloride solution; if much iron is present the hydroxide is redissolved and reprecipitated. Hydrochloric acid (7—8 c.c. excess) is added to the filtrate, which is diluted to 400 c.c., copper is precipitated with lead, and the zinc titrated with ferrocyanide.

CHEMICAL ABSTRACTS.

**Determination of nickel with  $\alpha$ -benzildioxime in presence of chromium compounds.** F. G. GERMUTH (Chemist-Analyst, 1928, 17, 3, 7).—Contamination of the nickel precipitate by chromium is avoided by the addition of 0.25 g. of potassium hydrogen tartrate and 0.2 c.c. of 10% cupric ammonium chloride solution.

CHEMICAL ABSTRACTS.

**[Mechanical properties and crystal orientation of] copper and brass.** O. BAUER, VON GÖLER, and G. SACHS (Z. Metallk., 1928, 20, 202—208).—The tensile strength of hard-rolled  $\alpha$ - and  $\beta$ -brass increases almost linearly with the angle of the test-piece to the direction of rolling; with pure copper and a zinc-copper alloy (2% Zn) the tensile strength falls to a slight minimum when the test piece is taken at an angle of 45° to the direction of rolling. After annealing at 500° the con-

verse holds true; copper has maximum strength at an angle of 45° to the direction of rolling, and the strength of brass falls slightly as this angle increases from 0° to 90°. The elongation of rolled copper annealed for 30 min. at 500° is about 20% in directions parallel to and at right angles to the direction of rolling, but rises sharply to 75% in a direction midway between these. The impact strength (notched-bar test) of cold-worked copper and brass rises sharply with the temperature of annealing above 200° to a maximum at 450—600°, and then falls almost as abruptly in the case of  $\alpha$ -brasses, but rises slightly with ( $\alpha + \beta$ )-brasses. The impact strength of hard-worked copper-zinc alloys rises to a maximum with 85% Cu; after annealing at 600° for 2 hrs. the maximum impact strength is obtained with 85—70% Cu. Characteristic röntgenograms and photographs of fractured test-pieces of hard-worked and annealed copper and brass sheets are reproduced, together with photomicrographs of heat-treated brass.

A. R. POWELL.

**Gold-silver-copper alloys.** F. E. CARTER (Amer. Inst. Min. Met. Eng. Tech. Pub., 1928, No. 86, 17 pp.).—Physical properties are reported for alloys of gold with silver or copper; zinc is a useful deoxidiser for the 14-carat alloys. Increase of density on rolling indicates gas inclusions.

CHEMICAL ABSTRACTS.

**Platinum metals and their alloys.** F. E. CARTER (Amer. Inst. Min. Met. Eng. Tech. Pub., 1928, No. 70, 24 pp.).—The treatment of platinum ores is described and physical properties of platinum metals are tabulated. Applications of the metals are discussed.

CHEMICAL ABSTRACTS.

**Dissolution of aluminium and its alloys in acids.** X. WACHÉ (Rev. Mét., 1928, 25, 331—346).—The dissolution of commercial aluminium in hydrochloric acid occurs in two stages, a rapidly increasing rate of dissolution in the first stage being followed by a more or less steady rate after the "period of induction." Rise of temperature or increase of concentration of acid increases the rate of attack, though the more important factor is the quantity and distribution of iron and silicon in the metal. The more highly disseminated impurity causes more rapid dissolution of the alloy, and the effect of heat-treatment is reflected entirely by the greater or lesser homogeneity of the resultant alloy.

C. A. KING.

**Mechanical properties of aluminium alloys.** R. KARNOP and G. SACHS (Z. Physik, 1928, 49, 480—497).—Data are given for the tensile strength and hardness of single crystals of the following aluminium alloys under various conditions of mechanical deformation: Al 99%, Mg 0.5%; Al 95%, Cu 5%; Al 94%, Mg 0.5%, Cu 5%. The results are discussed with reference to the crystal structure of the specimens before and after deformation.

R. W. LUNT.

**Determination of lead as chromate in Babbit metal, and determination of copper by Walker and Whitman's method.** G. SCHESTAKOV (J. Chem. Ind. [Russia], 1927, 4, 907—908; Chem. Zentr., 1928, i, 1307—1308).—The alloy containing lead, copper, tin, and antimony is dissolved in aqua regia, citric acid is added, and the liquid is neutralised with ammonia; it is then acidified with acetic acid, boiled, and the lead is precipitated with potassium dichromate, the copper in

the filtrate being precipitated with hydrogen sulphide or dextrose.

A. A. ELDRIDGE.

**Resistivity of chromium-plated metals to the action of chemical reagents.** T. MURAKAMI (J. Soc. Chem. Ind. Japan, 1928, 31, 132—136).—Commercial chromium is ordinarily in the passive state, and is highly resistant to acids and alkalis, except hydrochloric acid. The new surface obtained by breaking the commercial metal is no longer resistant to acids. Moreover, passive chromium in contact with zinc, iron, or some other metals in acids becomes active and is not resistant to acids. The chromium film of chromium-plated metals is generally pin-holed, and the plated metals are easily dissolved by acids. The fresh chromium film is active and not resistant to acids, but after some time it becomes passive. The chromium film prepared by the method of Kato and the author (B., 1928, 576) is resistant to many reagents and gases. The length of time after plating and pin-holes in the chromium film are the two important factors for testing the resistivity of chromium-plated metals.

K. KASHIMA.

**Autogenous welding with coal gas.** A. MÜLLER and B. BIBUS (Gas- u. Wasserfach, 1928, 71, 566—569).—Coal gas with a calorific value of 4230 kg.-cal./m.<sup>3</sup> gives when burnt with oxygen a flame temperature of about 1980° compared with 3200° for the oxy-acetylene flame. The useful heat generated is about 3850 and 2600 kg.-cal./m.<sup>3</sup>, respectively, for acetylene and coal gas, and the oxygen consumption 1 and 0.45 m.<sup>3</sup>/m.<sup>3</sup>, but the cost of operation of the oxy-coal gas burner is only about  $\frac{1}{3}$ — $\frac{1}{2}$  that of the oxy-acetylene burner. On the other hand the oxy-acetylene flame with a standard burner will weld a sheet of metal 4—5 times as thick as that which can be welded by an oxy-coal gas flame from the same burner, so that for heavy work the oxy-acetylene flame is not only much more efficient but also more economical. For light work with aluminium or brass the cost of the oxy-coal gas burner is 5—15% less than that of the oxy-acetylene burner.

A. R. POWELL.

**Trend of flotation.** A. J. WEINIG and I. A. PALMER (Quart. Colorado School Mines, 1928, 23, [2], 1—90).—A second, revised edition giving a complete review of modern American practice in flotation with a bibliography up to January, 1928 (cf. B., 1927, 192).

A. R. POWELL.

**Danger of mercury and amalgam dental fillings.** A. STOCK (Z. angew. Chem., 1928, 41, 663—672).—The use of mercury in dental fillings (copper or silver amalgam) is especially deprecated as dangerous both to dentists and patients, as air containing 0.001 mg. of mercury per m.<sup>3</sup> is injurious to health.

C. IRWIN.

**Absorption of atmospheric moisture by molten metals.** P. KOSOVSKI (Messager russe ind. mét., 1927, 50—51).—With metals of low m.p. (e.g., tin), relatively large amounts of water may sometimes be found in the cavities formed by shrinking on cooling. With metals of high m.p. (e.g., iron or copper) hydrogen is formed and cracks may develop on rolling. The casting of iron or copper should be carried out in as dry an atmosphere as possible.

CHEMICAL ABSTRACTS.

**Dilatometry of light metals.** M. HAAS (Z. Metallk., 1928, 20, 283—285).—A description of technique.

**Tungstic oxide.** GERO and IREDELL.—See VII.

PATENTS.

**Manufacture of iron [castings].** M. G. WOODMAN, Assr. to D. G. FISHER and V. F. SHANTZ (U.S.P. 1,677,420, 17.7.28. Appl., 16.5.27).—Between 1 and 3 lb. of salicylic acid are added per ton of molten iron before casting.

F. G. CLARKE.

**Multiple-hearth calcining furnace.** R. O. PIKE (U.S.P. 1,674,919, 26.6.28. Appl., 19.11.23).—The combustion chamber is situated between two ore hearths, and is surrounded by an annular air chamber from which air is supplied to the combustion chamber through radial passages in the hearth above and through fuel burners.

B. M. VENABLES.

**Operation of open-hearth furnaces.** A. L. STEVENS, Assr. to A. L. STEVENS CORP. (U.S.P. 1,677,664, 17.7.28. Appl., 16.5.27).—An open-hearth furnace is operated with a neutral atmosphere in the furnace chamber at atmospheric pressure.

C. A. KING.

**Manufacture of steel in the Siemens-Martin furnace.** F. SIEMENS A.-G., R. DURRER, F. C. SIEMENS, and A. SPRENGER (B.P. 293,326, 24.12.26).—In order to produce a protective layer of gas free from hydrogen or hydrocarbons over the metal in the furnace after melting down, gas produced in a "tapping" producer from coke without the use of steam is introduced through ports under the gas mixing chamber, and is directed on to the surface of the metal. Various modifications in the design of the furnace chamber are indicated.

C. A. KING.

**Increasing the permeability of silicon steel.** J. C. KARCHER, Assr. to WESTERN ELECTRIC CO. INC. (U.S.P. 1,677,139, 17.7.28. Appl., 19.9.24).—The steel is heated to 1000—1250° and cooled at not more than 10° per min., reheated to 550—800°, and cooled at a rate slower than quenching in water but faster than quenching in air.

F. G. CLARKE.

**Producing permanent magnets.** L. E. HOWARD and A. D. POTTS, Assrs. to SIMONDS SAW & STEEL CO. (U.S.P. 1,677,674, 17.7.28. Appl., 9.10.26).—The magnet consists of tungsten steel containing 0.05 to 1% V.

H. ROYAL-DAWSON.

**Bonding metal.** F. C. MATHISON, Assr. to C. L. REDFIELD (U.S.P. 1,677,354, 17.7.28. Appl., 2.9.24).—An iron or steel body is coated with a deposit of copper, then with a thin film of tin, and finally with the lead-base bearing metal by the babbiting process.

H. ROYAL-DAWSON.

**Extraction of metals from ores.** P. R. BLAWIEY (B.P. 293,340, 2.4.27).—The ore, particularly cassiterite or other tin ore, after concentration is calcined in compressed heated air and, while still hot, is reduced wholly or partially to the metallic state by means of a reducing gas heated under pressure. The reduced ore is concentrated by flotation and then washed in molten metal, the operation being conducted in several stages at different temperatures to remove the bulk of the metal from the gangue. The remainder of the metal may be recovered from the residual material by causing the particles to slide down a channel-shaped electrode in an

electrolytic cell and then treating them with an acid or other solvent or a gas, such as chlorine.

M. E. NOTTAGE.

**Ore concentration.** B. R. TUNISON (U.S.P. 1,677,416, 17.7.28. Appl., 9.2.24).—An oily, sulphur-containing constituent extracted from crude petroleum by means of caustic alkali is used as froth-flotation agent.

F. G. CLARKE.

**Manufacture of highly porous, coherent lead aggregates.** I. G. FARBENIND. A.-G. (B.P. 270,763, 9.5.27. Ger., 7.5.26).—Porous aggregates of lead suitable for accumulator plates, or for filtering or adsorption purposes, are formed from a homogeneous alloy of lead and an alkali or alkaline-earth metal, or a mixture of lead and an organic compound, from which the added material is extracted by suitable methods. C. A. KING.

**Metal powder and method of producing it.** E. KRAMER, and HARTSTOFF-METALL A.-G. (HAMETAG) (B.P. 293,609, 11.10.27).—Metal powders or granules having a grain dimension of 0.5–0.01 mm. consist of granules of hollow or bowl shape. The powder as produced in this manner is particularly adapted for making small pressings, e.g., dynamo brushes, current collectors, etc.

C. A. KING.

**Removing gases from and cleaning the surfaces of metals in vacuums by electrical means.** J. VON BOSSE (B.P. 286,632, 12.4.27. Ger., 7.3.27).—The metals are used in the form of electrodes in an evacuated space and a current of a few hundred to a few thousand volts is applied.

M. E. NOTTAGE.

**Mercury condenser.** E. K. PETERS (U.S.P. 1,672,499, 5.6.28. Appl., 10.1.27).—The condenser comprises a tower packed with sponges, which rest upon perforated partitions held by a vertical, central support. The latter enables the entire packing to be withdrawn as a unit, after removal of the top of the tower. The mercury vapour is sucked downwards through the tower, and the sponges are sprayed with water.

F. G. CLARKE.

**Alloy.** A. C. G. GWYER and H. W. L. PHILLIPS, Assrs. to BRIT. ALUMINIUM Co., LTD. (U.S.P. 1,676,856, 18.7.28. Appl., 26.8.26. U.K., 19.7.24).—See B.P. 243,405; B., 1926, 132.

**Determining composition of copper and zinc alloys.** M. POLANYI and S. VON BOGDANDY (U.S.P. 1,676,709, 10.7.28. Appl., 25.2.27. Ger., 24.3.26).—See B.P. 268,306; B., 1927, 784.

**Mixing apparatus particularly for amalgamating or alloying metals.** B. D. WARNE and S. CALVER (B.P. 294,276, 21.4.27).

**Lacquering metals** (B.P. 283,162).—See XIII.

## XI.—ELECTROTECHNICS.

**Pressure electrolysis of water.** J. E. NOEGGERATH (Chem. Met. Eng., 1928, 35, 421–423).—The principal difficulty in pressure electrolysis, whereby compressors can be eliminated, is that of economically maintaining the gases in a sufficiently pure state. The author electrolyses potassium hydroxide solution in tubular cells with axial nickel cathodes working at 180 atm. A compensating line of large capacity is connected to both compartments, so that differences in pressure

between the two will not alter the electrolyte levels. Such cells may find employment for storage of energy, the hydrogen to be used in motors, or in locomotives on branch lines of electric systems. Dissociation requires lower voltage, and hence less power consumption with increasing pressure, and, in view of the simple construction of the cells, it is claimed that they can replace ordinary cells for the production of electrolytic hydrogen for chemical purposes.

C. IRWIN.

**Ageing phenomena in quartz mercury-vapour lamps.** W. MEYN (Z. wiss. Phot., 1927, 25, 345–353).

—Devitrification of the quartz plays only a secondary part in the ageing effect, for ageing takes place in lamps even where devitrification is absent. In studying the ageing effect the band spectrum of Stark and the continuous spectrum of Küch and Retschinsky are of minor importance, hence the line spectrum only was investigated, at wave-lengths less than 4000 Å. A Hilger quartz spectrograph was used, fitted with an arrangement which brought the whole spectrum sharply into focus on the same plate. The decrease of intensity with age was measured by adjusting the time of exposure till a given line showed a standard intensity. The fall of intensity is due not to the presence of foreign gases (e.g., the inert gases) which some have supposed can diffuse through the quartz at high temperature, but to a black deposit which appears on the inner surface of the quartz and consists of two parts, one of which can be removed by rubbing or by the action of acids, and the other penetrates to a depth of 0.1 mm. into the quartz itself. This latter layer may be due to the presence of very finely-divided mercury, or, more probably, to the separation of small particles of elementary silicon formed under the influence of the ultra-violet radiation and the high temperature.

