

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

JULY 17 and 24, 1931.\*

### I.—GENERAL; PLANT; MACHINERY.

**Measurement of thermal conductivity.** L. JOLY (Compt. rend., 1931, 192, 797—799).—The arrangement consists of three adjoining chambers, 1, 2, and 3, each 2 m. cube. The wall between 1 and 2 is of cement 75 mm. thick, that between 2 and 3 of the material to be tested. Chamber 2 is maintained at a const. higher temp., say 60°, and the temp. in 1 and 3 give approx. the comparative conductivity of the material. To determine the conductivity more accurately, in chamber 2 is placed, with axis horizontal, a cylindrical box, 40 cm. in diam., with double walls and glass wool between and a thermometer each side, open at one end. This open end is placed against the wall of the material under examination. Inside the box is an electric heater. The current necessary to maintain the temp. inside and outside the box the same is a measure of the conductivity across the section of material enclosed by the open end of the box. C. A. SILBERRAD.

**Diatomaceous earth. I. Structure and properties of diatoms in heat-insulating materials.** W. HUGILL (Trans. Ceram. Soc., 1931, 30, 123—137).—The life history and structure of the various types of diatoms are described in some detail and forthcoming papers on the effect of structure on insulating properties etc. are indicated. J. A. SUGDEN.

**Construction of acid pumps.** H. WINKELMANN (Chem. Fabr., 1931, 4, 195—196).—A detailed description of two types of pump for corrosive liquids: one type depends on the use of non-corrodible alloys, e.g., Si-Fe or bronze; the other on the use of an inert immiscible liquid, such as oil. H. F. GILLBE.

**Mechanism of the atomisation of liquids.** R. A. CASTLEMAN, JUN. (Bur. Stand. J. Res., 1931, 6, 369—376).—Theoretical. The mechanism of the air-stream atomisation of liquids is discussed at length, especially in its relation to the internal-combustion engine. Correlation of previous measurements of the size of the droplets with geometrical and physical considerations and with Rayleigh's work on the rate of collapse of liquid columns shows: (i) that the higher the air speed the smaller is the diam. of the ligaments and the shorter their life, and hence the smaller are the droplets formed; and (ii) that, in accordance with observation, at sufficiently high air speeds the droplets will appear to be formed directly from the bulk of the liquid. H. F. GILLBE.

**Laws of movement of bodies in a fluid.** R. H. KIRKUP (Fuel, 1931, 10, 196—205).—The theory of the motion of spherical and irregularly shaped bodies in a fluid is summarised and applied to the fall of coal

and dirt in air and water. The size classifications of coal suitable for treatment by wet- and pneumatic-separation processes, respectively, are discussed. Whereas a coal sized 1—3 mm. can be treated successfully in a pneumatic separator, the classification 0.5—3 mm. gives unsatisfactory results; the fraction 0.5—1 mm. should therefore be removed and treated separately. A. B. MANNING.

**Complete combustion devices for furnace tube boilers.** Y. SEKIGUCHI (J. Fuel Soc. Japan, 1931, 10, 29—30).

**Recent advances in the production and measurement of high vacua.** S. DUSHMAN (J. Franklin Inst., 1931, 211, 689—750).

**Portable crusher.**—See II. **Cooling on evaporation of solvents.**—See III. **Sulphuric acid [and gas washing].**—See VII. **Continuous kilns.**—See VIII. **Surface pyrometers.** **Heat-resistant alloys.**—See X. **[Turboviscosimeter for] paints.** **Machinery for lacquers.**—See XIII.

See also A., June, 675, **Dichlorodifluoromethane as refrigerant.** 703, **Colorimeter.** **Thermometer for precision calorimetry.** 704, **Fractional-distillation apparatus for gas analysis.**

### PATENTS.

**Furnaces.** J. SCOGNIO (B.P. 346,872, 25.1.30).—In a furnace the main part of the gases pass over the firebridge and down a passage behind it, then pass on rearwardly through an opening in the bottom of the wall bounding the passage. Two sheets of O-containing gas, e.g., air, impelled by steam jets are blown from the front of the furnace and from the firebridge, respectively, and converge at a point at the top of the above-mentioned passage. B. M. VENABLES.

**Destructor furnaces.** C. C. ILVING (B.P. 347,573, 24.10.29).—The furnace comprises a shaft in and from which the clinker is supported and removed by a rotating perforated pyramid; the clinker drops to a lower chamber and is there cooled by the air for combustion, being finally discharged by rotating arms. B. M. VENABLES.

**Heat interchangers for fluids.** V. MICKELSEN, W. M. W. BRUNYATE, and F. E. REBBECK (B.P. 347,904, 17.5.30).—Twisted strips are placed within the tubes of a heat-exchanger, and are rotated either by the flow of fluid along them, or indirectly by means of a propeller in the main pipe and gearing, or by a combination of the two influences. B. M. VENABLES.

**Improving the thermal economy of adsorption processes performed by the aid of bodies of large**

\* The remainder of this set of Abstracts will appear in next week's issue.

**superficial area and with expulsion of the adsorbed substances by means of scavenging media.**

**METALLGES.** A.-G. (B.P. 348,006, 29.10.30. Ger., 11.12.29).—The heat of the products of the regeneration period is saved in a heat accumulator and used for other purposes. The condensed vapours from a previous regeneration may be used as the heat-accumulating substance.

B. M. VENABLES.

**Drying of moist substances.** "SACHTLEBEN" A.-G. F. BERGBAU U. CHEM. IND. (B.P. 347,911, 21.5.30. Ger., 6.12.29).—A rotary drum dryer is provided with both indirect heating by hot gases outside the drum flowing concurrent with the material and with direct heating countercurrent; the latter process is effected in several stages, the hottest gases being introduced some distance up the dryer, *i.e.*, at a point where the material is still very wet. To effect this the lower end of the drum is preferably divided into a number of smaller cylinders grouped round the axis, the axial passage at that end being used to lead the hottest gases up the kiln and extending beyond the small tubes for the material.

B. M. VENABLES.

**Liquid for producing low temperatures for cooling purposes.** A. SCHMIDT, Assr. to I. G. FARBEN-IND. A.-G. (U.S.P. 1,777,183, 30.9.29. Appl., 6.6.29. Ger., 6.7.28).—Mixtures of chlorosulphonic acid (100 pts.) and solid CO<sub>2</sub> (25–100 pts.) are claimed to produce temp. down to  $-70^{\circ}$ .

H. ROYAL-DAWSON.

**[Mercury]-vapour generators.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of A. J. NERAD (B.P. 348,004, 15.10.30. U.S., 16.10.29).—In a vapour generator where the amount of liquid must be reduced to the barest minimum, the Field tubes, which are the heat-receiving elements, are connected to as many drums as convenient. Each drum has three concentric walls and two small annular spaces for the vapour and liquid, respectively, which are conveyed by pipes to or from the upper and lower parts of a single drum of usual construction.

B. M. VENABLES.

**Construction of workshops such as dye-works etc. for the purpose of dispersing mist caused by the generation of steam.** B. BAUER (B.P. 347,411, 17.3.30. Austr., 20.7.29).—Distribution conduits for preheated humidified air are formed in the roof of the factory between upper and lower glass walls forming skylights; preferably also there is a third, uppermost, glass wall protecting the inner upper one from the atm. Rows of distribution conduits alternate with rows of chimney-like outlet ventilators.

B. M. VENABLES.

**Mechanical preparation of pulverised solid materials.** M. MADORE (B.P. 347,914, 23.5.30).—The apparatus comprises a drying circuit, a pulverising circuit, a classifying circuit, and a storage vessel for finely-ground material, *e.g.*, coal. The material is air-borne in the pulveriser and classifier and is transferred elsewhere by worm conveyors. The conveyor which feeds the pulveriser is arranged to run full and to collect, in order: fine coal from storage in excess of that required for consumption, oversize coal from the classifier, and, lastly, new dried coal from another worm-conveyor which also runs full and discharges what it can at this

point, sending the excess back to the dryer and re-filling itself with new undried coal on the way. Thus, once the apparatus is full, the new coal introduced in any period is exactly equal to the consumption of fine coal and the pulveriser and classifier are at all times working with a constant mass of material in circuit.

B. M. VENABLES.

**Feeding solid material to pulverising or like machines.** E. W. GREEN and G. R. UNTHANK (B.P. 347,372, 24.2.30).—The material is fed by a worm attached to a ratchet wheel; the operating awl oscillates through an arc of constant length, but is held out of engagement during more or less of its stroke by a detent adjusted by any suitable governor. The feeder is driven from the pulveriser through a slipping or frangible safety clutch.