S. J. GREGG.

## PATENTS.

**Magnetic cores.** STANDARD TELEPHONES AND CABLES, LTD., Asses. of G. W. ELMEN (B.P. 277,289, 12.4.27. U.S., 11.9.26).—Finely-divided particles of an alloy capable of having imparted to it an initial permeability greater than that of iron, and very sensitive to mechanical strains, are mixed, under pressure, with an insulating material capable of retaining its insulating and binding properties at annealing temperatures of at least 800°; the mixture is heat-treated at 450–800° to develop magnetic permeability. The heat treatment may precede the compression. The magnetic element may consist of an alloy of nickel and iron, and the insulating particles may be intermixed with a lubricant.

M. E. NOTTAGE.

**Preparing electrical resistances from ceramic material.** M. HAUSER (B.P. 293,652, 26.4.27).—To the ceramic material containing silicon or ferrosilicon as conductor with or without other metallic admixtures, substances containing boric acid in the free or combined state are added whereby the temperature coefficient of the resistance may be varied from weakly negative to strongly positive. The resistances are fired in a reducing or a non-oxidising atmosphere.

M. E. NOTTAGE.

**Lead aggregates** (B.P. 270,763). **Metal powder** (B.P. 293,609). **Cleaning metals** (B.P. 286,632).—See X.

## XII.—FATS; OILS; WAXES.

**Classification of fats.** I. W. HALDEN (Chem. Umschau, 1928, 35, 55—56).—The following classification of fats and oils is suggested: (i) From marine and cold-blooded ("poikilothermal") animals; iodine value above 100. (ii) From warm-blooded ("homoiothermal") animals (excluding marine animals); iodine value below 100. E. LEWKOWITSCH.

**Behaviour of fats and oils in ultra-violet light.** M. HAITINGER, H. JÖRG, and V. REICH (Z. angew. Chem., 1928, 41, 815—819).—The fluorescence colours under ultra-violet light of several oils and fats, pure and in solution, are recorded, with reference to the detection of adulterants in butter, lard, cacao butter, olive oil, etc. A characteristic zoned fluorescence is noted when filter paper partially immersed in oil or oil solution is saturated by capillary rise of the liquid. E. LEWKOWITSCH.

**Hexabromide of elæostearic acid.** K. H. BAUER and E. ROHRBACH (Chem. Umschau, 1928, 35, 53—54).—By bromination of  $\alpha$ -elæostearic acid in light petroleum solution in the presence of ultra-violet light, a solid hexabromide, m.p. 139—141°, was obtained and a dark-red oily mixture of substituted bromo-acids. E. LEWKOWITSCH.

**Oxidation processes of drying oils.** S. MERZBACHER (Chem. Umschau, 1928, 35, 173—176).—Among the products of lixiviation and saponification of linoxyn formic, propionic, and azelaic acids were identified, and a mixture of crude acids was obtained containing an acid (caprylic or pelargonic) of m.p. 10°. E. LEWKOWITSCH.

**Detergent experiments on cotton.** R. M. CHAPIN (Oil and Fat Ind., 1928, 5, 208—212).—The detergent power of soap solutions was tested on cotton soiled with ointments of lamp-black and lard, vaseline, and mineral oils (cf. B., 1928, 417 for cotton soiled with non-oily carbon black). Of pure sodium laurate, myristate, palmitate, stearate, and oleate, the last-named was most powerful up to 40°, being surpassed by palmitate at 60° and by stearate at 80°. Addition of sufficient fat solvent (e.g., carbon tetrachloride) enhanced the detergent power. In parallel with the author's results on emulsification, excess of fatty acids increased and excess of alkali diminished the detergent power. E. LEWKOWITSCH.

**Avocado [pericarp] oil.** G. S. JAMIESON, W. F. BAUGHMAN, and R. M. HANN (Oil and Fat Ind., 1928, 5, 202—207).—Oil obtained from the dehydrated pulp of the avocado fruit by expression or extraction is dark green by transmitted, and red by reflected light; it has  $d_{25}^{25}$  0.9132,  $n_D^{25}$  1.47, acid value 2.8, saponif. value 192.6, unsaponif. matter 1.6%, iodine value (Hanus) 94.4, acetyl value 9.2, Reichert-Meissl value 1.7, Polenske value 0.2, iodine value of unsaturated acids 101.2. Separation of the acids by the lead salt-ether method, and fractionation of the methyl esters, gave the composition of the oil as glycerides of: oleic 77.3, linoleic 10.8, myristic trace, palmitic 6.9, stearic 0.6%, and arachidic acids trace. The pulp (68—85% of the fruit) contains at least 70% of the oil, which can be partially bleached by fuller's earth, shows good keeping properties, and is suitable for the manufacture of hard soap. E. LEWKOWITSCH.

**Malvaceæ. III. Hibiscus manihot, L. J. PIERAERTS** (Mat. grasses, 1928, 20, 8138—8139).—The seed contains water 10.80, total ash 4.56, water-soluble ash 2.68, total nitrogen 3.51, crude protein 23.34, ether extract 13.42, crude fibre 24.95, pentosans 11.44, phosphorus pentoxide 1.07%; the alkalinity of the total and soluble ash is 24.88 and 60.64% (as potassium carbonate), respectively. The oil, extracted with light petroleum of b.p. <60°, had  $d_{20}^{20}$  0.9194, acid value 6, saponif. value 197, iodine value 103, Crismer value (99.6% alcohol) 62°,  $n_D^{23}$  1.4695, unsaponifiable residue 0.92%, Hehner value 95.80%; Halphen reaction positive. The Millian-Becchi reaction gives strong reduction with formation of a silver mirror.

CHEMICAL ABSTRACTS.

**Isomerism among unsaturated fatty acids.** EIBNER and others.—See XIII. **Maize oil.** KRIZKOVSKY.—See XVII. **Acids in butter fat.** VIRTANEN. **Starch in oily seed-kernels.** GRIEBEL.—See XIX.

PATENTS.

**Treatment of oils or fats or mixtures of the same or fatty acids for the production of sulphuric acid compounds.** E. C. R. MARKS. From CHEM. FABR. STOCKHAUSEN & Co. (B.P. 293,480, 6.4.27).—After sulphonation of the oils the valuable fatty acid sulphates are separated from the unattacked oil by treatment with a neutral inert solvent, e.g., trichloroethylene. Further, the oil etc. dissolved in such a solvent may be subjected to sulphonation.

E. LEWKOWITSCH.

**Preservation and transportation of oils and fats.** A. NYROP (B.P. 293,342, 4.4.27).—By treating raw oleaginous vegetable material in emulsifiers with water and a film-producing material, e.g., "albuminates" or carbohydrates, and subsequent spray-drying, a paste or powder is produced which can be conveniently transported, will not melt at comparatively high temperatures, and from which the oil can be recovered for use. E. LEWKOWITSCH.

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

**Accelerating the drying of paints and varnishes.** H. A. GARDNER (Amer. Paint and Varnish Manufs.' Assoc., Circ. 335, July, 1928, 592—594).—Preliminary details are given of experiments on the rapid drying of oil paints and varnishes when exposed to a carbon arc at 55—60°. The drying times were reduced to one third or less of those required in a dark room at the same temperature. Apparatus employing lamps suitable for drying newly painted or plastered walls is described. S. S. WOOLF.

**Inhibition of mildew on paints.** H. A. GARDNER (Amer. Paint and Varnish Manufs.' Assoc. Circ. 335, July, 1928, 595—597).—Mildew formation on paints exposed in climates of high humidity and temperature is inhibited by the use of zinc oxide primers and the introduction of tung oil spar varnish or resins into the finishing coat, a firm hard surface being desirable. S. S. WOOLF.

**Methods of testing suitability of paints, varnishes, and lacquers for aeronautical purposes.** E. W. J. MARDLES (J. Oil and Colour Chem. Assoc., 1928, 11,

230—253).—The use of paints etc. for protection of wood and metal parts of aircraft is discussed generally, and a number of results indicating the influence of weight of film on protection afforded are quoted. It is shown that two coats are considerably better than one, and three coats slightly better than two, whilst further coats do not increase the protective value to any appreciable extent, and might actually be deleterious. Methods are described for determining various film properties, *e.g.*, scratch hardness, adhesion (bending test), resistance to erosion, resistance to water, oil, and petrol, "dope" solvents, etc. (mechanical rubbing test), drying times, and gloss, and typical results are tabulated.

S. S. WOOLF.

**Protective coatings for duralumin and other aircraft alloys.** H. A. GARDNER (Amer. Paint & Varnish Manufrs.' Assoc., June, 1928, Circ. No. 330, 482—508).—Details of the exposure tests of various types of paint on duralumin etc. begun in 1926 (*cf.* B., 1927, 683) are recapitulated and the results of a recent and final inspection are tabulated. Panels giving superior service were those coated with a zinc chromate paint, an iron oxide-zinc chromate paint, a zinc chromate primer followed by high-grade aluminium paint, and a lampblack paint. Various protective treatments of these light-weight alloys (other than painting) are described.

S. S. WOOLF.

**Paints for metal.** H. A. GARDNER (Amer. Paint & Varnish Manufrs.' Assoc., June 1928, Circ. No. 331, 510—568).—A dissertation on protective paints for iron, steel, copper, and for galvanised, tinned, and other metal surfaces. The individual characteristics of the pigments employed for this purpose are discussed and the methods of painting and types of paints used on various metals are described. Extended consideration is given to special fields, *e.g.*, marine paints, gas-holder paints, pipe coatings, radiator paints, slushing compounds.

S. S. WOOLF.

**Painting tests on panels impregnated with zinc chloride or creosote.** H. A. GARDNER (Amer. Paint & Varnish Manufrs.' Assoc., June, 1928, Circ. No. 334, 588—598).—The results of exposure tests, begun in Sept., 1926 (*cf.* B., 1927, 585), on various white paints applied to the impregnated panels and examined on May 1, 1928, indicated that freshly creosoted wood may not be satisfactorily painted with white paints made on a linseed oil base, even when various types of primers (red lead, aluminium powder, zinc dust, tin- and aluminium-foil sheets) are applied before the white paint coating. It is, however, considered probable that weathering of the creosoted wood for a year or so will produce a surface that will satisfactorily take paint coatings. Generally satisfactory results were obtained on zinc chloride-impregnated panels, although some darkening is noted.

S. S. WOOLF.

**Zinc oxide paints.** C. P. VAN HOEK (Farben-Ztg., 1928, 33, 2789—2790).—Straight zinc oxide-linseed oil paints are considered by many users to be deficient in flowing out properties, opacity, adhesion, and elasticity. This is attributable in part to the flocculating tendency of the weakly positively charged pigment, badly flowing masses that retain considerable amounts

of oil being formed. The extra oil necessarily added leads to poor opacity, to poor adhesion on wood (owing to absorption of "free" oil), and to poor weather-resistance of such paints used as primings. Further, the formation of zinc soaps causes undue hardness and lack of elasticity of the film. The addition of 5—10% of stand oil in the grinding process corrects the above faults to some extent.

S. S. WOOLF.

**Significance in paint technology of isomerism among higher unsaturated fatty acids and glycerides.** A. EIBNER, L. WIDENMAYER, and E. SCHILD (Chem. Umschau, 1927, 34, 312—320).—The different types of isomerism possible in the higher unsaturated fatty acids and glycerides are tabulated, characteristic examples being quoted. The acid isolated from the non-drying parsley seed oil (petroselic acid) is shown to consist largely of  $\Delta^5$ -oleic acid. The shifting of the double linking nearer to the carboxyl end of the chain than in normal  $\Delta^8$ -oleic acid manifests itself in the solid condition of the  $\Delta^5$ -acid at ordinary temperatures. " $\gamma$ -Linolenic acid" isolated from primrose oil (*Enothera biennis*) is shown to be  $\Delta^{5:8:11}$ -linolenic acid, the geometrically isomeric  $\alpha$ - and  $\beta$ -acids both being  $\Delta^{8:11:14}$ -linolenic acids. " $\delta$ -Linolenic acid," the presence of which in primrose oil is also indicated, is provisionally accepted as the geometrical isomeride of the " $\gamma$ "-acid. The " $\gamma$ "- and " $\delta$ "-acids should, therefore, more correctly be denoted as  $\alpha$ - and  $\beta$ - $\Delta^{5:8:11}$ -linolenic acids, or, for brevity, symmetrical  $\alpha$ - and  $\beta$ -linolenic acids. These should theoretically be slower drying than the  $\Delta^{8:11:14}$ -acids, the double linking system being nearer to the carboxyl group, but, since linseed oil exceeds primrose oil in content of the respective linolenic acids, definite conclusions on this point cannot be drawn from the greater drying power of linseed oil. The elæostearic acids, many place-isomerides of which are possible, differ from the linolenic acids by virtue of possessing three conjugated double linkings with consequent increase in drying tendency, but the exact relationship between  $\alpha$ - and  $\beta$ -elæostearic acids is not yet established. The presence of dilinolenic-monolinoleic glyceride (previously demonstrated by Schmidinger in a Dutch linseed oil and by Davidson in a linseed oil of unknown origin) is established in samples of Bombay, Riga, and La Plate linseed oils in yields of 36.8, 32.7, and 24.3%, respectively. This glyceride is probably the chief drying principal in linseed oil. A new glyceride, *dilinolenic-mono-oleic glyceride*, m.p. 72—73.5°, was isolated, which may account for all the oleic acid in the oil, and dispose of the fallacious "non-drying of oleic acid in linseed oil" view. The known total content of linoleic acid in linseed oil is not accounted for, and dilinoleic-monolinolenic glyceride is probably present in addition to those mentioned above.

S. S. WOOLF.

**Preliminary report of Sub-committee of the Royal Academy on modern pigments and mediums.** Feb., 1928. (J. Oil and Colour Chem. Assoc., 1928, 11, 260—269).—A general enquiry into artists' raw materials and the problems met with in painting pictures in oils. The nature of the canvas used and methods of priming are discussed, suggested improvements being the introduction of ramie cloth

in place of linen and cotton canvases, and the substitution of gelatin size by viscose solution. The causes of cracking and lowering of tone of pictures are analysed, factors such as changes in refractive index, changes in colour, and tendency to crack of drying oils, use of unsuitable mediums in the undercoating, transparency and oil absorption of various pigments being considered. In solid under-painting pigments of low oil absorption should be used, since they are least affected by changes in the drying oil and are unlikely to cause cracking. The surface of pictures is best protected by applying a thin coating of a solution of beeswax in turpentine, allowing to dry for some weeks, and polishing. The quality of water-colour paper and the permanency of artists' pigments are briefly mentioned.

S. S. WOOLF.

**Use of oil shales in the preparation of ultramarine.** B. K. KLIMOV and V. A. LANIN (Trans. State Inst. Appl. Chem., Moscow, 1927, No. 5, 89—105).—The substitution of bituminous shales of the Leningrad area for infusorial earth and tar in the preparation of ultramarine was successful, and even permitted the employment of less sulphur. Experimental yields were normal, but factory results were not good. Suitable shales contained volatile matter 30—46.11, non-volatile organic matter 8—10.65, silica 21.51—26, lime 6—8.36, alumina 5—6.29, moisture 0—4.21%.

CHEMICAL ABSTRACTS.

**Large-scale titanium pigment production based on old laboratory process.** B. K. BROWN (Chem. Met. Eng., 1928, 35, 427—428).—The plant described has a capacity of 10 tons per day of titanium dioxide working the Blumenfeld process. Ilmenite is treated with sulphuric acid in insufficient quantity to convert all the titanium into sulphate, as the coating of sulphate on the particles in any case makes penetration difficult. The solid reaction mass is agitated with cold water and the unattacked ilmenite returned for further treatment. Metallic iron is added to reduce any ferric salts present, and as much ferrous sulphate as possible crystallised from the clarified solution. Titanium sulphate is hydrolysed from a concentrated solution by a method not indicated, and the titanium dioxide filtered in Sweetland presses. It is washed, dried, and calcined. The finished product contains less than 0.005% of total iron and the pigment made has an oil absorption of about 25. The dioxide can also be prepared as a colloidal dispersion containing 600 g./litre  $\text{TiO}_2$ .

C. IRWIN.

**Microscopy of body colours.** H. WAGNER and J. KESSELRING (Z. angew. Chem., 1928, 41, 833—837).—The use of the microscope in determining the nature of the constituents of natural and artificial pigments is illustrated by several examples. Various colloidal constituents may be identified by their adsorptive power for certain dyes; e.g., kaolin is coloured by basic dyes, colloidal silica by both acid and basic dyes, kieselsguhr by basic dyes, and hydrated alumina by acid dyes. Suitable dye solutions contain 1% of Brilliant Green, Acid Violet, or Orange II. Crystalline substances, such as fluorspar, calcite, or dolomite, are detected by the polariscope.

A. R. POWELL.

**Lacquer solvents.** H. A. GARDNER (Amer. Paint and Varnish Manufs.' Assoc., Circ. 335, July, 1928, 597—606).—Ethyl  $\alpha$ -hydroxyisobutyrate and synthetic ethyl lactate (as distinguished from the fermentation product) are recently developed solvents suitable for use in cellulose lacquers—nitrate and acetate. Their chemical and physical properties are detailed.

S. S. WOOLF.

**Resin from *Pinus silvestris*. II. Solid constituents.** B. ARBUZOV (J. Russ. Phys. Chem. Soc., 1928, 60, 707—719; cf. B., 1927, 971).—The crystalline solid, m.p. 118—130° (33—35% of the total), which separates from the raw resin on keeping has  $[\alpha]_D$ —102.23° to —60.47° in alcohol (in benzene about 20° less). The dispersion was almost constant,  $[\alpha]_F/[\alpha]_C=2.37$ —2.39, closely similar to that of the solid from the resin of *Pinus maritima*. Fractional crystallisation from acetone yielded a solid, m.p. 132—134°,  $[\alpha]_D$ —112.20° to —111.08°, which on keeping became yellow, and the rotation changed slowly, in benzene from —41.69° to +12.9° in 365 days, and in alcohol from —60.47° to +4.5° in 430 days, whilst the m.p. fell to 90°. This was due to atmospheric oxidation. The solid (colophony) obtained by distilling the liquid resin had  $[\alpha]_D$ +31.65° to —21.82° in benzene and 0° to —74.79° in alcohol, depending on the conditions of distillation and dispersion.

M. ZVEGINTZOV.

**Testing of sealing wax.** R. NITSCHÉ (Farben.-Ztg., 1928, 33, 2794—2796).—Sealing waxes are graded by examination of their combustibility, degree of non-adhesion to the seal, adhesion to paper, and "flow" and "drop" points (Ubbelohde). Standard methods of testing and specifications for three grades are detailed.

S. S. WOOLF.

**Survey of modern synthetic resins.** A. A. DRUMMOND (Trans. Inst. Rubber Ind., 1928, 4, 40—58).

**Sulphate-turpentine.** POSTOVSKI and PLJUSNIN.  
**Cellulose ester solvents.** WOLFF.—See V.

PATENTS.

**Lacquering of metal surfaces.** BRIT. THOMSON-HOUSTON CO., LTD., Assees. of L. E. BARRINGER (B.P. 283,162, 5.1.28. U.S., 5.1.27).—A glycerol-phthalic acid type resin is applied by the usual processes to a metal surface as a primer for subsequent cellulose lacquer coatings.

S. S. WOOLF.

**Preparation of [resinous] condensation products.** I. G. FARBENIND. A.-G., Assees. of K. SCHIRMACHER and A. VOSS (G.P. 446,999, 16.12.21).—Neutral resins, soluble in linseed oil, are obtained by the action of formaldehyde etc. in presence of zinc chloride etc. if desired, on the condensation products of benzyl chlorides with aromatic hydrocarbons or their derivatives. E.g., the oily product from benzyl chloride and xylene is boiled with paraformaldehyde and acetic acid in presence of zinc chloride to give a highly viscous oil suitable as a plasticiser for films. From naphthalene and xylyl chloride and formaldehyde, a resin giving light-fast lacquers is obtained. Other formaldehyde condensations described are those with benzylphenols (for a copal substitute in light-fast oil varnishes), with



“triresol”—chlorobenzyl chloride (for a thermo-hardening resin), with salicylic acid—benzyl chloride (for a shellac substitute), and with benzyl- $\beta$ -naphthol (resin for light-fast lacquers). C. HOLLINS.

**Production of condensation products from waste and wash-liquors and distillates of phenol-formaldehyde resin manufacture.** E. GLASER (Austr.P. 106,018, 27.5.21).—The waste liquors or distillates are treated below 60° with acids or acid salts to precipitate (after some days) a resin suitable for lacquers. C. HOLLINS.

**Resinous composition.** V. H. TURKINGTON, Assr. to BAKELITE CORP. (U.S.P. 1,677,417, 17.7.28. Appl., 7.7.27).—A non-phenolic resin is added to a phenol-fatty oil-methylene reaction product, whereby the separation of insoluble compounds is prevented. F. G. CLARKE.

**Manufacture of coloured masses or solutions thereof.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 293,067, 24.2.27).—Organic dyes, containing sulphonic or carboxylic groups, in the form of their amine salts (other than guanidine salts), are incorporated with cellulose esters or ethers, synthetic or natural resins, etc., or varnishes containing these. Examples of suitable amines are mono- and di-cyclohexylamines, piperidine, benzylaniline, Auramine base, Victoria Blue base, Rhodamine B base, Safranin base. C. HOLLINS.

**Printing colours or inks, and hectograph compositions, inking rollers, etc.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 292,655 and 292,894, 16.3.27).—Sorbitol, or one of its derivatives obtained by the action of heat or alkylating or acylating agents, is added (A) as a solubilising agent to printing pastes (including vat dye printing pastes), printing inks, hectograph inks, etc., (B) as a thickening agent to carriers for such printing pastes and inks, e.g., hectograph compositions, inking rollers, blanketing cloth, etc. For both purposes sorbitol acetate is particularly useful. C. HOLLINS.

**Manufacture of printing inks.** J. AZZOPARDI (B.P. 293,238, 29.9.27).—Printing inks of good consistency and adhesiveness and having sufficient penetration, drying capacity, etc. are produced from straight steam-still asphalt, a volatile mineral oil, and a pigment such as lampblack, with or without the addition of turpentine, rosin, and resinates. C. O. HARVEY.

**Condensation product of urea and formaldehyde.** A. GAMS and G. WIDMER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,676,543, 10.7.28. Appl., 20.5.26. Switz., 5.6.25).—See B.P. 253,094; B., 1926, 756.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Changes in rubber on ageing.** M. SAGAJLLO (Przemysl Chem., 1928, 12, 184—190).—A sample of aged rubber contained, in 1927, 22.8% of acetone-soluble substances as compared with 2.26% in 1925. The residue after extraction with acetone contained 6.0% of chloroform-soluble substances as against 2% originally, and the content of substances hydrolysed by alcoholic caustic soda in the residue after chloroform extraction had risen from 2 to 10%. R. TRUSZKOWSKI.

**Hydrogen-ion concentration [in rubber sera].** L. R. VAN DILLEN (Arch. Rubbercultuur, 1928, 12, 144—146; Med. Proefstat. Rubber Buitenzorg, 1928, No. 30, 84—86).—Experiments on the coagulation of latex with sodium fluosilicate indicate that there is a relation between the clarity of the serum and the  $p_H$  of the solution. A good coagulation without loss of rubber is obtained only between  $p_H$  4.5 and 4.4. This may be obtained by suitable dilution of the latex. A. R. POWELL.

**Colouring of cold-cured rubber.** W. E. SANDERSON (Trans. Inst. Rubber Ind., 1928, 4, 22—39).—See B., 1928, 493.

#### PATENTS.

**Manufacture of vulcanised rubber [accelerators].** W. P. TER HORST, Assr. to RUBBER SERVICE LABORATORIES CO. (U.S.P. 1,672,548, 5.6.28. Appl., 6.5.27).—Tetrasubstituted guanidines such as can be made by interaction of cyanogen chloride with secondary bases (piperidine, ethyl-*o*-toluidine) are used as vulcanisation accelerators. C. HOLLINS.

**Vulcanisation of rubber.** R. V. HEUSER, Assr. to A. C. BURRAGE (U.S.P. 1,677,235, 17.7.28. Appl., 5.6.28).—Rubber is heated with a vulcanising agent and a substituted guanidine containing an aryl group in each of the amino-groups and a *p*-substituted aryl group in the imino-group. B. FULLMAN.

**Manufacture of rubber and compounds thereof.** DUNLOP RUBBER CO., LTD., and D. F. TWISS (B.P. 293,502, 19.4.27).—Colophony and viscous liquid or adhesive semi-solid rubber accelerators and/or anti-agers and/or softening agents (e.g., aldehyde-anilines, oleic acid, etc.) are mixed in the molten or dissolved condition. The product, flakes or fragments of m.p. or softening point 80—150°, is incorporated with rubber. B. FULLMAN.

**Polymerised vinyl alcohol etc.** (U.S.P. 1,672,156).—See III.

#### XV.—LEATHER; GLUE.

**Chemistry of liming [hides].** G. D. McLAUGHLIN, J. H. HIGHBERGER, and E. K. MOORE (J. Amer. Leather Chem. Assoc., 1928, 23, 318—336).—Amines are formed prior to liming and small quantities are formed during the liming process. A number of commercial lime liquors contain small quantities of volatile amine, which have an effect on the unhairing. The presence of salt inhibited the action of the amine. Diamines, e.g., ethylenediamine, possess greater unhairing power than methylamine, but their action occurs in the initial stage of liming. Secondary and tertiary amines were not detected in commercial mellow limes. An increase in the concentration of primary amines in a lime liquor diminished the time of unhairing. Preliminary swelling in pure lime did not reduce the time of unhairing in a lime-amine liquor when skin was immersed in a lime-amine solution, and equilibrium was reached after 24 hrs. between the amine, lime solution, and the skin; 20% of the original amine present was found in the skin and 80% in the liquor. Lime was absorbed more rapidly in the presence of amine, probably due to the activation of the protein carboxyl group by the amine. D. WOODROFFE.

**Re-use of salt in the curing of animal skins.** G. D. McLAUGHLIN, I. H. BLANK, and G. E. ROCKWELL (J. Amer. Leather Chem. Assoc., 1928, 23, 300—318).—The re-use of salt in curing causes a 1—3% increase in the initial inoculation, and introduces organisms which are more resistant to salt and show proteolytic activity in presence of a higher percentage of salt.

D. WOODROFFE.

**Miscellaneous tannery investigations.** G. GRASSER (J. Coll. Agric. Hokkaido Imp. Univ., 1928, 20, 219—232).

—I. **Soluble methylene compounds of tannin anhydrides.** It is concluded that 2 mols. of tannin condense when heated, forming insoluble phlobaphen, which with formaldehyde yields soluble methylenephlobaphen; the latter with dilute sodium hydroxide solution forms a sodium derivative not precipitable by acid or gelatin, but with concentrated sodium hydroxide solution forms a sodium compound of phlobaphen, from which the original phlobaphen is liberated on acidification.

II. **Methylene derivatives of tannin.** Solubility relationships are recorded.

III. **Phloroglucinol reaction of tannin.** The extract is placed on filter paper impregnated with 2% vanillin solution and acidified with concentrated hydrochloric acid; in presence of phloroglucinol a cherry-red colour develops.

IV. **Behaviour of the albumin-globulin and of the coriine of the skin towards protein reagents.**

Positive reactions with albumin-globulin (1), coriine (2), and gelatin (3) were obtained as follows:—biuret 1, 2, 3; Molisch 3; sulphur reaction 1, 2; phenol precipitation 2, 3; mercuric chloride 1, 2, 3; tannin 1, 2, 3; potassium mercuric iodide 1; picric acid 2, 3.

V. **Rate of conversion of green into violet chromic salts.** Addition of sodium acetate or oxalate appears (visually) to promote the conversion of green into violet chromic sulphate solutions.

CHEMICAL ABSTRACTS.

**Combination chrome and vegetable tanning.** G. GRASSER and HIROSE (J. Coll. Agric. Hokkaido Imp. Univ., 1928, 20, 203—217).—In leather tanned 1 day with a mixture of basic chromic sulphate and oak-gall tannin, contraction on boiling increased with increasing proportion of the latter; in leather tanned 8 days the contraction for chromium:tannin  $\geq 1:1$  was the same as for chrome leather. Leathers tanned 1 day with the ratio 7:3 to 3.5:6.5 showed minimum acid swelling, but leathers tanned 8 days in liquors containing any chromium showed the slight swelling characteristic of chrome leather.

CHEMICAL ABSTRACTS.

**Colour-producing constituents of cacao bean.** ADAM.—See XIX.

PATENTS.

**Manufacture of tanning materials from sulphite-cellulose waste liquors.** E. J. ROSENZWEIG (F.P. 620,394, 16.7.26).—Sulphite-cellulose waste liquors free from iron are heated with alkali carbonates or hydroxides for a short time at not too high a temperature. The product may be acidified with sulphuric, hydrochloric, or acetic acid, or mixed with a synthetic tannin.

D. WOODROFFE.

**Manufacture of synthetic tannins.** SOC. CHIM. POUR L'IND. DU CUIR (F.P. 573,416—7, 1.2.23).—Aromatic sulphonic acids are condensed with (A) 15—40% of their weight of acetone, mesityl oxide, or phorone, or (B) 20—40% of their weight of lactic acid in presence of a dehydrating agent, e.g., 1—2% of cupric or mercuric sulphate. The products in each case are neutralised with an aliphatic or aromatic base. D. WOODROFFE.

**Dyeing leather.** A. J. CLERMONT (F.P. 614,460, 13.4.25).—Leather with a very greasy surface is treated with a lukewarm solution of soap, rinsed, mordanted with ammonia or soda, dyed, dried, staked, and finished as usual. Vegetable-tanned leather is preferably degreased with a fat solvent. D. WOODROFFE.