B. M. VENABLES.

**Ball or tube mills.** E. G. STONE (B.P. 347,413, 18.3.30).—The mill comprises a number of cylinders parallel to and grouped round the axis of rotation, each one being provided, at one end, with a trap door and spoon-like feeding device operated by tappets when the cylinder is in a suitable position to receive an increment of its charge, and, at the other end, with outlet screens for delivering two sizes of product. The framing of the whole mill may be reciprocated during operation.

B. M. VENABLES.

**Multi-stage apparatus for mixing, stirring, emulsifying, etc.** I. G. FARBENIND. A.-G. (B.P. 347,966, 8.7.30. Ger., 13.7.29. Addn. to B.P. 301,701; B., 1929, 268).—In an apparatus on the principle of the prior patent, a method of introducing the gas in a finely-divided state is described.

B. M. VENABLES.

**Mixing of materials under vacuum.** MODERN CONCRETE DEVELOPMENT Co., LTD., and A. C. KNIPE (B.P. 347,839, 27.3.30).—In a concrete mixer the discharge door remains latched until a vac. has been produced.

B. M. VENABLES.

**Storing and mixing of [semi-fluid] materials.** C. B. THORNE (B.P. 347,628, 23.1.30).—A cylindrical silo is divided into a number of compartments by radial partitions, and materials (*e.g.*, paper pulp) are removed through the bottom of each compartment in succession by a rotating rake which delivers in turn to another rake, a conveyor, an elevator, and a splitting device which cuts out part of the mixed material for delivery and returns the remainder to a distributor and thence to the several compartments of the silo.

B. M. VENABLES.

**Separation of intermixed divided materials.** R. PEALE, W. S. DAVIES, and W. S. WALLACE, Assees. of R. PEALE (B.P. 347,162, 15.10.29. U.S., 31.8.29).—Material, *e.g.*, coal and shale, which varies greatly in size, but not much in sp. gr., is treated on a reciprocating pneumatic table the upward air currents of which are at first sufficiently intense to drive the smaller particles to the top and are later suddenly reduced, stratification in the second zone taking place according to sp. gr. [Stat. ref.]

B. M. VENABLES.

**Filters.** H. S. HELE-SHAW and T. E. BEACHAM (B.P. 347,627, 23.1.30).—The prefilter is contained in a vessel the upper part of which is filled with a gas under pressure; the filter vessel is adjacent and contains a pack filter

with axial outlet for filtrate. When the filter needs cleaning, the pack is removed from its vessel and the pressure gas from the other vessel allowed to enter the axial passage, thus blowing off the cake.

B. M. VENABLES.

**Leaf filters.** E. A. ALLIOTT, and MANLOVE, ALLIOTT & Co., LTD. (B.P. 347,194, 23.1.30).—In a filter comprising a number of leaves in a pressure-tight casing, which when closed forms a reservoir for the prefilter and when opened serves for the discharge of the cakes, the fluid prefilter remaining in the lower part of the casing is drained by filtering through a separate small leaf or by an extension of one or more of the main leaves. Methods of forming the necessary channels and of attaching the leaves are described. B. M. VENABLES.

**Removal of solid particles from liquids.** DORR Co. (B.P. 347,618, 23.12.29. U.S., 24.12.28).—A method of removing sand, in cleaned condition, from, e.g., sewage is described. The total sewage flows through a thickener at such a rate that the organic matter is carried off; the settled sand is raked to the periphery and passes under one wall of the thickener to the lower end of a rake-classifier, by which it is lifted above the liquid level and freed from offensive matter by a water-wash. The classifier and thickener are also in communication at an intermediate level, so that organic matter removed from the sand flows back to the thickener.

B. M. VENABLES.

**Removal of solid particles from liquids.** DORR Co. (B.P. 349,473, 23.12.29. U.S., 24.12.28).—Square thickeners may be used in the process described in the preceding abstract.

B. M. VENABLES.

**Centrifugal separators.** AKTIEBOLAGET SEPARATOR (B.P. 347,993, 26.8.30. Swed., 31.8.29).—A separator in which the supply pipe and receiving vessels are formed in a hinged lid and the delivery spouts in the main body is described.

B. M. VENABLES.

**Continuous-discharge centrifugal machines.** T. BROADBENT & SONS, LTD., and W. HALLITT (B.P. 347,985, 11.8.30).—A centrifugal separator of the type having a helical rake to discharge solid matter has the relative motion produced by differential sun-and-planet gearing, the fixed element of which is a sun-pinion attached to a central shaft within the driving shaft.

B. M. VENABLES.

**Washing of gases and vapours.** M. AURIG (B.P. 347,303, 30.1.30).—The gas is passed together with a washing liquid through the spaces between a number of nested corrugated plates which are spaced far enough apart to prevent the spaces becoming filled with veils of spray, the separation taking place by impingement of the particles of higher sp. gr. upon wetted surfaces. The plates may be assembled in such a way that the passages alternately contract and diverge, producing corresponding compressions and expansions in the gas.

B. M. VENABLES.

**Gas filters for use in gas analysis.** AKTIEBOLAGET CARBA, ASSEES, of PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAG (B.P. 347,170, 14.12.29. Swed., 17.12.28).—Prior to analysis by diffusion or other method a sample of the gas is filtered through crystals of NaCl or similar material; the saturated solution which forms

on the surface of the crystals causes the gas to acquire a const. moisture content and catches the solid impurities, which are allowed to drain away with it. The NaCl filter may be followed by one composed of wadding or other fibrous material. B. M. VENABLES.

**Apparatus for measuring and testing the physical properties of materials.** [Leather-stretching test.] H. BRADLEY, and BRITISH BOOT, SHOE, AND ALLIED TRADES' RESEARCH ASSOC. (B.P. 347,715, 1.2.30).—A disc of patent leather or other sheet material which is to be stretched until it fails by cracking or otherwise is clamped between annular flanges and a load applied to the centre by means of a ball-headed plunger. The plunger is hollow and telescopic and contains a spring; the end opposite the ball is advanced by a screw, and verniers are provided to measure the advance of the ball end, i.e., the stretch, and the contraction of the plunger itself, i.e., the load applied.

B. M. VENABLES.

**Crystallisers.** W. G. HALL (B.P. 348,950, 8.7.30. U.S., 31.7.29).—See U.S.P. 1,769,779; B., 1931, 183.

[Suspended] furnace roofs. H. RÖMER and G. KARREBERG (B.P. 349,266, 2.5.30).

**Refrigerating apparatus.** METERS, LTD., and C. C. APPLETON (B.P. 349,179, 12.3.30).

**Burning of fuel in furnaces.**—See II. Cement kiln.—See IX. Gas purification.—See XI.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Classification and development of carbonaceous minerals.** H. BRIGGS (Proc. Roy. Soc. Edin., 1931, 51, 54—63).—The points obtained by plotting the O content against the C content (on the ash-, moisture-, S-, and N-free basis) for a series of coals from lignites to anthracites lie within a narrow continuous band, the median line of which represents the gradual change in rank of the mineral with age. A similar procedure with other types of carbonaceous minerals, e.g., cannel, paraffin shales, torbanites, and petroleums, shows that each type is characterised by a distinct "development line," which may be utilised in classifying such minerals. Each line is directed towards an end-product of definite C:H ratio, e.g.,  $C_{5n}H_{6n}$  for the paraffin shales. The further from the coal line in the direction of the origin of the chart an analysis is situated, i.e., the higher the H proportion, the more suitable is the mineral as a source of oil.

A. B. MANNING.

**Behaviour of solid fuels during oxidation.** IV. **Combustion of the banded ingredients—vitrain, clarain, durain, and fusain.** B. MOORE (Fuel 1931, 10, 205—211; cf. B., 1927, 130).—The four banded ingredients of a coal seam differed considerably in "spontaneous heating tendency," "relative ignition temp.," and "combustible capacity," as determined by the method described previously (B., 1925, 486). No correlation could be observed between these results and the composition of the ingredients as shown by their proximate and ultimate analyses. Coal dusts rich in fusain may be regarded as less liable to undergo spontaneous heating during storage than those resulting from the pulverisation of the whole coal, and should be

suitable, from the ignition temp. and combustible capacity aspects, for use as pulverised fuel.

A. B. MANNING.

**Gray-King high-temperature coal assay.** J. BROWN (J.S.C.I., 1931, 50, 190 T).—The plugging of the tar tube is greatly facilitated by cutting off 95 mm. of the limb containing the plug. This is rejoined to the main tube, after filling, by a short rubber tube.

**Caking, swelling, and expansive pressure of coking coals.** G. LAMBRIS (Brennstoff-Chem., 1931, 12, 181—187).—Determinations of coke yield, caking index (Meurice), swelling power (B., 1929, 270), expansive pressure (Korten-Damm and Koppers; cf. B., 1931, 373), softening point, bitumen extractable by  $C_6H_6$  under pressure, gas evolution (B., 1928, 802; 1929, 666), plasticity (J.S.C.I., 1921, 40, 193 T), and yields of low-temp. tar (Fischer's Al assay apparatus), have been made on 12 coking coals. Apart from a rough parallelism between the swelling powers and expansive pressures of 8 of the coals, no definite correlation could be observed between any of these properties. Of the strongly caking coals some neither swelled nor exerted any expansive pressure on coking; all the swelling coals caked. Although all the coals exerting pressure on coking were both swelling and caking coals, the converse was not necessarily true. On heating the coals in N at 200° the expansive pressures fell to zero within 4—8 hr.; within the same period the swelling powers fell 10—40%, whilst the caking indices either rose or fell slightly and with continued heating the caking indices gradually fell. The bearing of these results on the behaviour of coal in the coke oven is discussed. In general, coals which have been preheated until they no longer exert any expansive pressure also exhibit no "plasticity" when examined by Foxwell's method. Storage of the powdered coals in vessels containing air produced after three months a marked rise (75—90°) in the softening point, but had little effect on the caking index, swelling power, or expansive pressure.

A. B. MANNING.

**Briquetting of brown-coal coke.** F. SEIDENSCHNUR (Brennstoff-Chem., 1931, 12, 191—193).—By the carbonisation of brown coal in a current of inert heating gas at about 380° a coke was obtained containing 1—2% of residual tar and capable of forming satisfactory briquettes. The strength of the briquettes varied both with the tar and the  $H_2O$  content of the coke, the optimum  $H_2O$  content being 12%. A rotary briquetting press working at a pressure of 1800 kg./sq. cm. was used.

A. B. MANNING.

**Determination of the after-coking heat of coke.** G. AGDE and F. SCHIMMEL (Stahl u. Eisen, 1931, 51, 460—462).—The heat required to expel the volatile matter from coke can be calc. by deducting from the total heat of combustion of the coke the heat of combustion of the residue obtained by again coking the material at a higher temp. than that at which it was originally produced and the heat of combustion of the volatile material produced in the second coking. The last-named is calc. from the quantity and composition of the coke before and after re-coking; in these analyses N is determined by combustion of the coke with  $PbCrO_4$  in a current of  $CO_2$ , removal of any O with red-hot Cu

gauze and  $CO_2$  with KOH, and measurement of the N in a nitrometer.

A. R. POWELL.

**Apparatus for the determination of the reactivity of coke.** W. J. MÜLLER and E. JANDL (Brennstoff-Chem., 1931, 12, 187—191; cf. B., 1930, 647).—The following modifications have been made in the apparatus of Agde and Schmitt: a constant-pressure device has been attached to the  $CO_2$  reservoir; a specially designed sensitive stopcock has been provided to regulate the flow of gas; and the two nitrometers have been replaced by a single one, comprising a storage vessel and a measuring tube, and so designed that the flow of gas can be maintained const. throughout the whole experiment. Complete removal of the air from the apparatus is accelerated by evacuating and filling with  $CO_2$  several times before starting the determination. The results obtained by the modified apparatus show considerably less fluctuation than the results of previous determinations.

A. B. MANNING.

**Portable equipment for crushing and quartering samples of coal, coke, or other lumpy materials.** E. S. PETTYJOHN (Ind. Eng. Chem. [Anal.], 1931, 3, 163—164).—Details of construction of a portable steel quartering hopper and a portable steel hand crusher are given. The instruments are designed to facilitate reduction in the wt. and size of lumps of material when working away from the laboratory.

E. S. HEDGES.

**Light creosote from beech wood.** J. GADAMER and G. WESTERBURG (Arch. Pharm., 1931, 269, 307—326).—PhOH, *o*-cresol, guaiacol, creosol, homoveratrole, and ethylguaiacol (*benzoate*, m.p. 65°, and its *nitro*-derivative, m.p. 124°) were identified. Two substances (*semicarbazone*,  $C_8H_{13}ON_3$ , m.p. 230°, and *oxime*,  $C_{16}H_{15}O_2N$ , m.p. 160°, respectively) were also isolated.

A. A. LEVI.

**Oils and schists of S. Romedio.** G. ROBERTI (Annali Chim. Appl., 1931, 21, 116—127).—Investigation of these schists and of the oil they contain shows that the ordinary methods of desulphuration used in petroleum refineries are incapable of removing the S compounds present in the oil. The conversion of the oil into ichthyobenzine, ichthyol, etc. is recommended.

T. H. POPE.

**Separation of *n*-octane from petroleum by distillation and crystallisation.** R. T. LESLIE and S. T. SCHICKTANZ (Bur. Stand. J. Res., 1931, 6, 377—386). A 100—130° fraction from an Oklahoma crude oil contains over 1% of *n*-octane, which is separated by groups of fractional distillations alternating with fractional crystallisations. The *n*-octane isolated is of over 94% purity. Apparatus for the distillations and for the crystallisations at low temp. are described.

G. DISCOMBE.

**Determination of the toluene content of a mid-continent petroleum.** J. H. BRUUN, R. T. LESLIE, and S. T. SCHICKTANZ (Bur. Stand. J. Res., 1931, 6, 363—367).—The distillates from an Oklahoma crude oil of boiling range 70—130° contain 0.3% PhMe. By fractional distillation the PhMe is concentrated in the fraction 98—99°, but the highest PhMe percentage (32%) is found in that of b.p. 107—108°. On nitration of the distillates with a mixture of 10 vols. of  $H_2SO_4$

(*d* 1.84), 2 vols. of oleum (23% SO<sub>3</sub>), and 15 vols. of HNO<sub>3</sub> (*d* 1.50) the PhMe is converted quantitatively into dinitrotoluene (cf. A., 1896, i, 269; 1901, i, 441; B., 1931, 187).

G. DISCOMBE.

**Components of petroleum distillates. II. Aniline-point determination of low-boiling paraffin-naphthene mixtures.** A. SCHAARSCHMIDT, W. HOFMEIER, and H. LEIST (Z. angew. Chem., 1930, 43, 954—956).—The aniline-point method for determining the naphthene content of binary mixtures of methylcyclohexane, cyclohexane, *n*-hexane, and *n*-heptane, may be employed, using the formula  $N = 100(A - A_m)/(A - a)$ , where *N* is vol.-% naphthene, *A* and *a* are the aniline points of the components (*A* being < *a*), and *A<sub>m</sub>* is the aniline point of the mixture. The accuracy of the method is about ± 1%, but falls rapidly with falling b.p. of the components. The method may also be applied to ternary and quaternary mixtures of these hydrocarbons.

E. S. HEDGES.

**Detonation characteristics of some aliphatic olefine hydrocarbons.** W. G. LOVELL, J. M. CAMPBELL, and T. A. BOYD (Ind. Eng. Chem., 1931, 23, 555—558).—The knocking tendencies of 25 unsaturated hydrocarbons in admixture with petrol have been compared with that of PhNH<sub>2</sub>, employing a bouncing-pin method. The compounds chosen were of the straight- and branched-chain types with one and with two double linkings. Very great differences in knocking tendency were observed even among isomerides, the position of the double linking having considerable influence. With straight-chain olefines the tendency to knock rose in an homologous series with the length of the saturated chain. In an isomeric series the tendency decreased progressively with centralisation of the double linking. The tendency to knock is roughly determined by the length of the longest saturated C chain. With branched-chain olefines the tendency to knock decreases with the introduction of a double linking, and it is related both to the position of this linking and to the branched structure of the mol.

H. INGLESON.

**Chemical nature of Ural benzine and its catalytic aromatisation.** N. D. ZELINSKI and J. K. JURIEV (Bull. Acad. Sci. U.S.S.R., 1930, 851—861).—Ural benzine, a works' distillate which had been treated with H<sub>2</sub>SO<sub>4</sub>, contained no thiophen, but a high proportion of compounds of mercaptan and sulphide type. These were removed by treatment with M'OH, prolonged shaking with HgCl<sub>2</sub>, filtration, and fractional distillation over molecular Cu recently reduced by means of H. The aromatic hydrocarbons were determined by measuring the diminution in vol. when shaken with oleum (10% SO<sub>3</sub> for the lower fractions, 7% SO<sub>3</sub> for the higher ones); the proportion of total aromatic hydrocarbons was 33%. Similar determinations were made of the aromatic hydrocarbons in the various fractions after these had been subjected to catalytic dehydrogenation by passing their vapours over platinised C at 300—310°. Calc. on the original benzine, the total proportion of aromatic hydrocarbons thus formed amounts to 16.75%. Hence this benzine is capable of yielding 50% of aromatic hydrocarbons, the proportions—

for some of its fractions being considerably higher. Analysis of the residue indicates the absence of any further cyclic compounds, such as pentamethylene hydrocarbons. Surachani benzine contains no aromatic hydrocarbons, but yields higher proportions than the Ural product after catalytic dehydrogenation.