**Rendering leather soft and pliable.** E. DESPARMET and F. SCHMITT, Assees. of ASSOC. PARISIENNE POUR L'IND. CHIM. (F.P. 615,951, 1.10.25).—Neutral or acid esters of the higher fatty acids with aliphatic, aromatic, or terpene alcohols, alone or in mixtures, or dissolved in suitable solvents or with additions of substances which increase the absorption of the ester by the hide fibres, or of soluble dyestuffs or pigments, are used on leather instead of glycerin or castor oil. The most effective products are benzyl ricinoleate or butyl oleate in acetone solution. D. WOODROFFE.

**Waterproofing leather and composition therefor.**

E. D. VAN TASSEL, and VAN TASSEL Co. (B.P. 293,062, 31.1.27).—Paraffin wax is heated to 153° or higher with 5—30% of rubber-like gum, and the leather is immersed in the mixture at 90—100°.

D. WOODROFFE.

**Gluing process and preparation of glue therefor.**

C. KLINGSPOR (B.P. 293,232, 9.9.27).—A fluid condensate of phenol and formaldehyde is added at about 75° to a solution of glue in water or to the water in which glue is steeped. The glued articles are gradually heated to a temperature not above 200°, advantageously 145°, during 10—36 hrs.

D. WOODROFFE.

**Leather cement.** I. G. FARBENIND. A.-G., Assees. of F. FUNCKE (G.P. 444,387, 4.8.25).—Methylene chloride is added to an acetone solution of nitrocellulose containing a plasticiser.

D. WOODROFFE.

**Fibres from skins of fish** (B.P. 284,297).—See V.

## XVI.—AGRICULTURE.

**Influence of potash manuring with increasing nitrogen dressings on the yield and quality of barley in 1927.** H. WIESSMANN and E. SCHRAMM (Z. Pflanz. Düng., 1928, 7B, 314—322).—Potash manuring alone does not increase the protein content of barley, but a small increase results from an additional fairly heavy dressing of nitrogen. The "weight per thousand grains" was not affected by potash salts, but the conjoint use of nitrate tended slightly to increase the proportion of small grains at the expense of the large ones.

A. G. POLLARD.

**Comparison of the Neubauer seedling method and the Lemmermann citrate method [for determining the phosphate requirement of soils] with field trials at Aschersleben.** G. SCHULZ (Z. Pflanz. Düng., 1928, 7B, 297—301).—Extensive comparative experiments are recorded. Good general agreement is

shown among the three methods for cereals, but results diverge considerably with sugar beet. By increasing the limiting figure for phosphate in the Lemmermann process from 20 to 25 mg. slightly better agreement is shown.

A. G. POLLARD.

**Nitrogen fixation by micro-organisms.** A. RIPPEL and H. POSCHENRIEDER (J. Landw., 1928, 76, 101—112).—Examination of the energy relationships of nitrogen fixation in the nodules of legumes shows the process to be less economic than was hitherto supposed if the exothermic formation of ammonia from its elements is considered. Alternatively, since the necessary hydrogen can, in the circumstances, arise only from organic decomposition, the nitrogen fixation process may be considered from this point of view.

A. G. POLLARD.

**Tapioca. I. Sampling the standing crop. II. Coeruleomolybdate method for determination of phosphates.** V. R. GREENSTREET (Malayan Agric. J., 1928, 16, 59—69, 70—75).—II. The effect of certain constituents of the soil on the accuracy of the method is described. In silica-free solutions of tapioca tuber and oil-palm leaflet, the results obtained are in satisfactory agreement with those given by the gravimetric method, but with tapioca leaf and stem, pineapple ash, and soil, divergences of as much as 31% may occur. The use of the strongly acid "Kjeldahl" solution without removal of silica invariably leads to low results.

F. R. ENNOS.

**Relative solubility of phosphoric acid and potassium in German and tropical soils.** H. VAGELER (Z. Pflanz. Düng., 1928, 11A, 89—93).—Determinations of phosphoric acid and potassium soluble in 10% hydrochloric acid and taken up by plants in the Neubauer method were made on series of German and Rio Magdalena soils. In the case of the German soils there was no parallel between the acid-soluble and Neubauer potash, but the two methods gave comparable results in a number of the tropical soils. In contrast to the potash, tropical soils were much lower in acid-soluble phosphate than the German soils.

H. J. G. HINES.

**Solubility of rock phosphates as affected by plant roots.** M. DOMONTOVITSCH and A. SCHESTAKOV (Z. Pflanz. Düng., 1928, 11A, 108—112).—Oats and millet grown in sand cultures supplied with phosphorite as a source of phosphoric acid made little growth. When buckwheat and lupins were grown in the same pots the growth of the cereals was much improved, particularly in the case of lupins. At the same time the  $p_H$  value of the soil solution was increased slightly by both buckwheat and lupins, resulting in increased solubility of the phosphate.

H. J. G. HINES.

**Uptake and utilisation of plant nutrients.** W. U. BEHRENS (Z. Pflanz. Düng., 1928, 11A, 93—107).—Vegetation experiments with oats were made in sand cultures. The effects of supplying nutrients by continuously percolating solutions were compared with those obtained by supplying full amounts of nutrient initially. It was shown that the uptake from solutions containing only 2.2 mg./litre of nitrogen and 0.65 mg./litre of phosphoric acid was as good as from stronger solutions. In discussing the evaluation of insoluble phosphates in

the light of these experiments, stress is laid on the period at which the nutrients become available and are taken up by the plant.

H. J. G. HINES.

**Mathematical expression of the action of nutrients.** W. U. BEHRENS (Z. Pflanz. Düng., 1928, 11A, 150—155).—The Mitscherlich equation is slightly modified and extended.

H. J. G. HINES.

**Effects of manurial treatments on the chemical composition of gooseberry bushes. I. Effects on dry matter, ash, and ash constituents of leaves and stems of terminal shoots and of fruits, and on total nitrogen of fruits.** T. WALLACE (J. Pomology, 1928, 7, 130—145).—A deficiency in soil potash influenced the growth and composition of gooseberry bushes, and is characterised by (a) high dry matter content of the leaves and stems of terminal shoots, (b) low ash content in the dry matter of stems and berries, and (c) high proportion of calcium, magnesium, sodium, and phosphorus in the ash of stems, leaves, and berries. Variations in the lime content of the ash resulting from a potash deficiency are greatest in the leaves and smallest in the berries. The reverse is the case with the phosphate content of the ash. The effect of a deficiency of a second nutrient element on the ash analysis is masked by a deficiency of potash.

A. G. POLLARD.

**"Spontaneous" ignition in stable manure.** L. H. JAMES, G. L. BIDWELL, and R. S. MCKINNEY (J. Agric. Res., 1928, 36, 481—485).—A case of "spontaneous" combustion of a pile of heating stable manure is reported. Oxygen aeration of a small section of the heating material produced a rise of temperature of 26.5° in 30 min., thus affording evidence in agreement with the theory that spontaneous ignition is the result of rapid oxidation.

E. A. LUNT.

**Toxic value of fluosilicates and arsenicals as tested on the plum curculio.** O. I. SNAPP (J. Econ. Entomol., 1928, 21, 175—178).—The order of diminishing toxicity is: sodium fluosilicate; arsenates; lead, barium, calcium, zinc, magnesium, manganese, aluminium; sodium fluosilicate and lime; scorodite. Sodium fluosilicate is toxic to peach foliage and fruit.

CHEMICAL ABSTRACTS.

**Making mechanical analyses of soils in fifteen minutes.** G. J. BOUYOUOS (Soil Sci., 1928, 25, 473—480).—If the particles of the soil are grouped into three main groups, viz., combined sand, silt, and clay or colloids, these groups can be determined in 15 min. by the hydrometer method.

H. J. G. HINES.

**Pentosan content in relation to winter hardiness in the apple.** W. A. DELONG (Sci. Agric., 1928, 8, 501—523).—In general, the least hardy varieties of apples have the greatest pentosan content. The pentosan content is not correlated with the total loss or rate of loss of moisture at 75° in either aerial or subterranean portions of the tree. The discrepancy between results obtained by the hydrochloric acid-phloroglucinol and fermentation-copper reduction methods is not due to substances precipitable by phloroglucinol and soluble in 95% ethyl alcohol at 60°. The small fraction of the phloroglucide precipitate soluble in alcohol is probably derived from hexoses.

CHEMICAL ABSTRACTS.

**Saidel's method for the investigation of aqueous soil solutions.** E. ANGELESCU (Bul. Soc. chim. România, 1928, 10, 14—25; cf. Saidel, IV<sup>me</sup> Conf. de Pedologie, Rome, 1925, 2, 556).—Polemical against Saidel, whose method differs from that of Wolf (Landw. Jahrb., 1873, 2, 391) only in the manner of calculation, which takes no account of substances remaining from a previous extraction. C. HOLLINS.

**Sulphur content of Alberta soils.** F. A. WYATT and J. L. DOUGHTY (Sci. Agric., 1928, 8, 549—555).—In semi-arid soils the sulphur content is often greater than the phosphorus content, and increases with depth. Concentration zones of soluble salts are formed 3—6 ft. below the surface; the zone of sulphate concentration is usually below the lime layer. The result of leaching is shown better by the depth of the zone of calcium carbonate accumulation than by that of sulphate concentration. Alberta alkali soils are described.

## CHEMICAL ABSTRACTS.

**Determination of nitrogen in fertilisers.** THOREL (Ann. Falsif., 1928, 21, 257—261).—For fertilisers containing nitric nitrogen, the aqueous extract of 4 g. is diluted to 200 c.c., and nitric nitrogen determined on 20 c.c. by Schloesing's method. Potassium is determined in another 25 c.c., and in the absence of urea or its soluble compounds ammoniacal nitrogen is determined on 50 c.c. by means of magnesium. The insoluble residue from the extract is submitted to the Kjeldahl treatment, and the ammoniacal nitrogen present found by distillation in the Schloesing-Aubin apparatus. The organic nitrogen also is thus obtained. Total nitrogen is determined on 1 g. by a Kjeldahl distillation, with a preliminary destruction of nitric nitrogen by means of ferrous chloride and hydrochloric acid, and the organic nitrogen deducted from this gives the ammoniacal nitrogen. If nitric nitrogen is absent, the same procedure is followed, omitting the treatment with ferrous chloride. D. G. HEWER.

**Determination of nicotine in sprays.** R. SCHERPE (Zentr. Bakt. Par., 1927, II, 71, 93—105).—The fluid (6 c.c.) is mixed with saturated lead acetate solution (0.6 c.c.) and powdered sodium sulphate (0.32 g.), the mixture filtered into a graduated cylinder, and the residue washed with 1% sodium sulphate solution. Silicotungstic acid (15% solution, 3 c.c.) is added, the liquid diluted to 20 c.c., and kept for 24 hrs.; any surface precipitate is shaken down and the height of the column  $h$  is read after 2 hrs. If  $h > 2$  cm.,  $h \times 0.70 =$  mg. of nicotine; if  $h < 2$  cm.,  $h \times 0.83 =$  mg. of nicotine. CHEMICAL ABSTRACTS.

**Soils of Bowie, Denton, Freestone, and Red River Counties.** G. S. FRAPS (Texas Agric. Exp. Sta. Bull., 1927, No. 375, 48 pp.).

## PATENTS.

**Producing soluble phosphate fertilisers.** F. G. LILJENROTH (B.P. 282,619, 20.4.27. Sweden, 23.12.26).—Crude calcium phosphate is leached with a solution containing sulphuric acid and ammonium sulphate, the latter obtained from the precipitated calcium sulphate by treatment with ammonia and carbon dioxide. The ratio of acid to sulphate in the solution is 7 : 2 by weight.

The product is an ammonium phosphate possibly containing some sulphate. B. FULLMAN.

**Manufacture of insecticides for spraying plants and pickling seeds.** FARBENFABR. FORM. F. BAYER & Co. (F.B. 610,919, 27.11.25. Ger., 3.2.25).—Mercuriated organic compounds are treated with alkali in the form of alkali phenoxides, borax, etc. to give soluble insecticides which are applied in solution with salt, ferrocyanide, or other addenda. Suitable mercuriated compounds are anhydronitro(hydroxymercuri)-phenol or -cresols, chloro(sulphatomercuri)phenol, aceto-mercuriphenol, cyanomercuricresol, anhydrohydroxy-mercuriphenol, mercuribenzoic acid, chloro(mercuri)-naphthols, etc. C. HOLLINS.

**Eradication of plant pests.** I. G. FARBENIND. A.-G. (F.P. 615,634, 5.5.26. Ger., 15.5.25).—Aqueous ammonia, alone or in conjunction with other parasite-destroying agents, is used in such amount as will combine with the humic acid of the soil and thus lose its injurious action on the plants themselves. The quantity recommended is 1000—1500 kg. of 0.5—1.0% aqueous ammonia for each hectare of soil infested with parasites. C. HOLLINS.

**[Apparatus for] drying crops.** J. CAMPBELL (B.P. 294,383, 3.8.27).

## XVII.—SUGARS; STARCHES; GUMS.

**Working white sugar masseccutes [in the beet sugar factory].** F. NOSEK (Z. Zuckerind. Czechoslov., 1928, 52, 457—461).—Microscopical observation of the centrifuging of granulated masseccutes shows the mother-liquor frequently to contain such an amount of extremely fine grain as to impart to it a milky appearance. On turning on cold or particularly hot water for covering, after the adhering syrup has been washed out, the crystal begins to be attacked. Steam has a similar effect, though less severe than with hot water, but it also causes the formation of fissures in the wall of sugar in the centrifugal through which the condensed water penetrates, causing further etching of the crystal. J. P. OGILVIE.

**Chemical theory of [sugar] saturation.** L. O. SHNAIDMAN (Nauch. Zapiski, 1928, 5, 273—278).—The alkalinity and polarisation of a solution of sugar precipitated from a sugar-lime solution by carbon dioxide, and treated with a current of air free from carbon dioxide, depend on the time elapsing before analysis. In the first stage of saturation a colloidal solution of lime forms; with decrease in alkalinity this begins to gelatinise. The amorphous calcium carbonate adsorbs both sucrose and the calcium oxide (1:1) compound; as the amorphous substance crystallises the absorption decreases. CHEMICAL ABSTRACTS.

**Crystallisation of second-product [sugar] fill-mass.** B. E. KRASILSCHIKOV (Nauch. Zapiski, 1928, 5, 309—312).—With a fill-mass of 93.63—93.17° Brix, crystallisation progressed with cooling; only below 45° did the coefficient of supersaturation slightly increase. With a fill-mass of 94.55—94.09° Brix, and with more rapid cooling, the coefficient of supersaturation immediately increased and remained high.

CHEMICAL ABSTRACTS.

**Washing the filter-press mud [in sugar manufacture].** B. E. KRASILSCHIKOV (Nauch. Zapiski, 1928, 5, 287—290).—Cold water removes more sugar than ammoniacal hot water, which removes a greater proportion of solids. CHEMICAL ABSTRACTS.