T. H. POPE.

**Production of petroleum hydrocarbons from carbon monoxide and hydrogen at the ordinary pressure.** E. R. MÜLLER [with S. KALLENBERG] (Ingen. Vetensk. Akad. Handl., 1930, No. 106, 39 pp.; Chem. Zentr., 1931, i, 1205).—A description of the application of Fischer and Tropsch's process. 245 g. of pine charcoal (88.17% C) gave 20 g. of gas oil (not including C<sub>2</sub>H<sub>6</sub> and C<sub>2</sub>H<sub>4</sub>), 26 g. of benzine and 14 g. of petroleum hydrocarbons, and 15 g. of vaseline.

A. A. ELDRIDGE.

**Determination of sulphur in liquid fuels. I.** F. GARELLI and B. SALADINI (Annali Chim. Appl., 1931, 21, 136—140).—The procedure recommended is as follows: 10 c.c. of H<sub>2</sub>O are pipetted into the calorimetric bomb, the Pt or quartz crucible containing the igniting Fe wire coil being charged with a few crystals of NH<sub>4</sub>NO<sub>3</sub> and with a definite wt. of the liquid fuel from a Sprengel pyknometer. The bomb is at once closed, the compressed O<sub>2</sub> introduced, and, after the bomb has been placed in the calorimeter, the fuel ignited. After 1 hr. the gas is allowed to escape from the bomb and the H<sub>2</sub>SO<sub>4</sub> formed determined as usual.

T. H. POPE.

**Oxidation of paraffin oil by air.** G. ARDITI (Compt. rend., 1931, 192, 944—946).—Air was bubbled for varying periods and at varying temp. through a paraffin oil free from unsaturated compounds and from S. Oxidation did not occur in 1500 hr. at temp. up to 85°, but was apparent after 12, 3, and ½ hr. at 110°, 123°, and 150°, respectively, and markedly at room temp. under ultra-violet light. The free acidity, total acidity, and viscosity for various periods up to 82 hr. at 110°, 123°, and 150° vary substantially similarly.

C. A. SILBERRAD.

**Use of lead tetraethyl in internal-combustion motors.** P. DIDIER (Chim. et Ind., 1931, 25, 803—810).—EtOH and aromatic hydrocarbons act as diluents in increasing the possible compression ratio. The efficacy of EtOH is due to its high latent heat of vaporisation. The importance of PbEt<sub>4</sub> to France in case of war is stressed. A French Government committee, on the strength of the British committee's interim report, has advised that "ethyl" containing 0.08% PbEt<sub>4</sub> is unsuitable for use in towns.

C. IRWIN.

**Stabilisation of [lubricating] oils for chronometers.** P. WOOG, (MLLE.) E. GANSTER, and J. GIVAUDON (Compt. rend., 1931, 192, 923—925; cf. B., 1926, 37).—The possible stabilising effect of various "antioxigens" on sheep's-foot oil (as a typical triglyceride lubricating oil) has been examined. Addition of, e.g., 0.5% of β-naphthol causes a marked decrease in the rate at which acidity is produced in the air, provided the oil is in darkness, but in light an increase. The effect of darkness can, however, be obtained by adding a red dye, e.g., Soudan I, II, or III, Scarlet 2R, etc. Brass on which a drop of oil thus stabilised has been

placed shows no sign of attack by the time it has been blackened by the untreated oil. C. A. SILBERRAD.

**Movement of bodies in a fluid.**—See I.  $H_2SO_4$  [and gas washing]. **Absorption of  $H_2S$ .**—See VII. **Resins from low-temp. tar.**—See XIII.

See also A., June, 677, Adsorption of H on charcoal. 678, Sorption of H by Pt metals. 689, Explosions of  $C_2H_2$ -electrolytic gas mixtures. **Flames and their propagation.** 690, Behaviour of antiknocks. 704, Gas analysis by fractional distillation. 705, Slow-combustion pipette. 708, Japanese black shale. **Origin of Japanese petroleum.** 751, Organic Pb compounds.

## PATENTS.

**Pneumatic coal-cleaning plants.** A. F. BURGESS. From ROBERTS & SCHAEFER CO. (B.P. 347,578, 26.11.29).—A number of pneumatic shaking tables are arranged in series in such a way that dusty air from one table or set is roughly cleaned in a cyclone and supplied to another table or set. B. M. VENABLES.

**Burning of fuel in furnaces.** STOCKHOLMS AKTIEBOLAGET PRIVAT (B.P. 347,174, 20.12.29. Ger., 17.1.29).—Combustion is facilitated by blowing incandescent particles of fuel, together with hot gases and a part of the air for combustion, at high velocity from below into the layer of fuel on the front portion of the grate. The incandescent fuel particles may be obtained by sucking off gases laden therewith from a suitable part of the fire space or smoke channels; in addition, particles falling through the grate or other coke dust may be mixed with these gases. Furnaces for carrying out the process are described; they embody also means for preheating the air for combustion and for predrying the fuel. A. B. MANNING.

**Heating of coal or the like.** F. PUENING, Assr. to KOPPERS Co. (U.S.P. 1,781,079, 11.11.30. Appl., 3.9.27).—Coal for coking purposes is dried and preheated at 100–300° in externally heated vertical conduits. The elevator conveying the raw coal to the top of the preheater chamber also conveys the heated coal to the top of the hot coal bin, the arrangement being such that one end of each elevator bucket is charged with raw coal and the other end with heated coal, whereby part of the sensible heat of the latter is utilised in evaporating the moisture from the raw coal. A. B. MANNING.

**Combustion and gasification of carbonaceous materials.** F. C. GREENE (B.P. 347,575, 25.10.29).—The fuel is progressively advanced through zones wherein its temp. is progressively raised until it reaches the hot exposed surface of the mass, over which air is passed. The air current is so adjusted that the  $CO_2$  first produced by combustion of the fuel is reduced to CO in passing over the incandescent material. In one form of apparatus for carrying out the process the fuel is supported on a frusto-conical surface carrying laterally extending spiral platforms for effecting the required movement of the fuel, over the surface of which the air passes in a downward direction. Provision may be made for separately withdrawing the light distillates evolved in the zones of intermediate temp. A. B. MANNING.

**Distillation of solid carbonaceous material.** H. L. DOHERTY (U.S.P. 1,781,871, 18.11.30. Appl., 8.5.25).—The material is distilled in an upward current of hot inert gases, which, together with the volatile products of distillation, are withdrawn through a number of gas-offtakes distributed over the cross-section of the retort in such a manner as to ensure a uniform distribution of the heating gases in the charge of fuel, and so avoid "channeling." The fuel is charged into the retort through a corresponding number of separately controlled charging conduits. A. B. MANNING.

**Cooling of coke and obtaining a nitrogen-hydrogen mixture.** D. TYRER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 347,601, 28.1.30).—The hot coke is charged into a large cooling chamber into which water is sprayed at a depth where the coke has cooled to about 550°, while air is injected at a higher level, where the temp. is about 900°. The air supply is so regulated that after the catalytic conversion of the CO produced into its equivalent of  $H_2$ , and after purification, the gas mixture formed consists of  $N_2$  and  $H_2$  in the stoichiometric ratio of 1:3. A. B. MANNING.

**Production of water-gas.** S. J. McAULIFFE, Assr. to FULLER LEHIGH Co. (U.S.P. 1,780,759, 4.11.30. Appl., 21.9.21).—Pulverised coal and steam are injected horizontally into a narrow vertical retort having side walls of relatively large area. These walls are heated to 1500–1600° by flues communicating with a combustion chamber disposed below the retort and designed to burn pulverised fuel. A. B. MANNING.

**Water-gas generators.** A. BREISIG (B.P. 347,520, 4.6.30. Austr., 5.6.29).—In the distillation and gasification of bituminous fuel by means of alternately working water-gas generators, the hydrocarbon vapours generated in the distillation zone during the blast period in the gasification zone are condensed in a zone of cold fuel surmounting the distillation zone. The mixed water-gas and distillation gases are preferably withdrawn through a duct which extends to the centre of the fuel column at the top of the distillation zone. A. B. MANNING.

**Desulphurisation of gas.** F. W. WERNER and E. T. JOHNSTON (U.S.P. 1,779,024, 21.10.30. Appl., 16.9.26).—The gas is treated with CaO at 550°, preferably in a rotating drum heated externally by flue gases. The S compounds are decomposed with the formation of CaS and the liberation of  $H_2$ , which, in the presence of the CaO, interacts with the  $N_2$  in the gas to produce  $NH_3$ . A. B. MANNING.

**Production of acetylene.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 347,493, 8.5.30).—Gaseous hydrocarbons are submitted to incomplete combustion, preferably at 700–1100°, under pressures of 0.5–5 atm., in the presence of a catalyst containing free Si. A. B. MANNING.

**Apparatus for generating acetylene.** I. G. FARBENIND. A.-G. (B.P. 347,537, 28.6.30. Ger., 1.8.29).—The carbide receptacle is flared downwardly and its lower part, which projects into the generating chamber, is designed as a cage the carbide in which is sprayed with  $H_2O$  in considerable excess. The spraying  $H_2O$  is circulated by a pump which draws it, roughly freed

from CaO sludge, from the generating chamber, and delivers it to the spraying device. A. B. MANNING.

**Distillation of (A, B) tar and (B) like hydrocarbons.** BARRETT CO., ASSEES. OF S. P. MILLER (B.P. 347,107 and 347,240, [A] 16.1.30, [B] 18.12.29. U.S., [A] 16.1.29, [B] 18.12.28).—(A) Tar is brought into intimate contact with hot distillation gases, *e.g.*, coke-oven gases, in a non-externally heated still, under such conditions that a high-m.p. pitch is produced. The enriched gases leaving the still are passed through a condenser wherein they are brought into indirect contact with the incoming tar, which is thereby preheated sufficiently to distil off part of the light oils therein. (B) Intimate contact between the hot gases and the tar is brought about by providing horizontal rollers which dip into a body of tar maintained at the bottom of the still, and which are rotated at a relatively high speed, and thereby maintain a fine spray within the still. A. B. MANNING.

**Rotary retorts for treatment of oil shale and the like.** T. M. DAVIDSON (B.P. 347,121 and 347,142, 17.10.29).—A rotary retort is provided with a longitudinal vapour-outlet pipe situated in that part of the cross-section of the retort which is remote from the part where the material piles up during the rotation; the outlet pipe is non-circular in cross-section, and serves also as a support for reciprocating scrapers operating upon the interior of the pipe and of the rotating retort. Forms of gastight joints between the rotating and fixed parts are described. B. M. VENABLES.

**Treatment of shale or the like.** W. H. HAMPTON (U.S.P. 1,778,515, 14.10.30. Appl., 16.12.20).—Ground shale is digested with heavy oil at 260–370°, and the solid matter is then separated by means of a centrifuge. Vapours evolved during the digestion are separately condensed. The solid is finally destructively distilled to recover additional bituminous matter. A higher yield of better-quality oil is obtained by this treatment than by direct destructive distillation. T. A. SMITH.

**Cracking of [hydrocarbon] oils.** E. C. HERTHEL and E. W. ISOM, ASSRS. TO SINCLAIR REFINING CO. (U.S.P. 1,781,390, 11.11.30. Appl., 7.1.25).—Cracking stock is pumped from a bulk-supply tank upwardly through a vertically arranged tubular heater and back to the tank, the oil being maintained at cracking temp. throughout the cycle. The vapours escaping from the tank are refluxed in direct contact with fresh charging stock, and the reflux and admixed fresh oil are together forced upwardly into the oil circulating through the lower part of the heater to assist circulation. H. S. GARLICK.

**Crude oil and natural gas separator.** M. H. KOTZEBUE, ASSR. TO TRUMBLE GAS TRAP CO. (U.S.P. 1,782,783, 25.11.30. Appl., 22.9.24).—A vertical vessel is fitted with a float-controlled valve by means of which a supply of oil is retained in the lower portion of the separator. A gas offtake extends down the separator and is surrounded by a spiral pathway on to which the oil and gas from the well are delivered. Good separation of oil from gas takes place as a result of the oil flowing in thin films down the pathway. An inclined screen is interposed in the gas offtake to assist in the removal of entrained oil. T. A. SMITH.

**Conversion of hydrocarbons into those of lower b.p.** J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 347,727, 2.1.30).—The hydrocarbons are treated with free halogen, preferably Cl, and the products obtained, admixed, if desired, with a further quantity of the initial material and/or with at least part of the H halide formed during the first stage of the process, are subjected to the action of activated Al at 150–250°.

A. B. MANNING.

**Conversion of high-boiling hydrocarbon oils.** E. E. BARTELS, ASSR. TO STANDARD OIL CO. (U.S.P. 1,782,160, 18.11.30. Appl., 12.7.26).—Oil heated to the inversion temp. under a pressure of 4–5 atm. is passed through a confined passage at a rate sufficient to prevent the deposition of coke. The vapour passes through a dephlegmator countercurrent to a stream of condensate and is then condensed. Part of the condensate is returned to the dephlegmator and the vapours leaving the condenser serve to preheat the fresh oil.

J. A. SUGDEN.

**Cracking of hydrocarbon oils.** R. J. DEARBORN, ASSR. TO TEXAS CO. (U.S.P. 1,783,010, 25.11.30. Appl., 19.1.28).—Oil is passed through a heating coil of restricted cross-section, where it is raised to substantially cracking temp., and delivered into an enlarged cracking drum. Further oil is simultaneously cracked in the vapour phase at a relatively high temp. and the cracked products are passed in direct contact with the oil in the cracking drum, thereby maintaining its temp. and effecting liquid-phase cracking. H. S. GARLICK.

**Cracking of hydrocarbon oil.** J. G. HAWTHORNE, ASSR. TO W. M. CROSS (U.S.P. 1,782,686, 25.11.30. Appl., 9.7.27).—Oil is raised to a conversion temp. in a primary heating stage and the vaporised products are reheated and superheated by passage through conduits in a reheating stage, and then cooled to a predetermined temp. selected to effect separation of the heavier and lighter fractions. The heavier fractions are discharged from the system as liquid, whilst the lighter are refluxed and condensed, the reflux condensate being recycled to the primary heating stage. A balanced pressure is maintained between the material undergoing superheating and the combustion gases, the heat of which is used to heat in turn the primary heating stage.

H. S. GARLICK.

**Cracking and processing of petroleum hydrocarbons.** J. C. BLACK, ASSR. TO GASOLINE PRODUCTS CO., INC. (U.S.P. 1,782,676, 25.11.30. Appl., 17.5.24. Renewed 17.4.30).—A primary supply of oil is heated in a furnace to cracking temp. and discharged, under sufficient pressure to prevent material vaporisation, into a C-precipitating and reaction chamber wherein is introduced a secondary supply of oil heated in a tubular heating element in the same furnace to incipient cracking. The temp. of the mixed hydrocarbons is sufficient to crack the secondary supply of oil and any carbonaceous matter is deposited from the liquid mixture in a soft granular form. The heated hydrocarbons are then fractionated and condensed, those fractions suitable for re-cracking being returned to the system. Alternatively, the vapours from the secondary supply of oil may be led direct to the fractionator, and only the

unvaporised portion discharged into the reaction chamber.

H. S. GARLICK.

**Cracking of hydrocarbons.** J. E. BELL and E. W. ISOM, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,782,056, 18.11.30. Appl., 12.11.24).—Cracking takes place in a series of stages of a pressure still in which the temp. is progressively lowered and the heat-exchanging surface is increased in proportion. The vapours are refluxed with fresh oil in such a way that the lightest oils pass off, but the heavier oils mix with the fresh oil passing to the first stage. Thus the concentration of tarry matter in the first (highest-temp.) stage of the cracking is reduced and tar deposition on the heating surface is prevented. The concentration of tar progressively increases in the succeeding cooler stages and is drawn off. The heavier cracked oils carried along with the fresh oil to the first stage thus undergo repeated cracking and only the lightest oils pass from the dephlegmator to the condenser.

J. A. SUGDEN.

**Cracking of hydrocarbons.** H. K. ROGERS, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,781,884, 18.11.30. Appl., 11.10.28).—The cracked vapours from the pressure still are passed into a fractionating tower at an intermediate point so that stripping of the oil may take place in the lower part. The vapours passing from the condenser proper are further condensed by undergoing indirect heat exchange with a scrubbing medium in which the remainder of the uncondensed vapours have been absorbed later. This heated medium, containing absorbed vapours, passes to the stripping tower while the remainder of the uncondensed cracked vapours leaving the heat-exchanging condenser are recovered in the scrubber.