**Undetermined sugar losses in the process of defeco-saturation.** L. O. SHNAIDMAN (Nauch. Zapiski, 1928, 5, 322—328).—The loss of sugar is caused by the absorptive power of the lime; its magnitude depends on the amount of lime precipitated in the saturation process, the lime added in the defecation process, the concentration of sucrose, the temperature, and the reaction of the saturated juices. The absorbed substances normally contain sugar representing a loss of 0.7—0.9%. CHEMICAL ABSTRACTS.

**Acids of maple syrup.** E. K. NELSON (J. Amer. Chem. Soc., 1928, 50, 2006—2008).—These are present mainly as salts. They have been isolated from samples from two different localities by acidification followed by (a) ether extraction, (b) removal of volatile acids in a vacuum, and (c) precipitation as lead salts. Malic acid predominates (approx. 0.1% of syrup), with formic, acetic, and citric acids (approx. 0.01% of each) and traces of fumaric, succinic, and an unknown acid of high m.p. Tartaric and tricarballic acids were not detected (cf. von Lippmann, A., 1915, i, 362). H. E. F. NOTTON.

**Acids of maple sugar "sand."** E. K. NELSON (J. Amer. Chem. Soc., 1928, 50, 2028—2031).—The sandy deposit of calcium salts formed during the concentration of maple syrup contains 51% of mineral matter. In addition to malic acid, small quantities of formic, acetic, fumaric, succinic, and citric acids are present, and traces of *d*-tartaric acid, tricarballic acid, and an unknown acid (*hydrazide*, m.p. 173—175°) have been isolated. H. E. F. NOTTON.

**Flavour of maple syrup.** E. K. NELSON (J. Amer. Chem. Soc., 1928, 50, 2009—2012).—The ethereal extract of maple syrup from which most of the sugar has been precipitated by means of barium hydroxide contains acids, phenolic derivatives, and a *substance*, m.p. 212°, which is not identical with hydrophlorone, but yields a volatile quinone with ferric chloride. The maple flavour is destroyed by dissolving in alkali, and it cannot be recovered unchanged after adsorption on norit. Direct ether extraction of the syrup yields a product with an intense odour of maple, which arises principally from unstable resinous phenolic substances. The extract also contains a *product*, m.p. 210—212°, which does not yield a quinone on oxidation, and *aldehydes*, one of which, m.p. 74—76°, resembles but is not identical with vanillin or ethylvanillin. It is oxidised by ferric chloride to a substance resembling dehydrodivanillin. H. E. F. NOTTON.

**Manufacture of maize starch and the extraction of the residues and maize oil.** O. K. A. KRIZKOVSKY (Chem.-Ztg., 1928, 52, 425—428, 466—467, 486—487, 526—528).—A detailed description of the various stages in the process of manufacture employed in Germany. 100 pts. of maize containing 13 pts. of water and 5 pts. of water-soluble matter yield on the average 52.33 pts. of starch, 22.77 pts. of fodder, 3.87 pts. of oil

cake, and 2.5 pts. of maize oil, all calculated on the dry basis. F. R. ENNOS.

**Chloroform test in evaluation of the quality of starch.** W. PLAHL (Z. Unters. Lebensm., 1928, 55, 295—296).—The abnormal colour of starch of inferior quality becomes much more evident when the sample is moistened with chloroform. W. J. BOYD.

**Honey.** ELSER.—See XIX.

#### PATENT.

**Extraction of sugar from beet.** R. G. W. FARNELL (B.P. 293,066, 23.2.27).—Beet cosettes are dried and extracted with water at a temperature not exceeding 50—60° and under reduced pressure, which may be varied. F. R. ENNOS.

### XVIII.—FERMENTATION INDUSTRIES.

**Autolysis of bottom-fermentation yeast.** W. WINDISCH, P. KOLBACH, and E. F. ROTHENBACH (Woch. Brau., 1928, 45, 251—256, 261—265, 281—285, 298—302).—The autolysis was carried out on 30-g. portions of washed yeast, freed from hop resins by treatment with 10% sodium carbonate solution, in 250 c.c. volume and adjusted to the required reaction by addition of 0.1N-hydrochloric acid and caustic soda. By working at 47°, autofermentation and bacterial action were suppressed. Total solids and "formol" and total nitrogen were determined on the filtrates from the boiled digestions. The controls were stirred for 15 min. with 250 c.c. of water at 15° in place of the digestion. Fresh yeast must be employed for this work, as changes occur in a few days even at 2°. Autolysis proceeds most rapidly at  $p_H$  5, the optimum for yeast pepsinase, and for 3-hr. digestions the most favourable temperature is 55°, for 6-hr. digestions 52°, the latter conditions giving 89% of the total nitrogen in solution. The proteins are still further broken down if after a time the reaction is readjusted to  $p_H$  6.25, apparently owing to the action of a tryptase or pepsidase. The autolysis of yeast plasmolysed by chloroform proceeds most rapidly at about  $p_H$  6. F. E. DAY.

**Fermentation carbon dioxide.** F. STOCKHAUSEN and F. WINDISCH (Woch. Brau., 1928, 45, 277—281, 289—298, 305—311, 317—324, 329—333).—Fermentations carried out in flasks in which the carbon dioxide was retained under pressures of 0.0066—1.0 atm. are compared with similar fermentations in flasks plugged with cotton wool. In all cases those under pressure show a slight retardation of the rate of fermentation, though the same final values for  $p_H$  and attenuation are reached and clarification is improved. The yeast, though less in quantity, is of better microscopical appearance and fermentative activity. Similar results are obtained at all the pressures employed and at 0.0066 atm. applied from the end of the first to the end of the fifth day. Since also yeast increase,  $p_H$ , and attenuation reach the same final values at 2.5°, 10°, and 20°, it is concluded that the difference between open and closed fermentation is mainly due to more complete exclusion of air from the latter. A series of four successive fermentations under 0.0066 atm. pressure indicates that under such conditions yeast maintains its

vitality better than in open fermentations. The possible industrial uses of carbonic acid are discussed.

F. E. DAY.

**Colorimeter [for wort etc.].** K. S. FELIX, HELMRICH, and WANDERSCHECK (Woch. Brau., 1928, 45, 312—315).—A parallel beam of light from an incandescence lamp is passed down a vertical cylinder carrying a window at the lower end. The cylinder can be lowered by rack and pinion into a glass-bottomed vessel containing the liquid to be tested. The ray then passes through a yellow filter, to decrease sensitivity to the violet end of the spectrum, and finally impinges on a potassium photo-electric cell. The current passing through this is read on a milliammeter. The zero of the instrument is obtained by bringing the movable cylinder down till the window is in contact with the bottom of the containing vessel and inserting a removable comparison slide of suitable colour. The ammeter reading is taken, the slide removed, and the cylinder raised till the depth of liquid in the container is sufficient to bring back the ammeter reading to the original figure. This depth is measured on a scale attached to the cylinder. The method of calibrating the instrument is described. The use of the yellow filter and of the principle of substitution of colour against a standard slide have overcome the original difficulty of the inconstancy of the potassium cell after a period of rest. F. E. DAY.

**Stabilisers for wines. [Determination of volatile acids.]** FONZES-DIACON (Ann. Falsif., 1928, 21, 266—272).—Salicylic acid (0.5 g. per litre) is recommended to be added to the sample of wine as a preservative in preference to sodium benzoate. Volatile acids in such wines may be determined by the method of Blarez, which involves direct titration in a colourless solution, but a correction of 0.1 g. should be made for combined salicylic acid. The Roos-Mestrezat method gives slightly higher results (0.05) and a correction of 0.15 g., representing 0.1 for salicylic acid and 0.05 as the mean difference for the two methods, should be applied. In Duclaux's method the volatile acidity is always low, and the French official method, although not influenced by the stabilisers, gives much too low results.

D. G. HEWER.

**Spectrochemical detection of fruit wine in wine.** O. E. KALBERER (Z. Unters. Lebensm., 1928, 55, 214—230; cf. B., 1927, 686).—The absorption spectra of various red and white wines with and without addition of fruit wine were examined with reference to the characteristics of the extinction curves. When 15% or more of fruit wine was present, it was detected by the shape of the extinction curve. The absorption spectrum is little affected by the presence of sulphur dioxide, but it is considerably affected by decolorisation with charcoal.

W. J. BOYD.

**Detection of isopropyl alcohol in still wine by means of piperonal.** G. REIF (Z. Unters. Lebensm., 1928, 55, 204—214).—The alcohol is distilled from 10 c.c. of the sample on the boiling water-bath and the distillate diluted to 30 vol.-%, 0.3 c.c. of the diluted distillate is mixed with 5 c.c. of 0.5% alcoholic piperonal solution and 1 c.c. of water, 20 c.c. of sulphuric acid warmed to 40° are added, and after 5—10 min. 3—4 c.c. of the

mixture are warmed for 3—5 min. on the boiling water-bath. A greenish-brown colour indicates the absence of isopropyl alcohol, whilst a reddish-brown to red colour shows its presence. By this method 1% of isopropyl alcohol can be detected without first destroying the aldehydes, terpenes, and acetal. Satisfactory results were also obtained using anisaldehyde instead of piperonal. The method can also be used for the detection of isobutyl alcohol. Fermentation amyl alcohol may be removed by adding 0.04 g. of animal charcoal and 2 c.c. of water to 0.3 c.c. of the diluted distillate and filtering, the filtrate being tested as described above. If 30 c.c. of 30% acetic acid are added to the reaction mixture after it has been warmed on the water-bath, in the presence of isopropyl alcohol a rose or rose-red coloration develops and persists for some time.

W. J. BOYD.

#### PATENTS.

**Manufacture of baking yeast.** H. CLAASSEN (B.P. 280,861, 26.8.27. Ger., 19.11.26).—From a mother yeast which is cultivated in a solution containing purely organic nutriment, two or more cultures are prepared in molasses solution, which contains increasing quantities of inorganic nitrogen nutriment in addition to the organic one. Increasing proportions of albumin are found in the yeasts, the first culture containing about 55%, and the last about 60%. The last culture is not compressed, but separated from the wort and used as store yeast for the manufacture of baking yeast in molasses solution alone or with the addition of other organic nitrogen nutriment.

C. RANKEN.

**Production of pale, strongly hopped beers.** HOFBRAUHAUS WOLTERS UND BALHORN A.-G. (B.P. 283,879, 18.1.28. Ger., 18.1.27).—The waste steam from the whole of the brewing operations is condensed and used for brewing the beer, its heat being utilised if desired. The condensate may be mixed with pre-treated deep-well water, in order to decrease the hardness of the latter prior to being used for mashing or spraying.

C. RANKEN.

**Acetification of vinegar.** H. FOX (B.P. 293,539, 30.5.27).—The apparatus consists of a revolving liquor receiver provided with sprayer pipes of different lengths, having perforated nozzles at the outer ends arranged to give a uniform distribution of liquor over the birch bed surface. On the discharge side of the pump a trap pipe is attached to prevent the resultant vapour and gases escaping through the pump. An atmospheric condenser on the outside of the vat acts as an outlet for the gases and vapour, returning the condensed vapour back to the vat, whilst the gases escape by a pipe to the atmosphere or may be collected.

C. RANKEN.

**Production of alcohol and acetone from vegetable matter.** A. C. THAYSEN, L. D. GALLOWAY, and W. E. BAKES (B.P. 293,514, 30.4.27).—Vegetable residues and tissues containing hemicelluloses or polysaccharides which liberate pentoses by hydrolysis are passed through a steeping drum containing dilute acid. The steeped material is pressed to remove surplus acid and steamed at atmospheric pressure in vats. The soluble carbohydrates are extracted by sterilised water and the

neutralised mash is fermented with pentose-fermenting types of micro-organisms, the fermented liquor being subsequently distilled to collect the volatile products.

C. RANKEN.

**Manufacture of yeast.** A. P. HARRISON, Assr. to FLEISCHMANN Co. (U.S.P. 1,676,437, 10.7.28. Appl., 19.10.25).—See B.P. 277,476; B., 1927, 889.

## XIX.—FOODS.

**Neutralisation of milk and its detection.** R. STROECKER (Z. anal. Chem., 1928, 74, 1—28; cf. Tillmans and Luckenbach, B., 1925, 967).—A method depending on the fall in the electrical conductivity of milk is described. Although by the use of a mercuric chloride-hydrochloric acid serum and variously treated milk samples a better comparison of their respective acidities could be made than was possible from the corresponding Soxhlet-Henkel figures, for practical purposes it was found better to employ dialysed ferric hydroxide solution in hydrochloric acid as the serum, the fall in conductivity being found from the relation: conductivity of serum diluted with water + conductivity of the hydrochloric acid used diluted with water—conductivity of the acid-treated serum = fall in conductivity. Lactic acid alone in the acidified serum caused only negligible change in its conductivity. The method consists in mixing 50 c.c. of milk with 10 c.c. of double-distilled water and 40 c.c. of dialysed ferric hydroxide solution, the whole being vigorously stirred for some moments. After filtration, 20 c.c. of the filtrate are mixed with 30 c.c. of double-distilled water, and the specific conductivity at 18° is measured. Separate measurements are similarly made of the conductivities of a further 20 c.c. of the serum when treated with 25 c.c. of double-distilled water and 5 c.c. of 0.2*N*-hydrochloric acid, and of a mixture of 45 c.c. of double-distilled water with 5 c.c. of 0.2*N*-hydrochloric acid. The relation quoted above is then applied. Temperature control is essential. A method of constructing a practical "neutralisation curve" is explained. The conductivity method permits of the accurate determination of neutralisation over 1°, and is independent of slight variations in the milk, such as raised sodium chloride content. It is practicable for skimmed, dried, watered, or heated milk, no dilution or bringing into solution being required. The influence of the iron solution is only slight, but preservatives can considerably affect the results.

R. A. PRATT.

**Apparent viscosity of milk as influenced by some physical factors.** G. M. BATEMAN and P. F. SHARP (J. Agric. Res., 1928, 36, 647—674).—The viscosity coefficient of milk (whole, skimmed, or condensed skimmed) increases with the shearing force applied and approaches a constant at high shear values. Under conditions usually obtaining in processes for determining the viscosity of milk and milk products, the curvature of the viscosity-pressure curve is in the region of its greatest value, and thus small changes in pressure markedly affect the values obtained. Mechanical agitation may decrease the viscosity of whole milk, but does not affect that of skimmed or homogenised milk. The viscosity of skimmed milk increases with age, but aged skimmed

milk tends to regain its original viscosity after repeated passage through the capillary. Homogenising of whole milk increases its viscosity, but does not affect that of skimmed milk. The viscosity of skimmed milk is increased by pasteurising at 62° for 30 min. Freezing of skimmed milk decreases its viscosity, but after being maintained in a frozen condition for several days the viscosity returns to its original value. Similar effects result by alternate freezing and thawing for several days. The viscosity of skimmed milk is not a strictly linear function of the total solid content.