J. A. SUGDEN.

**Splitting of hydrocarbons such as acetylene or the like.** J. MACHTOLF, Assr. to C. HOSTMANN-STEINBERG'SCHE FARBENFABR. G.M.B.H. (U.S.P. 1,782,540, 25.11.30. Appl., 9.12.26. Ger., 11.12.25).— $C_2H_2$  alone or mixed with other hydrocarbons is subjected to thermal dissociation in a suitable container. The product, including  $H_2$  and soot, is passed through a separator and a portion of the gas still containing a certain quantity of soot in suspension is repeatedly circulated through the splitting chamber and separating device until the chamber is entirely free from soot and only pure gas is being circulated.

H. S. GARLICK.

**Manufacture of unsaturated hydrocarbons.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 347,620, 17.1.30).—Liquid or solid saturated hydrocarbons boiling above  $180^\circ$  are oxidised, preferably in the liquid phase, e.g., by blowing with air at  $140$ – $160^\circ$ , and the products are subjected to a sufficiently high temp. to cause them to split off  $H_2O$  and/or  $CO_2$ . Both stages of the process may be carried out catalytically. The oxidation products may be freed from carboxylic acids, e.g., by treatment with alkali, or may be separated into fractions sol. and insol. in light petroleum, before being heat-treated.

A. B. MANNING.

**Apparatus for distilling hydrocarbon oil.** H. G. SCHNETZLER, Assr. to STANDARD OIL Co. (U.S.P. 1,779,918, 28.10.30. Appl., 10.3.27).—Condensed vapours from a fractionating column are received into a vessel fitted with a weir. The liquid flowing over the weir is distributed

in several compartments, from any one of which it may be returned to the fractionating column for refluxing purposes or passed to storage. The compartments are so arranged that the flow over the weir is divided in the ratio 2 : 8 : 4 : 3, and one or more of these portions may be returned as reflux.

T. A. SMITH.

**Treatment of (A) [hydrocarbon] oil, (B) hydrocarbons.** J. DELATTRE-SEGUY, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,782,809 and 1,782,811, 25.11.30. Appl., 28.6.26).—(A) In the distillation of hydrocarbon oils, these are caused to flow continuously, by gravity in a thin film, over an unheated inclined surface. The heat necessary for distillation is supplied by radiation to the upper surface of the oil film by means of a heating surface placed above and spaced away from the upper surface of this film. The inclined surface and the heating surface are situated in a closed chamber provided with means for removing and condensing the evolved vapours. (B) The oil is passed in a continuous film (1 in. thick or less) by gravity along an inclined shallow channel in a cracking zone (or shell) heated to at least  $455^\circ$  at a pressure greater than 100 lb./in.<sup>2</sup> Heat is applied to the upper surface of the oil film in order to avoid deposition of C, either by heating the upper portion of the shell externally with furnace gases, or by passing a molten material along a trough within the shell placed immediately above the oil channel.

R. W. L. CLARKE.

**Distillation of petroleum oils.** BARRETT Co., Asses. of A. A. MACCUBBIN and J. ZAVERTNIK (B.P. 346,407, 30.9.29. U.S., 15.12.28).—Petroleum is continuously distilled by passing it through a pipe coil where it is heated to a temp. sufficient to vaporise a large proportion of the lighter fractions, and is then discharged into a vapour-separating chamber at approx. atm. pressure, with the resultant separation of oil vapour and the production of a residual oil. The residual oil, which may be a lubricating oil stock, is drawn off while still hot and subjected to a high vac. in a vac. still so that approx. all the oil distillable at that temp. is vaporised without additional heat.

H. S. GARLICK.

**Distillation of material and cracking of oil.** F. T. SNYDER (U.S.P. 1,781,934, 18.11.30. Appl., 8.6.25).—Solid material (coal, garbage, sawdust, oil shale, etc.) is distilled in a producer-like retort where the necessary heat is provided by combustion of the solid matter remaining after the material has passed the distillation zone. The vapours leaving the retort are cooled and the heavier oils, which are condensed, are passed back to the middle zone of the retort, where they undergo cracking. Free C which is deposited on the fuel passes down into the combustion zone. The light oils from the cooler are condensed and the uncondensed vapours undergo heat exchange in the cooler with the hot vapours from the retort. These reheated vapours are passed into the top of the retort, where they preheat the fresh fuel. This precaution prevents the condensation of oils upon the cold fuel and the consequent "sticking" of the descending charge.

J. A. SUGDEN.

**Making motor fuel.** B. FIXMAN (U.S.P. 1,781,872, 18.11.30. Appl., 18.11.26).—Raw heavy oil passes through a pressure still and is sprayed in the liquid



phase to meet a stream of recycled oil in the vapour phase. The mixture of oils is passed, at a pressure sufficiently reduced to form a completely gaseous phase, into an electrostatic precipitator, where solid particles of C are removed. The vapours are then cooled and the light oils pass to a condenser, while the unconverted fraction is collected. According to the rate at which this fraction is collected, it is automatically pumped at a higher pressure through a second pressure still and superheater and becomes the recycle oil supply. A heat balance is maintained between the once- and twice-treated oils so that there is no fall in temp. in the condenser as the light oils distil off. J. A. SUGDEN.

**Treatment of hydrocarbons.** R. CROSS, Assr. to CROSS DEVELOPMENT CORP. (U.S.P. 1,782,808, 25.11.30. Appl., 20.1.26).—A process for refining cracked and other hydrocarbon vapours consists in fractionating the vapours and passing those of the lighter fractions first through towers containing Cu or oxides and salts of metals having an affinity for S, whereby desulphurisation is effected, and then through towers containing highly adsorbent material such as fuller's earth or bentonite, which effect purification and polymerisation. The purified vapours then pass to rectifying and condensing stages. R. W. L. CLARKE.

**Treatment of [removal of sulphide impurities from] hydrocarbons.** D. L. THOMAS (U.S.P. 1,781,826, 18.11.30. Appl., 23.7.27).—Sulphide impurities etc. are removed from oils, preferably in the vapour phase, by exposure to the combined action of atomic H (produced by electric arc) and a catalyst such as Sn or Pb. The gases after treatment pass over an Fe<sub>2</sub>O<sub>3</sub> purifier to remove the H<sub>2</sub>S. J. A. SUGDEN.

**Dewaxing of hydrocarbon oils.** A. R. GREIG, Assr. to TEXAS CO. (U.S.P. 1,782,467, 25.11.30. Appl., 20.12.27).—The economy of operation of centrifugal apparatus engaged in the separation of wax from chilled hydrocarbon oil is markedly improved by interrupting the flow of wax-bearing oil periodically to the centrifuge, when the capacity of the apparatus becomes reduced by the accumulation of solid wax and ice. A heating medium or steam is then introduced into the bowl of the machine by the usual oil inlet. The normal process is resumed when the accumulated solids have been removed from the working surfaces of the apparatus. R. W. L. CLARKE.

**Dewaxing of oils.** E. F. BURCH, Assr. to CREW LEVICK CO. (U.S.P. 1,782,028, 18.11.30. Appl., 14.3.27).—The oil containing precipitated wax is fed into a centrifuge so arranged that the wax is collected in an outer chamber and is separated from the incoming mixture by a chamber through which the separated oil is passed. Thus the separated wax may be melted and allowed to run out by suitable steam-heating, without any appreciable amount of heat reaching the incoming mixture and so redissolving some of the wax. J. A. SUGDEN.

**Recovery of waste clays [from petroleum refining].** W. D. RIAL and W. R. BARRATT, Assrs. to RICHFIELD OIL CO. OF CALIFORNIA (U.S.P. 1,782,744, 25.11.30. Appl., 5.1.27).—Clay from petroleum purification is washed with naphtha to remove oil, and then

with an "acid oil," obtained from acid sludge by separating its H<sub>2</sub>O-sol. constituents, to remove oil-insol. compounds, such as gums and resinoids. After a further washing with naphtha, the clay is heated to 82–204° and treated with superheated steam to eliminate odour and hydrate the clay. W. J. WRIGHT.

**Lubricant.** G. BROWN and F. LEVY (B.P. 346,379, 9.1.30).—Hg (1 pt.) is intimately mixed with petroleum jelly (2 pts.) to form a bearing lubricant, which may be subsequently rendered fluid by the addition of up to 10 times the wt. of a fluid mineral oil. [Stat. ref.] H. S. GARLICK.

**Engine lubricant.** R. E. CLOUGH, Assr. to CASTOR-LUBE REFINING CO. (U.S.P. 1,782,501, 25.11.30. Appl., 13.4.27).—Castor oil is gradually heated up to, and maintained at, not above 311° (at ordinary pressure) until not more than 4.2 wt.-% has been distilled off, and is mixed with mineral oil (20–98%). E. LEWKOWITSCH.