A. G. POLLARD.

**Determination of milk in caramel etc.** W. HARTMANN (Pharm. Zentr., 1928, 69, 337—339; cf. Lührig, B., 1927, 236).—The Reichert-Meissl values of a series of mixtures of butter fat and coconut oil were determined using 1 g. of the fat saponified with 4 g. of 5% glycerin-potash solution. The values obtained after correction by the divisor figure for the butter fat were considerably higher than those calculated from the Reichert-Meissl numbers of the component fats, for all mixtures up to 75% of butter fat. With increasing butter fat content above 75% the values found approached the calculated values until at 90% of butter fat they agreed. A further correction using the divisor figure 1.06 should be applied for all mixtures of butter fat and coconut oil containing less than 75% of butter fat. Still higher results are obtained if the proportion of glycerin-potash solution used for saponification is increased. It is important to maintain the exact length (52 cm.) of the condenser of the Polenske apparatus.

W. J. BOYD.

**Cryoscopic examination of milk and determination of the "cryolac number" and the chlorine-sugar number.** J. FIEBE and W. KORDATZKI (Z. Unters. Lebensm., 1928, 55, 251—262).—The milk of cows inoculated with various pathogenic sera showed no alteration in f.p. depression or in osmotic pressure. The f.p. depression of normal milk serum was between 0.537° and 0.576° (mean 0.552°). The "cryolac number" was between 393 and 435 (mean 413), indicating that the chloride and lactose account for approximately 75% of the total depression. The chlorine-sugar number varied from 1.63 to 6.63 for individual animals. Neither microscopical examination of the milk nor clinical examination of the animal showed any relation between high chlorine-sugar number and mastitis. The f.p. depression and "cryolac number" are shown to be useful in determining the amount of water added to milk.

W. J. BOYD.

**Evaluation of milk.** J. GERUM (Z. Unters. Lebensm., 1928, 55, 274—283).—The interpretation of analytical data for samples of milk is discussed. The importance of weighing all the data together, and in particular the usefulness of cryoscopic determinations in deciding whether abnormalities are due to watering or skimming of the milk or to the condition of the animal, are emphasised. The administration of aloes to the cow produces a marked but delayed lowering of the fat content of the milk.

W. J. BOYD.

**Action of halogens on milk and its constituents.** A. J. J. VANDE VELDE (Natuurwetensch. Tijds., 1928,

10, 66—72).—Chlorine and bleaching powder cause coagulation, the casein being richer in chlorine in the latter case. The action of bromine is similar. Successive small quantities of iodine are completely absorbed, and appear in the ash and the casein; the milk regains its white colour on keeping between each addition, and coagulation occurs only after considerable quantities have been added (1 g. of iodine powder to 100 c.c. of milk). Milk may be sterilised by suitable addition of iodine. The casein precipitated after several small additions contained 19.8% I and was stable. Chlorine and bromine oxidise lactose to mucic acid; iodine has practically no effect. S. I. LEVY.

**Chemical composition of certain Italian milk-foods.** L. SETTIMI (*Atti R. Accad. Lincei*, 1928, [vi], 7, 586—589).—Roman ricotta is prepared by heating the whey, obtained by coagulating milk with rennet in the manufacture of cheese, at 75—80° in presence of an organic or mineral acid. Two types exist, differing in the proportion of fat, the difference being probably due to the use of whey from whole milk in one case and of whey from de creamed milk in the other. Their mean percentage compositions are: water, 56.0, 66.3; total nitrogenous compounds, 8.51, 8.3; soluble nitrogenous compounds, 1.04, 1.1; fatty matters and phosphatides, 33.8, 23.5; lecithin, 1.16, 0.94; lactose, 0.9, 1.1; ash, 0.48, 0.49; acidity as lactic acid, 0.21, 0.23%; g.-cal. per 100 g., 353, 257. The soluble nitrogen compounds are mainly proteoses and peptones, with small proportions of amino-acids, and the ash is essentially calcium phosphate, with small amounts of chlorides and sulphates of sodium, potassium and magnesium. Two types of mozzarella are also made, the Roman from de creamed milk and those of Cardito from partly de creamed milk; their respective compositions are: water, 67.0, 61.0; total nitrogenous compounds, 27.0, 16.0; soluble nitrogen compounds, 5.1, 2.15; amino-nitrogen, 0.25, 0.12; fats and phosphatides, 3.0, 20.0; lecithin, nil, 1.05; lactose, trace, 0.5; ash, 2.42, 2.4%; g.-cal. per 100 g., 139, 253; the ash is mostly sodium chloride (47%) and calcium phosphate (30%), with small amounts of the phosphates, chlorides, and sulphates of sodium, potassium, and magnesium.

T. H. POPE.

**Fat losses in buttermilk.** W. B. COMBS (*World's Butter Rev.*, 1928, 2, No. 3, 9—10, 20).—The fat content of buttermilk is greatest in the spring. The Mojonner (Röse-Gottlieb) method for the determination of fat agrees with the butyl alcohol method, but the result is higher than is given by Babcock's method.

## CHEMICAL ABSTRACTS.

**Determination of butyric and caproic acids in butter fat.** A. I. VIRTANEN [with L. PULKKI] (*Z. anal. Chem.*, 1928, 74, 321—330).—The Reichert-Meissl distillate contains 89% of the butyric acid and 95% of the caproic acid present in the butter fat. Solubility and other influences are such that the amount of caprylic acid is practically constant and equivalent to 3.5 c.c. of 0.05*N*-alkali. The amount of capric acid is negligible. To determine the amounts of butyric and caproic acids 100 c.c. of the Reichert-Meissl distillate (containing caprylic acid equivalent to 3.2 c.c.

of 0.05*N*-alkali) are distilled and 50 c.c. of distillate are collected. This distillate, which contains caprylic acid equivalent to 3.05 c.c. of 0.05*N*-alkali, and the residual liquid in the distillation flask are titrated with 0.05*N*-alkali. If *B* and *C* are the amounts of butyric and caproic acids, expressed as c.c. of 0.05*N*-alkali, in 100 c.c. of the Reichert-Meissl distillate and *x* and *y* the titrations of the distillate and residue therefrom, respectively, then  $B + C = x + y - 3.2$  and  $0.745B + 0.925C = x - 3.05$ . These relations are valid only when the distillation occupies about  $\frac{1}{2}$  hr. The amounts of butyric and caproic acids in the entire Reichert-Meissl distillate are thus calculable. The amounts of butyric and caproic acids in the butter fat itself are obtained by multiplying these figures by 100/89 and 100/95, respectively. The amount of butyric and caproic acids in 10 specimens of Finnish butter varied between 3.12% and 4.24% (mean 3.79%) and 1.38% and 2.12% (mean 1.75%), respectively. The solubilities in water at 15° of caproic, caprylic, and capric acids are 0.891, 0.072, and 0.0026 g./100 c.c. of water, respectively. J. S. CARTER.

**Prolamine of wheat flour.** R. HERZNER (*Z. Unters. Lebensm.*, 1928, 55, 262—274).—The proteins extracted from wheat flour by different alcohol-water mixtures are not identical. A study of the molecular binding power, specific rotations, and elementary analyses of different fractions reveals the presence of two main groups of prolamines. W. J. BOYD.

**Mastication test for sand in flour.** W. PLAHL (*Z. Unters. Lebensm.*, 1928, 55, 294—295).—Determinations of ash, ash insoluble in hydrochloric acid, and total mineral matter by treatment with chloroform sometimes fail to detect the presence in flour of amounts of sand sufficient to render the material uneatable. In such cases the mastication test may be of decisive importance. Fine sand, however, may not be detected by the teeth. W. J. BOYD.

**Chemical composition and nutritive value of yellow and white oats.** F. HONCAMP, W. SCHRAMM, and H. WIESSMANN (*J. Landw.*, 1928, 76, 113—127).—The higher feeding value of yellow oats depends on a slightly larger content of digestible protein, but more particularly on its higher starch equivalent and lower proportion of husk. A. G. POLLARD.

**Colour-producing constituents of the cacao bean.** W. B. ADAM (*Analyst*, 1928, 53, 369—372).—Unfermented cacao beans contain a catechu-tannin, a compound of the catechin series ("cacao catechin"), and a compound of the latter with caffeine which is resolved into its components by boiling chloroform. The colouring substances are complex alteration products of the tannin and the catechin. Cacao catechin, m.p. 229°, was obtained from unfermented Accra cacao beans by extraction with light petroleum and then with chloroform, removing fat and xanthine bases, extraction with ether under specified conditions, and purification with lead acetate. The catechin was determined colorimetrically by means of Mitchell's ferrous tartrate reagent. (Gambier catechin and acacatechin may be satisfactorily compared by this



reagent.) Cacao catechin must be used as a standard of comparison, since gambier catechin gives a bluer tint. The tannin is determined by shaking the dry ether-extracted powder with water at 60°, filtering, and adding an equal volume of a saturated solution of cinchonine sulphate. After coagulation of the precipitate the solution is filtered through a weighed alundum crucible, washed with dilute cinchonine sulphate solution, and dried. About 0.8% of catechin was found in unfermented beans, but fermentation decreases the proportion until none is present in fully fermented beans. Tannin varies from 1.7% in fermented Guayaquil beans to 2.37% in unfermented West African and 3.48% in very poorly fermented Machala beans. The average tannin content is about 1.9% on completion of fermentation.

D. G. HEWER.

**Determination of hydrocyanic acid in Lima beans with special reference to the most favourable  $p_H$  for glucoside fission.** S. K. HAGEN (Z. Unters. Lebensm., 1928, 55, 284—291).—The following method is recommended: 50 g. of the finely ground beans are shaken with a mixture of 244 c.c. of "citrate" and 156 c.c. of 0.1N-sodium hydroxide giving a  $p_H$  value of 6 (Sørensen, Compt. rend. Lab. Carlsberg, 8), and after 3 hrs. 50 c.c. of 30% tartaric acid solution are added; 250 c.c. of the liquor are distilled off in a strong current of steam into 50 c.c. of water containing 1—2 c.c. of 2N-sodium hydroxide. To the distillate 1 g. of potassium iodide is added and the hydrocyanic acid is determined by titration with 0.05N-silver nitrate (1 c.c.=2.70 mg. HCN). The experimental error of the method amounts to 0.1 mg. of hydrocyanic acid. The presence of the active glucoside-splitting ferment of the beans is indispensable in the above method.

W. J. BOYD.

**Occurrence of starch in oily seed-kernels used as foodstuffs.** C. GRIEBEL (Z. Unters. Lebensm., 1928, 55, 236—239).—The appearance, distribution, and detection of starch grains in sapucaja nuts (*Lecythis*), pecan nuts (*Carya olivae-formis*, Nutt.), cedar nuts (*Pinus cembra*), and pine kernels (*Pinus pinea*) are described.

W. J. BOYD.

**Relationship between digestibility of fish flesh prepared by different methods and its water content.** B. GLASSMAN (Z. Unters. Lebensm., 1928, 55, 231—235).—Fish flesh roasted by "dry heat" is more rapidly digested by pepsin *in vitro* than that prepared in "boiling oil" and then de-oiled, because by the former process a product of higher moisture content is obtained and the presence of the moisture moderates the polymerisation of the protein by heat. The presence of oil hinders the digestion only by mechanically preventing contact of substrate and ferment. There is no loss of protein in the "boiling oil" process.

W. J. BOYD.

**Effect of onion and garlic on the growth of bacteria in sausages.** E. DANIELSEN (Z. Unters. Lebensm., 1928, 55, 291—294).—The sterilising effect of onion and garlic on the growth of various aerobic and anaerobic bacteria likely to occur in sausages was found to be negligible.

W. J. BOYD.

**Sanitary evaluation of flesh foods.** L. M. HOROVITZ-VLASOVA (Z. Unters. Lebensm., 1928, 55, 239—246).—Determinations of alkalinity or acidity of the aqueous extract,  $p_H$ , refractive index, intensity of biuret reaction, total nitrogen, nitrogen precipitated by tannin and by phosphotungstic acid, oxidisability, and iodine value were found to be of no value as an indication of the freshness of flesh foods. The Eber reaction is useless because it is negative till after putrefaction has set in. Testing with red litmus paper for ammonia evolved on boiling with sodium hydroxide or magnesia is of no practical value because not only are ammonium salts decomposed, but ammonia is evolved from the proteins. By warming the extract or the finely minced meat in ammonia-free water after addition of magnesia for 5 min. at 50° no ammonia is removed from the proteins. Accordingly the following test is recommended: 1 g. of flesh in 10 c.c. of water and 0.1 g. of magnesia are heated at 50°, and if after 5 min. red litmus paper held above the mixture turns blue the flesh is judged to be unsound. About 0.02% of ammonia is detectable by this test. The Sørensen titration method was found useful, but the preparation of the extract introduces a source of error.

W. J. BOYD.

**Cooking of foods under pressure.** J. FROIDEVAUX (Ann. Falsif., 1928, 21, 252—257).—Broth prepared in an autoclave is richer in amino-acids than when made by the ordinary method, and contains more ammoniacal nitrogen than the meat from which it was prepared. The broth is poorer in nitrogenous material, in fat, and in reducing substances than ordinary broth, but the meat is better cooked under pressure and the method is more economical.

D. G. HEWER.

**Determination of the inorganic constituents of honey.** E. ELSER (Z. Unters. Lebensm., 1928, 55, 246—251).—The author has extended and improved the methods previously described (B., 1925, 819). The removal of ammonium salts after incineration is omitted. Phosphoric acid is precipitated as strychnine phosphomolybdate and the suspension compared with a standard preparation using a nephelometer. Iron is determined colorimetrically as the thiocyanate, manganese colorimetrically as permanganate after oxidation with potassium persulphate. Potassium is precipitated as cobalt-nitrite and the washed precipitate is titrated in hot sulphuric acid solution with potassium permanganate. Calcium and chlorine are determined as described previously. Analyses are given of the inorganic constituents of four samples of Austrian honey.

W. J. BOYD.

**Acids of figs.** E. K. NELSON (J. Amer. Chem. Soc., 1928, 50, 2012—2013).—The following acids have been isolated: (a) from normal Adriatic figs, acetic (0.027% by weight) and citric (0.32%); (b) from Adriatic figs affected with "black neck," acetic (0.32%) and citric (0.24%); (c) from normal Calimyrna figs, acetic (0.026%), citric (0.35%), and a little malic; and (d) from Calimyrna figs affected with internal rot, acetic (0.056%), citric (0.33%), and a little malic acid.

H. E. F. NOTTON.

**Chloride content of canned sauerkraut.** M. E. STARK (J. Lab. Clin. Med., 1927, 12, 561—563).—

Average values for sodium chloride are 1.86 g. per 100 c.c. of juice and 1.74 g. per 100 g. of solid.

## CHEMICAL ABSTRACTS.

**Commercial production of sauerkraut.** E. LE FEVRE (U.S. Dept. Agric., Circ. 35, 1928, 30 pp.).

**Behaviour of fats in ultra-violet light.** HAITINGER and others.—See XII.

**Tapioca.** GREENSTREET.—See XVI. **Maize starch.** KRIZKOVSKY.—See XVII.

## PATENTS.

**Obtaining an alimentary extract from fish.** A. EHRENREICH (B.P. 284,636, 1.2.28. Belg., 1.2.27).—The flesh of the fish is heated with dilute hydrochloric acid at 70–100°, and the liquid is afterwards neutralised with sodium carbonate and concentrated *in vacuo* to a thick extract, which may be dried *in vacuo* and compressed into cubes or tablets if desired.

F. R. ENNOS.

**Preservation of fruits, vegetables, etc.** H. G. ZELLNER (B.P. 293,341, 4.4.27).—Fruits or vegetables with a skin or rind are scrubbed and washed in a solution made by intermixing copper sulphate and potassium permanganate with alkali hydroxide solution. After rinsing in fresh water, followed by a final washing with a solution containing sodium pentasulphide and in alkali hydroxide, the fruits or vegetables are dried.

C. RANKEN.

**Production of enzymes for use in manufacture of bread.** H. A. KOHMAN, K. IRVIN, and E. S. STATELER, Assrs. to FLEISCHMANN Co. (U.S.P. 1,676,446, 10.7.28. Appl., 3.10.21).—See B.P. 186,924; B., 1924, 30.

**Apparatus for heat treatment of cereal substances.** C. W. CHITTY, D. W. KENT-JONES, and WOODLANDS, LTD. (B.P. 294,001, 26.4.27).

**Animal feed.** A. B. SCHREIBER (B.P. 293,989, 27.7.27).

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Alkaloids of tobacco.** A. SCHMUK (U.S.S.R. State Inst. Tobacco Invest., Bull. 44, 1928, 47 pp.).—A summary is given of the chemistry and transformations of the alkaloids of tobacco, together with a critical review of the methods suggested for the determination of nicotine in tobacco.

T. H. POPE.

**Occurrence of arsenic in tobacco.** H. POPP (Z. angew. Chem., 1928, 41, 838–839).—For the determination of arsenic in tobacco 200 g. are heated with fuming nitric acid until the organic matter is completely decomposed and all chlorine expelled. The solution is evaporated repeatedly with sulphuric acid to remove nitric acid, and arsenic is tested for by the mercuric chloride colorimetric method. Tobacco from the Palatinat contained 5.1, from Macedonia 0.7, from Java 0.33, and from Brazil 4.6 pts. of arsenic per million. This quantity is so small that no danger of poisoning is to be feared.

A. R. POWELL.

**Determination of nicotine in tobacco and tobacco extracts.** R. R. L. WORSLEY (Egypt. Min. Agric. Tech. Sci. Service Bull., 1927, 73, 5 pp.).—The following

method is suitable for use in warm climates. Nicotine sulphate solution (100 c.c.) is shaken for 10–15 min. in a well-stoppered 400 c.c. bottle with 20% sodium hydroxide solution (20–25 c.c.) and exactly 100 c.c. of light petroleum, b.p. 50–60° or above. After settling for 10 min. 40 c.c. are decanted without filtering and poured into 50 c.c. of distilled water. An excess (about 5 c.c.) of 0.1N-sulphuric acid is added, the mixture shaken gently for 1 min., and titrated with 0.01N-sodium hydroxide solution. A better end-point is frequently obtained by adding 5 c.c. of 0.01N-sulphuric acid and titrating again.

CHEMICAL ABSTRACTS.

**Determination of alkaloids by the mercurimetric method.** A. JONESCO-MATIU and H. VARCOVICI (Bul. Soc. Chim. România, 1928, 10, 5–8, 9–13).—I. A 1% solution of the alkaloid (1 c.c. or more) is shaken with 5 c.c. of Mayer-Valzer reagent (mercuric iodide in 10% potassium iodide solution), the precipitate is separated and washed by means of a centrifuge, and dissolved in mixed acid (1 pt. of nitric acid, 2 pts. of sulphuric acid). To the diluted solution a little permanganate is added to remove nitrous acid, then 12 drops of 10% sodium nitroprusside solution, and the mixture is titrated with 0.1N-sodium chloride until the turbidity disappears. The equivalents for atropine, aconitine, papaverine, narcotine, brucine, veratrine, and emetine are worked out from the pure alkaloids.

II. The method is applicable to atropine, quinine, strychnine, morphine in vegetable extracts such as belladonna, quinquina, nux vomica, opium, and jusquiame.

C. HOLLINS.

**Apparatus for determination of alkaloids in lupins.** F. KAMINSKI (Chem.-Ztg., 1928, 52, 467).—The extraction with alkaline ether-chloroform mixture is made in a bottle closed at the top with a stopper and tap, and having a side tube near the base connected through a three-way tap to a burette consisting of two 25-c.c. bulbs. On completion of the extraction, the liquid is blown over into the burette, from which aliquot portions may be drawn off for evaporation.

F. R. ENNOS.

**Determination of synthetic camphor in its pharmaceutical preparations.** J. BOUGAULT and (Mlle.) B. LEROY (J. Pharm. Chim., 1928, [viii], 8, 49–55).—From a study of the volatility, solubility in dilute sodium hydroxide solution, and extraction from acid and alkaline solutions of the camphoroximes the following method has been deduced: 0.5 g. of camphor dissolved in 5 c.c. of alcohol at 90° is mixed with a solution of 1 g. of hydroxylamine hydrochloride in 5 c.c. of water, and to the mixture are added 2 c.c. of sodium hydroxide solution. The liquid is boiled for 2 hrs. in a water bath, cooled, 20 c.c. of water and 3 c.c. of sodium hydroxide solution are added, and, if a precipitate or cloudiness due to camphene or borneol is formed, the liquid is filtered. The excess of alkali is exactly neutralised with hydrochloric acid and the camphoroxime is extracted with ether, 20, 10, and 10 c.c. being used in three extractions. The extracts are each washed with 5 c.c. of water, and the combined extract is allowed to evaporate spontaneously at room temperature for 12 hrs. and then dried over calcium chloride for 12 hrs. and weighed. The weight

is increased by 4% to compensate for volatility of the oxime, and the amount of camphor is 30400C/167, where *C* is the corrected weight. E. H. SHARPLES.

**Essential oils of Travancore. VII. Oil from rhizome of ginger, *Zingiber officinale*.** K. L. MOUDGILL (J. Indian Chem. Soc., 1928, 5, 251—259).—The oil obtained in 2% yield from the scraped rhizomes of fresh, dry ginger has  $d_4^{27}$  0.869,  $n_D^{28}$  1.4891,  $[\alpha]_D^{28}$  —54°, acid value 1.0, ester value 7.4. The chief constituent of the oil is zingeribene (70%), which when heated at its b.p. yields a dextrorotatory monocyclic sesquiterpene, and undergoes an analogous change on prolonged contact with air. The initial oil exhibits a similar behaviour. Old samples of ginger, green ginger, and ginger scrapings give diminished yields of oils, and the oils from the last two differ in some respects from the oil from the rhizomes. H. BURTON.

**Essential oil of *Podocarpus ferrugineus*.** J. R. HOSKING and W. F. SHORT (Rec. trav. chim., 1928, 47, 834—838).—The oil was steam-distilled from leaves and terminal branches of the New Zealand "miro-pine" (0.09% in autumn, 0.14% in spring). The autumn oil,  $d_4^{25}$  0.9602,  $n_D^{25}$  1.4922,  $[\alpha]_{546}^{25}$  + 32.97°, contained 36% of *d*- $\alpha$ -pinene, 12% of cadinene, 5% of *d*-limonene and dipentene, 2% of cineole, 5% of resin, and 27% of a new diterpene, *mirene*, m.p. 102—104°, b.p. 151—152°/0.6 mm.,  $d_4^{21}$  0.9199,  $n_D^{21}$  1.4890,  $[\alpha]_{546}^{21}$  + 27.15° (in chloroform) (*monohydrochloride*, m.p. 97—98°). Acetic and *isovaleric* acids were also identified. The oil collected in spring contained much more *mirene* (47%) and less pinene (24%) and cadinene (6%). C. HOLLINS.

**Determination of essential oils in drugs.** R. BAUER (Pharm. Ztg., 1928, 73, 920—921).—After extracting the steam-distillate saturated with sodium chloride with pentane, the pentane is dried by filtration through filter paper and then distilled at a slightly raised temperature instead of allowing it to evaporate spontaneously. E. H. SHARPLES.

**Determination of essential oils in drugs by Stamm's method.** E. TIKKANEN (Pharmacia, 1926, [5]; Chem. Zentr., 1927, II, 1520).—Stamm's method has been critically examined in its application to different drugs. All determinations with one and the same powdered drug accurately agreed among themselves and gave satisfactory results. The method is inexpensive and can be carried out in 1½ hrs. E. H. SHARPLES.

**Synthetic menthol.** SCHIMMEL & Co. (Ber. Schimmel, 1927, 139; Chem. Zentr., 1927, II, 1519).—Synthetic menthol, m.p. 35°, produced by the authors, is indistinguishable in odour and appearance from that from peppermint oil. Its physical properties lie between those of racemic menthol produced from thymol or piperitone and those of natural *l*-menthol. E. H. SHARPLES.

**Nicotine in sprays.** SCHERPE.—See XVI.

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**Production of inactive menthol (the racemate of natural menthol).** RHEINISCHE KAMPFER FABR. G.M.B.H. (B.P. 285,394, 4.4.27. Ger., 15.2.27).—*i*-Menthol in the liquid mixtures resulting from the fractional

distillation of the hydrogenation products of thymol, *i*-menthone, or *isomenthone* is separated by removing *i*-neomenthol by freezing, and then fractionally distilling the residue with fresh hydrogenation product. The product of the hydrogenation of thymol or *isomenthone* below 160° contains *neoisomenthol*, m.p. 12—14°, b.p. 214.5°/760 mm. (*hydrogen phthalate*, m.p. 90—92°; *p*-nitrobenzoate, m.p. 56°). B. FULLMAN.

**Manufacture of derivatives of acridine.** A. G. GREEN (B.P. 293,617, 2.11.27).—Derivatives of acridines or acridinium salts, containing primary or secondary amino-groups, are converted into aldehyde-bisulphite compounds by treatment in neutral solution with bisulphite and formaldehyde, acetaldehyde, crotonaldehyde, glyoxal, etc. Examples are 3:6-diamino-acridine and acriflavine. The products are hydrolysed in the body, with production of the antiseptic acridine compounds. C. HOLLINS.

**Preparation of quinoline derivatives.** I. G. FARBENIND. A.-G., Assees. of R. SCHWABE, O. EISLEB, and H. JENSCH (G.P. 440,008, 29.3.24).—4-Amino-2-styrylquinolines having strong bactericidal properties are obtained by usual methods from quinaldines containing in 4-position a halogen, alkoxy-, hydrazino-, carbamyl, or carbazido-group. 4-Chloro-2-styrylquinoline, m.p. 117°, gives with alcoholic ammonia at 210—220° 4-amino-2-styrylquinoline, m.p. 173—175° (decomp.). 4-Chloro-6-ethoxy-2-styrylquinoline, m.p. 120°, similarly yields 4-amino-6-ethoxy-2-styrylquinoline, m.p. 212° (*glycollate*, m.p. 237°), also obtained by action of alcoholic ammonia on 4:6-diethoxy-2-styrylquinoline, m.p. 134° (from the chloro-compound and sodium methoxide). Reduction of 4-phenylhydrazino-2-styrylquinoline with zinc dust and acetic acid gives 4-amino-2-styrylquinoline; the 6-ethoxy-derivative is similarly prepared. 10-Chloro-8-methyl- $\beta$ -naphthaquinoline, m.p. 200° (from the 10-hydroxy-compound) is condensed with benzaldehyde to the 8-styryl compound, m.p. 128°, from which 10-amino-8-styryl- $\beta$ -naphthaquinoline, m.p. 226—227°, is prepared. 4-Amino-6-ethoxyquinaldine, m.p. 195°, gives with *m*-nitrobenzaldehyde 4-amino-6-ethoxy-2-*m*-nitro-styrylquinoline, which is reduced to the *m*-aminostyryl compound, m.p. 182°. 2-Styrylquinoline-4-carboxylic acid (*methyl ester*, m.p. 98°; *ethyl ester*, m.p. 77°) is converted by way of the ester into the *amide*, m.p. 274°, from which 4-amino-2-styrylquinoline is obtained by Hofmann's reaction: the *hydrazide*, m.p. 215°, yields the same product by way of the *azide* and the *urethane*, m.p. 202°. 4-Ethylamino-6-ethoxy-2-styrylquinoline, m.p. 178° [*glycollate*, m.p. 214—216° (decomp.)], and the 4-diethylamino-compound, m.p. 103°, and 4-diethylamino-6-ethoxyquinaldine, m.p. 74°, are also described. C. HOLLINS.

**Manufacture of organo-arsenic compounds.** R. W. E. STICKINGS, and MAY & BAKER, LTD. (B.P. 293,152, 3.5.27).—An aminoarylsinic acid is condensed with cyanogen bromide (or other halide) to give a carbamide derivative; if a suitable substituent is present *ortho* to the amino-group, e.g., hydroxyl, carboxyl, or amino-, cyclisation takes place. Examples are: *phenylcarbamide-p*-arsinic acid from arsanilic acid, *anhydro-3-ureido-4-hydroxybenzenearsinic acid* from 3-amino-4-hydr-

oxybenzenearsinic acid, and 2:4-diketo-1:2:3:4-tetrahydroquinazoline-7-arsinic acid from 3-amino-4-carboxybenzenearsinic acid. C. HOLLINS.

**Manufacture of metallo-mercapto-compounds.** W. CARPMAEL. FROM CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 293,363, 2.2.27).—A carbohydrate containing a thiol group is treated with a salt of a metal other than silver. The *aurothiol* derivatives from 1- and 3-thioglucoses, and *bismuth* and *cadmium* salts of 1-thioglucofucose are described. All are readily soluble in water. C. HOLLINS.

**Manufacture of hypnotic solutions.** I. G. FARBENIND. A.-G., Asses. of L. LAUTENSCHLÄGER, M. BOCKMÜHL, and R. SCHWABE (G.P. 447,161, 29.9.25. Addn. to G.P. 446,290; B., 1928, 623).—Hypnotics of the barbituric acid or diallylacetylamide type are dissolved in water-soluble, physiologically innocuous ethers or esters of polyhydric alcohols, such as glycerol diethyl ether, or glycol monoethyl ether. C. HOLLINS.

**Immunising product and method of producing the same.** E. C. R. MARKS. FROM PARKE, DAVIS & Co. (B. P. 293,401, 31.1.27).—Antigens (known as "ecto-antigens" from a supposed association with the ectoplasm) are obtained from micro-organisms by washing with a solvent (physiological saline etc.) for 10–15 min. B. FULLMAN.

**Manufacture of elastic, easily soluble preparations of pharmaceutical products, such as sticks, balls, etc.** W. CARPMAEL. FROM I. G. FARBENIND. A.-G. (B.P. 293,090, 25.3.27).—Sticks, balls or other shaped carriers for medicaments are made from gum tragacanth or the like rendered soluble by means of hydro-tropic substances, such as sodium toluene-*p*-sulphonate or sulphanilate. C. HOLLINS.

**Manufacture of organic gold[–nucleic acid] compounds.** A. GAMS and P. SCHEIDEGGER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,678,429, 24.7.28. Appl., 14.7.27. Switz., 25.8.26).—See B.P. 276,677; B., 1928, 389.