**Lubricating compositions.** A. E. WHITE. From PANOLEUM CO. (B.P. 347,574, 25.10.29).—Compositions of desired consistency and m.p. are produced by adding to a lubricating oil 5–15% of an amide or anilide of a higher fatty acid, e.g., stearanilide, and 3–5% of a metal soap. A. B. MANNING.

**Mechanically-clinkered gas generators or producers.** HUMPHREYS & GLASGOW, LTD., and A. R. GRIGGS (B.P. 349,383, 12.8.30).

**Pulverised solid material.**—See I. Oxygenated compounds. Mineral oil amino-sulphonates. C<sub>6</sub>H<sub>6</sub> from butane etc.—See III. Fibrous products.—See V. Rubber-coated fabrics.—See VI. Fuel for firing bricks.—See VIII. Cement mixture [for oil wells]. Asphalt emulsions and compositions. Tar for roads. Road-building material.—See IX. Sulphonation products. Waxes.—See XII. Coating compound. Colour lakes from petroleum. Synthetic resin varnishes.—See XIII.

### III.—ORGANIC INTERMEDIATES.

**Cooling on evaporation of solvents.** H. DABISCH (Farben-Ztg., 1931, 36, 1300).—By means of an apparatus based on the wet- and dry-bulb hygrometer, the cooling on evaporation of a range of solvents was determined under two sets of conditions (temp. 27° and 10°, and R.H. 63% and 93%, respectively). Five high-boiling solvents behaved anomalously, showing a max. instead of a min. temp. on the "wet thermometer." This is explained by their slight hygroscopicity. S. S. WOOLF.

**Determination of hexamethylenetetramine methylenecitrate and hexamethylenetetramine when mixed.** D. MAROTTA and F. DI STEFANO (Annali Chim. Appl., 1931, 21, 150–154).—The mixture is first rendered alkaline and distilled, the amount of CH<sub>2</sub>O distilling over corresponding with the hexamethylenetetramine methylenecitrate present. The residue is then made acid and again distilled, the CH<sub>2</sub>O thus obtained being derived from the free hexamethylenetetramine. T. H. POPE.

**Hydrolysis of acetyloxindoxyllic acid and the acetyloxindoxyls.** G. SPENCER (J.S.C.I., 1931, 50,

63—64 r).—Alkaline hydrolysis of *N*-acetylindoxyl acid gave about 20% of indigo and 50% of *N*-acetyl-anthranilic acid; HCl and FeCl<sub>3</sub> afforded mainly anthranilic acid and no indigo. Hydrolysis of *N*-acetylindoxyl with dil. Na<sub>2</sub>CO<sub>3</sub> in presence of air gave 55% of indigo, 8% of diacetylindigo, and 12% of isatin; dil. NaOH yielded mainly indigo (74%) and isatin, whilst acid FeCl<sub>3</sub> gave only isatin. *O*-Acetylindoxyl gave indigo (95%) and isatin (3—4%) with dil. Na<sub>2</sub>CO<sub>3</sub>; with H<sub>2</sub>O<sub>2</sub> and NaOH mainly isatin together with 18% of indigo and a little anthranilic acid were obtained. With acid FeCl<sub>3</sub> indirubin (18%) and isatin were produced. E. H. SHARPLES.

**Acetate from soda-pulp liquors.**—See V. **Ti compounds.**—See VII. **Diacetyl in butter.**—See XIX. **Trichloroethylene as disinfectant.**—See XXIII.

See also A., June, 692, **Catalyst for decomposition of MeOH.** 693, **Oxidation of MeOH with air.** 718—9, **Preparation of organo-Mg halides.** 722, **Fluorescence reaction for β-naphthol.** 740, **2-Chloropyridines.** 3-Aminopyridine. 748, **Thioindigotins of the naphthalene and anthraquinone series.**

#### PATENTS.

**Production of acetone from acetylene.** J. MARTIN and I. J. KRCHMA, Asss. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,779,676, 28.10.30. Appl., 18.5.29).—A mixture of irreducible metal oxides and a metal halide is used as catalyst, *e.g.*, Zn, Cr, and Fe<sup>+++</sup> oxides with ZnCl<sub>2</sub>, or Cr and Zn (or Ni) oxides with ZnCl<sub>2</sub>. C. HOLLINS.

**Hydration of acetylene [to acetaldehyde and crotonaldehyde].** G. F. HORSLEY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 346,288, 6.1.30).—The use of a Cd phosphate catalyst leads to 91% conversion in one passage of C<sub>2</sub>H<sub>2</sub> with steam at 100 reciprocal hr. into acetaldehyde and crotonaldehyde. C. HOLLINS.

**Manufacture of methylamine.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 344,971, 24.12.29).—Phthalimide, or other suitable imide, is methylated with CH<sub>2</sub>O and hydrolysed to give pure NH<sub>2</sub>Me. Examples are: phthalimide or *N*-hydroxymethylphthalimide with aq. CH<sub>2</sub>O in MeOH at 220°/70—90 atm.; *o*-sulphobenzoic imide with trioxymethylene, H<sub>2</sub>O, and MeOH at 220°. Diacetamide, succinimide, or cyanuric acid may also be used. C. HOLLINS.

**Manufacture of polymerisation products [of alkylene oxides].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 346,550, 20.3.30).—Ethylene oxide, led over lumps of KOH at 120—130°, evolves much heat and gives a wax, but over NaHSO<sub>4</sub> at 120° it gives dioxan and a little glycol ethylidene ether, b.p. 82°. Propylene oxide with alkaline catalysts gives an oil; with acid catalysts 2 : 5-dimethyldioxan, b.p. 117°, or 50°/2 mm. C. HOLLINS.

**Polymerisation of vinyl derivatives.** E. I. DU PONT DE NEMOURS & Co. (B.P. 319,587—8, 23.9.29. U.S., 22.9.28).—(A) Vinyl compounds (vinyl chloride or acetate, styrene) are polymerised, especially in a solvent (MeOH, ethylbenzene, BuOH, ethylene dichloride), by treatment with O<sub>3</sub>, with the assistance of

heat (90—120°) or light. The solvent may be pre-treated with O<sub>3</sub>. (B) A solution of a vinyl compound and a polymerisation catalyst are passed through a hot tube, and preferably are thereafter rapidly cooled; *e.g.*, vinyl chloride and benzoyl peroxide in PhCl, forced through a Sn-lined tube at 118°, give a new polymeride sol. in PhMe. C. HOLLINS.

**Production of the α-polymeride of vinyl chloride.** E. I. DU PONT DE NEMOURS & Co. (B.P. 319,591, 23.9.29. U.S., 22.9.28).—Vinyl chloride, preferably in a solvent (ethylene dichloride, MeOH, PhCl, AcOEt), is heated with a catalyst (benzoyl peroxide, ozonised solvent) above 80°, *e.g.*, at 120°. The α-polymeride is obtained substantially free from the β-compound. C. HOLLINS.

**Purification of synthetic alcohols.** J. R. PARK, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 344,796, 10.12.29).—Alcohols from hydrogenation of C oxides are treated with caustic alkali for several days, preferably with stirring with a current of air followed by keeping for some days before distillation. C. HOLLINS.

**Manufacture of oxygenated organic compounds.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 345,391, 16.1.30. Addn. to B.P. 312,388; B., 1929, 746).—Alcohols are the main product if in the process of the prior patent a petroleum of b.p. range 40—130° is oxidised in presence of 2% or more of a saturated aliphatic acid below C<sub>5</sub>; *e.g.*, hexane oxidised in presence of 10% of AcOH and 0.05% of Mn acetylacetonate with air at 145°/20 atm. gives mainly acetates of lower alcohols. C. HOLLINS.

**Manufacture of ketones [acetone from ethyl alcohol].** H. DREYFUS (B.P. 345,271, 18.12.29).—EtOH vapour passed over Ba(OH)<sub>2</sub> in a (Cu) tube at 250—350° gives Ba(OAc)<sub>2</sub>, or at 400—500° it gives C<sub>2</sub>Me<sub>2</sub> directly. C. HOLLINS.

**Preparation of ethyl esters.** A. R. CADE, Assr. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,781,050, 11.11.30. Appl., 7.7.23).—An org. Na salt (BzONa) is treated with Et<sub>2</sub>SO<sub>4</sub> in presence of the Et ester (BzOEt) as solvent and in substantial absence of H<sub>2</sub>O. C. HOLLINS.

**Production of lactic esters.** IMPERIAL CHEM. INDUSTRIES, LTD., W. R. H. HURLEY, and T. S. WHEELER (B.P. 346,486, 25.1.30).—The lactic ester of one alcohol is heated with another alcohol, *e.g.*, ethyl lactate with butyl alcohol, and the displaced alcohol is removed continuously or at intervals. C. HOLLINS.