**Manufacture of easily soluble anæsthetics of the *m*-aminobenzoic acid ester series.** O. BILLETTER, E. ROTHLIN, and J. PEYER, Assrs. to CHEM. WORKS, FORMERLY SANDOZ (U.S.P. 1,678,317, 24.7.28. Appl., 3.3.27. Ger., 10.3.26).—See B.P. 291,850; B., 1928, 623.

**Preparation of salts of 3-acetyl-amino-4-hydroxyphenylarsinic acid.** E. FOURNEAU, Assr. to ÉTABL. POULENC FRÈRES (U.S.P. 1,677,964, 24.7.28. Appl., 18.1.27. Fr., 19.1.26).—See B.P. 264,797; B., 1927, 573.

***n*-Amyl[yl]valeroylresorcinol. *iso*Butyrylresorcinol.** A. R. L. DOHME, Assr. to SHARP & DOHME, INC. (U.S.P. 1,677,116–7, 10.7.28. Appl., 29.4.25).—See A., 1926, 838.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Herschel effect by exposure to short wave-lengths.** LÜPPO-CRAMER (Z. wiss. Phot., 1928, 25, 308–315).—The bleaching-out of the latent image by a second

exposure to light, which up to the present has been confined to the red end of the spectrum, has now been shown to take place with yellow, green, and blue light, but only if the intensity used be very small—a condition which is not necessary for the longer wave-lengths. The lower intensity is required with the more penetrating short wave-lengths, so that the light may be absorbed largely in the surface of the grains where recombination of the silver and halogen takes place, as a result of which development will be inhibited to a certain extent. The ordinary phenomenon of reversal is discussed in the light of these new facts.

J. W. GLASSETT.

**Sterry effect with silver chloride.** LÜPPO-CRAMER (Z. wiss. Phot., 1928, 25, 316–319).—Reagents which attack silver (e.g., 1% solutions of potassium dichromate, quinone, cupric and ferric chlorides, ammonium persulphate) destroy the silver chloride latent image much more readily in the higher exposed portions than in the neighbourhood of the threshold value, where the exposure is small. In explanation of this phenomenon it is suggested that the traces of silver first produced by light form a very stable adsorption complex with the halide, and are therefore capable of resisting the relatively weak oxidising action of the above solutions. With further exposure, the stability of the complex decreases as the amount of silver to be adsorbed increases, thus facilitating the destruction of the latent image.

J. W. GLASSETT.

**Covering power of photographic silver deposits.** I. S. E. SHEPPARD and A. BALLARD (Phot. J., 1928, 68, 354–366).—The change in the covering power of silver deposits by varying time of development and exposure has been studied using a *p*-aminophenol hydrochloride developer. For the two emulsions studied the covering power at all exposures decreased with time of development, but tended to increase with increased exposure. The rate of reduction of the grain is also shown to affect covering power. The following accurate method of determination of small quantities of silver has been devised. A measured area of the emulsion is treated for a short while with concentrated sulphuric acid, for partial destruction of the gelatin, and then with a few drops of 1% bromine water. The silver bromide so formed is dissolved in aqueous ammonia and converted into sulphide by the addition of sodium sulphide. The tint of this precipitate is compared with a standard in a microcolorimeter, in which a linear relation was found to exist between the colour strength and the silver content of the solution. By this method it was found possible to determine 0.0025 mg. of silver with an accuracy of  $\pm 0.5\%$  and 0.01 mg. to  $\pm 0.2\%$ . J. W. GLASSETT.

**Economic preparation of silver gelatinobromide plates, and the duration of their sensitivity.** HERVÉ (Bull. Soc. franç. Phot., 1928, 15, 70–74).—Examination of emulsions made between 1881 and 1899 has led to the following conclusions: the sensitivity of silver gelatinobromide plates decreases with time, whatever the method of manufacture of the emulsion; emulsions ripened by boiling have a fine grain and are clean-working for a period of at least 40 years; fast emulsions, ammonia-ripened, have a coarser grain

and fog rapidly on keeping. The methods of making the boiled and ammonia emulsions are described.

W. CLARK.

**Destruction of the image by desensitisers.** LÜPPO-CRAMER (Z. wiss. Phot., 1928, 25, 282—287).—Weakening of the latent image on exposure to red light commences above a definite latent blackening so long as bromide ions are not present. Under certain conditions similar relations hold when desensitisers are used. Plates were exposed under a wedge, bathed in desensitiser, dried, given a diffuse exposure through a red filter, and finally developed. With emulsions with low free bromide content and a red filter not too deep, phenosafranine gave increased density at the low exposures and bleaching at the higher ones. Pinakryptol Green gave similar results, but to a less extent. With a dark red filter, with Pinakryptol Yellow with both medium and dark red filters, and when bromide was added to phenosafranine bleaching occurred at all exposures. The action of red light in presence of phenosafranine, and to a lesser extent Pinakryptol Green, is to give results similar to the Herschel effect under similar conditions.

W. CLARK.

**Developing properties of meta-derivatives.** A. and L. LUMIÈRE and A. SEYEWETZ (Bul. Soc. franç. Phot., 1928, 15, 90—92).—Homolka has shown that resorcinol has no properties as a developer, but that if methyl groups are substituted in the positions adjacent to the hydroxyl groups, the compound then acts as a developer. Pure specimens of 2:4-dimethylresorcinol, 2:4-dimethyl-*m*-phenylenediamine, and 2:4-dimethyl-*m*-aminophenol were prepared, but only the resorcinol derivative had the properties of a developer. Homolka's view that the absence of developing properties in resorcinol is due to its undergoing tautomerisation in presence of alkalis to form a diketone is questioned because it apparently cannot apply to the aminophenol and the diamine.

W. CLARK.

**Photomicrography in colour.** T. S. CURTIS (J. Amer. Ceram. Soc., 1928, 11, 609—632).—An outline of technique.

#### PATENTS.

**Manufacture of photographic silver halide emulsions.** O. MATTHIES, P. WULFF, W. DIETERLE, and B. WENDT, Asss. to I. G. FARBENIND. A.-G. (U.S.P. 1,673,522, 12.6.28. Appl., 10.7.26. Ger., 25.7.25).—See B.P. 255,846; B., 1927, 269.

**Colour photography or cinematography.** L. DUFAY (U.S.P. 1,673,350, 12.6.28. Appl., 3.8.26. Fr., 4.12.25).—See B.P. 262,386; B., 1927, 461.

### XXII.—EXPLOSIVES; MATCHES.

**The Trauzl test.** R. NEUBNER (Z. ges. Schiess- u. Sprengstoffw., 1928, 23, 1—5, 53—56, 82—87, 126—129, 162—166, 194—198).—It is proposed to modify the Trauzl lead block test by measuring the weight of explosive that is required to give on explosion an expansion of 300 c.c. This eliminates the gradual weakening resistance of the lead wall, the effect of which is a difficult factor to appraise in the usual form of the test in which fixed weights of explosive are used. As a

basis of comparison between explosives it is proposed to calculate from the weight of explosive required for an expansion of 300 c.c. the expansion that would be caused by 10 g., and to call this the coefficient of power (Kraftzahl, KZ). Directions are given for converting graphically the old Trauzl values into KZ values. The energetics of explosive reactions is discussed at some length, and expressions are obtained that enable the detonation velocities and brisance of explosives to be calculated.

S. BINNING.

**Apparatus for determining ignition and explosion points of explosives.** M. KOSTEVITCH (Z. ges. Schiess- u. Sprengstoffw., 1928, 23, 156—157).—An all-glass apparatus for determining the ignition point of explosives is described, which enables the first signs of ignition to be detected. For explosion point determinations a metal apparatus is used in which Wood's alloy is used as the heating agent.

S. BINNING.

**Relation between temperature of explosion of a powder and its rate of combustion.** H. MURAOUR (Compt. rend., 1928, 187, 289—290).—For mixtures of equal amounts of guncotton and nitroglycerin containing from 0 to 15% of centralite the logarithm of the rate of combustion is a linear function of the temperature of explosion.

J. GRANT.

**Duration and length of the explosion flames of various explosives.** H. KAST and H. SELLE (Z. ges. Schiess- u. Sprengstoffw., 1928, 23, 153—156).—Photographs taken on stationary and on rotating films are shown of the explosion flames of trinitrotoluene, tetryl, and kieselguhr-dynamite and several German coal-mine explosives. With trinitrotoluene three types of flame could be detected, a primary flame, a weaker secondary flame, and an after-combustion flame from the burning of detonation products in the air. No after-combustion flame was seen with the coal-mine explosives, but it is wrong to say that they give no measurable primary flame.

S. BINNING.

**Liquid air as an explosive.** C. BUNGE (Z. ges. Schiess- u. Sprengstoffw., 1928, 23, 44).—The view that liquid-air explosives are safer than the ordinary solid type is criticised. Even with liquid-air explosives many of the dangerous operations are still necessary, and in addition they are unexpectedly very sensitive to mechanical shock.

S. BINNING.

**Mixed acid.** VON BEZOLD.—See VII.

#### PATENT.

**Nitration of cellulosic materials.** A. P. H. DESBOROUGH, W. T. THOMSON, and R. S. G. KNIGHT (B.P. 293,190, 24.6.27).—Sheets of cellulosic material such as cotton are passed by a continuous band on to a roll placed above a shallow bath into which the nitrating acid is fed at the same rate as it is consumed. Only the periphery of the roll, which may consist either of spaced discs or of a perforated drum, dips below the acid.

S. BINNING.

### XXIII.—SANITATION; WATER PURIFICATION.

**Partial aeration of strong sewage with activated sludge.** W. D. HATFIELD (Ind. Eng. Chem., 1928, 20, 832—835).—Experiments carried out at Decatur (Illinois)

on a sewage which consists of 60% of normal city sewage and 40% of waste from a corn-products factory confirmed results obtained at Birmingham, that the volume of settled sewage which can be satisfactorily treated on percolating filters may be considerably increased by partial aeration with activated sludge prior to filtration. A pre-aeration plant is now in operation dealing with a dry-weather flow of 10 m.g./day with an aeration period of 2.5 hrs. and a settlement period of 2.6 hrs. In constructing this plant it was considered that the lower power consumption of Simplex aerators did not justify the larger initial cost of construction as compared with forced aeration through porous tiles. The excess sludge from the plant is passed into Imhoff tanks and digested with the fresh sludge. The gases produced are collected and utilised for heating the air before compression, thus reducing the power consumption in very cold weather.

C. JEPSON.

**Determination of stable and unstable organic matter in sewage-polluted liquids.** W. E. ABBOTT (Ind. Eng. Chem., 1928, 20, 406—407).—The approximate percentage of the carbonaceous matter which undergoes change during the first-stage absorption of polluted liquids has been determined, the acid dichromate absorption being taken as a rough measure of the total organic matter. The oxygen absorbed during the first stage is usually 10% higher than that absorbed up to the beginning of nitrification, as for several days the oxidation of the last portions of the fermentable carbonaceous matter proceeds simultaneously with nitrification, but in sewages free from suspended matter the first-stage oxidation represents the oxidation of most of the soluble carbonaceous matter. The average ratio of the oxygen absorbed during the first stage to the dichromate absorption was 0.72 for raw sewage, 0.88 for raw sewage centrifuged, 0.15 for an activated sludge effluent, and 0.25 for river water. Adeney and Dawson's acid dichromate method (B., 1927, 934) and Johnson's oxy-albuminoid absorption method (B., 1927, 318) have been compared. The latter is considered unsuitable to distinguish between readily fermentable and stable carbonaceous matter.

W. J. BOYD.

**Titration of carbon dioxide [in water].** L. SMITH and G. WODE (Z. angew. Chem., 1928, 41, 208—212).—A comparison is made on a series of natural waters of methods for determining total, free, and combined carbon dioxide. Free carbon dioxide is titrated with 0.05*N*-caustic soda using a standard amount of phenolphthalein as indicator, matching the colour against the same amount of indicator in a buffer of  $p_H$  8.4. For bicarbonate, titration using methyl-orange as indicator is recommended. The calculated total carbon dioxide from these determinations agrees well with that found by Westerburg's method, in which the solution is acidified with tartaric acid and distilled under reduced pressure, and the gas absorbed in baryta solution. Westerburg's method for bicarbonate (precipitation by baryta in presence of sugar and titrating the excess baryta) is inferior to the methyl-orange titration.

W. A. RICHARDSON.

**Effect of certain Illinois waters on lead.** O. W. REES and A. L. ELDER (J. Amer. Water Works' Assoc.,

1928, 19, 714—724).—A case of lead poisoning in this state led to an investigation of possible causes as, owing to the high mineral content of the water, such cases are rare. The amount of lead present in the water amounted to 0.08 p.p.m., which is below the amount usually considered allowable. It was found that with water containing a sodium bicarbonate alkalinity of 400 p.p.m., increasing amounts of sodium chloride produced a marked effect on the amount of erosion or lead-rusting, but had little or no effect on plumbosolvency. Samples from various parts of the state yielded negative results as to the causes of corrosion, but indicated that a protective layer was quickly formed on the surface of the lead which practically eliminated further corrosion.

C. JEPSON.

**Iodine in drinking waters.** J. B. ORR, W. GODDEN, and J. M. DUNDAS (J. Hyg., 1928, 27, 197—199).—Softening with lime (but not sand-filtration) decreases the iodine in water. For the determination, excess of potassium hydroxide should be added on collection, and the iodine in the water-soluble (10 c.c.) and insoluble residue determined separately.

CHEMICAL ABSTRACTS.

**Analysis of insecticides.** H. FRANÇOIS and L. SEGUIN (Ann. Falsif., 1928, 21, 282—286).—Methods are given for the analysis of liquid insecticides, *i.e.*, those containing formaldehyde and mercuric chloride, picric acid, nicotine and its salts, and garlic. D. G. HEWER.

**Impurities of the atmosphere.** A. J. H. KAM (Chem. Weekblad, 1928, 25, 298—301).—An account is given of methods of sampling and analysing factory exit gases.

S. I. LEVY.

PATENTS.

**Filtering apparatus. [Cleansing sand filters.]** W. PATERSON (B.P. 293,534, 25.5.27).—When a sand filter is cleansed by any form of back-wash the surrounding walls have to be comparatively high to retain the sand which is expanded by the back-flow; at the end of the operation this will leave a large body of water, perhaps dirty, above the sand. In this invention this water is disposed of after the sand has settled by use of collapsible walls, a dwarf wall at one end above which is a removable flap, or merely through openings made in a wall or walls.

B. M. VENABLES.

**Sterilisation of water.** J. MUCHKA (B.P. 290,166, 5.5.28. Austr., 7.5.27).—Water is first chlorinated (with free chlorine or chlorine compounds) and then freed from chlorine etc. by passage through an apparatus containing carbon or masses containing carbon. The apparatus is provided, after the manner of a sand filter, with a reflux device consisting preferably of a jet nozzle and tube, for redistribution and washing of the mass, which is carried out with sterilised water.

B. FULLMAN.

**Purification of liquid trades waste.** R. H. L. PENNELL and F. W. BRACKETT (B.P. 293,927, 29.4.27).—The liquid effluent is passed successively through a screen, a settling tank, and two or more filters, the required proportion of water for recirculation through the factory being drawn off before reaching the final filter.

F. R. ENNOS.