**Preparation of butadiene.** E. K. BOLTON and F. B. DOWNING, Asss. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,777,600, 7.10.30. Appl., 8.1.27).—Diacetylene and H<sub>2</sub> are passed over a reduced Ni catalyst at 40—80°. C. HOLLINS.

**Manufacture of butadiene or its homologues.** I. G. FARBENIND. A.-G. (B.P. 345,270, 18.12.29. Ger., 18.12.28).—Olefinic alcohols above C<sub>3</sub> give diolefines when passed as vapour, preferably with steam, over a dehydrating catalyst (dried alums, acid phosphates) at 200—300°. Allylcarbinol yields 85—90% of butadiene; β-methyl-Δ<sup>2</sup>-penten-δ-ol, b.p. 127—130°, gives 80% of dimethylbutadiene, b.p. 75.5—77°. C. HOLLINS.

**Mineral oil amino-sulphonates [detergents].** W. T. REDDISH, Assr. to TWITCHELL PROCESS Co. OF CINCINNATI (U.S.P. 1,780,144, 28.10.30. Appl., 28.5.28).—An oil-soluble mineral oil sulphonic acid is combined with "triethanolamine" or other hydroxylated alkylamine. C. HOLLINS.

**Manufacture of basic products [dialkylamino-alkyl derivatives of higher fatty acids].** I. G. FARBENIND. A.-G. (B.P. 346,387, 2.1.30. Ger., 2.1.29).—A higher fatty acid, ester, chloride, or amide is heated at 200–230° with a di- or poly-amino-alcohol, one NH<sub>2</sub> group of which is tertiary, to give thickening agents for cosmetics, emulsifying agents, etc. Examples are:  $\beta$ -amino- $\beta'$ -diethylaminoisopropyl alcohol with oleic acid, ethyl oleate, stearamide;  $\beta$ -anilino- $\beta'$ -diethylaminoisopropyl alcohol with oleyl or stearyl chloride. C. HOLLINS.

**Manufacture of benzene and by-products from butane, propane, ethane, or mixtures thereof.** F. PORTER, Assr. to CONTINENTAL OIL Co. (U.S.P. 1,777,894, 7.10.30. Appl., 27.4.27).—The initial gases are passed downward through a brick-packed tower at 800–950°, the speed being adjusted as the tower cools. C. HOLLINS.

**Indophenol intermediate and blue sulphur dye derived therefrom.** W. A. MANSS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,777,757, 7.10.30. Appl., 27.10.25).—NHPh<sub>2</sub> in 93% H<sub>2</sub>SO<sub>4</sub> is mixed with *p*-nitrosophenol in 64% H<sub>2</sub>SO<sub>4</sub>, the mixture being kept below 0° (e.g., –20°). The product is sulphurised in the usual manner. C. HOLLINS.

**Manufacture of multivalent metal salts of half-esters of phthalic acid.** W. J. BANNISTER, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,779,654 and 1,779,688, 28.10.30. Appl., [A] 8.7.29, [B] 13.6.28).—Phthalic anhydride and BuOH are heated in PhMe at 100°, aq. NaOH is added below 50° until alkaline to phenolphthalein, then aq. ZnSO<sub>4</sub>. The resin is freed from solvent and H<sub>2</sub>O by distillation below 80°. Alternatively, the alkyl hydrogen phthalate may be treated directly with ZnO in presence of the org. solvent. C. HOLLINS.

**Production of imino-ethers.** K. F. SCHMIDT and P. ZUTAVERN (B.P. 345,397, 18.1.30. Addn. to B.P. 331,947; B., 1930, 939).—Acid amides or lactams are acylated (e.g., with benzenesulphonyl chloride) and treated with an alcohol, whereby the *O*-acyl group is replaced by alkyl. Cyclic imino-ethers are thus obtained from piperidone (b.p. 161–165° in vac.) and leucine-lactam. C. HOLLINS.

**Manufacture of unsaturated ethers.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 345,253, 12.12.29).—An acetal vapour is led at 200–350° over a Pd, Ag, or other noble-metal catalyst, preferably on a carrier (asbestos, clay, SiO<sub>2</sub> gel, Si). Other metals of groups II–IV, V–VIII, or compounds of these or of group I metals, may be added. Vinyl ethyl ether is obtained from diethyl acetal; vinyl *n*-butyl ether, b.p. 92–93°, from di-*n*-butyl acetal; isopropenyl ethyl ether, b.p. 61–62°/735 mm., from  $\beta\beta$ -diethoxypropane; *n*-propenyl ethyl ether, b.p. 69°, from  $\alpha\alpha$ -diethoxypropane; *n*-butenyl methyl ether, b.p. 73–74°/749 mm.,

from  $\alpha\alpha$ -dimethoxybutane;  $\alpha$ -styryl ethyl ether, b.p. 93–96°/12.5 mm., from  $\alpha\alpha$ -diethoxyethylbenzene; and vinyl  $\beta$ -ethoxyethyl ether [glycol vinyl ethyl ether], b.p. 128°, from ethylidene di- $\beta$ -ethoxyethyl ether, b.p. 106–116°/14 mm. C. HOLLINS.

**[Production of] dye intermediates [nitro- and amino-derivatives of 2-(3 : 4-dichlorobenzoyl)benzoic acid].** R. J. LOVELUCK, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 345,277, 13.9.29).—2-(3 : 4-Dichlorobenzoyl)benzoic acid is nitrated with 97% HNO<sub>3</sub> at 15° or mixed acids at 80°, and the product is reduced. C. HOLLINS.

**Production of condensation products [*o*-benzoylbenzoic acids, their nitro- and amino-compounds, and anthraquinones] from *o*-chlorophenol.** R. TONKIN, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 345,204, 4.12.29).—*o*-Chlorophenol is condensed with phthalic anhydride, the product is nitrated, reduced, and cyclised to give a mixture of chloroaminohydroxy-anthraquinones. C. HOLLINS.

**Manufacture of halogenated 3 : 4 : 8 : 9 [2 : 3 : 7 : 8]-dibenzpyrene-5 : 10 [1 : 6]-quinones.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 345,650, 23.9.29).—Uniform products are obtained by mono- or di-chlorinating or -brominating 2 : 3 : 7 : 8-dibenzpyrene-1 : 6-quinone (*a*) in an organic medium (nitrobenzene) in presence of a carrier, (*b*) in aq. suspension, (*c*) in absence of diluent, (*b*) and (*c*) preferably in presence of a carrier (*S*). Bromination gives only a monobromo-compound. The products are yellow to orange vat dyes. C. HOLLINS.

**Liquid supersaturated solution of thiocarbamide.** P. SIEDLER and A. MOELLER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,780,634, 4.11.30. Appl., 16.5.29. Ger., 28.1.28).—Premature crystallisation of supersaturated solutions of thiocarbamide, e.g., in *o*-toluidine, is prevented by addition of 0.3–20% of colophony, Venetian turpentine, resins from CH<sub>2</sub>O and NH<sub>3</sub> or amines, low-temperature carbonisation tar, etc. The use of mixed toluidines in place of pure *o*-toluidine is made possible. C. HOLLINS.

**Manufacture of aminoarylthiazole compounds.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 345,735, 22.1.30).—2-Amino-benz- and -naphtha-thiazoles are obtained by action of Cl, SO<sub>2</sub>Cl<sub>2</sub>, etc. on arylthiocarbamides, Ar·NX·CS·NH<sub>2</sub>, where X is H, alkyl, or aralkyl. 2-Amino- (m.p. 126–128°), 2-amino-4-methyl- (m.p. 138°), 2-amino-6-ethoxy- (m.p. 160–163°), 6-chloro-2-amino-4-methyl- (m.p. 200–205°), and 2-amino-6-methyl- (m.p. 142°)-benzthiazoles, 2-imino-3-methyl- (m.p. 123°) and 12-imino-3-phenyl-benzthiazolines, and 2-amino- $\alpha$ -naphthathiazole, m.p. 259–261°, are thus prepared. Amongst the starting materials are: *o*-tolyl- (m.p. 156–158°), 5-chloro-*o*-tolyl- (m.p. 175–177°), and *p*-tolyl- (m.p. 188°)-thiocarbamides. C. HOLLINS.

**Aldehyde-amine condensation products [vulcanisation accelerators].** I. WILLIAMS and W. B. BURNETT, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,780,326 and 1,780,334, 4.11.30. Appl., [A] 13.3.25, [B] 12.2.26).—An aldehyde is caused to react with an amine in mol. ratio above 2 : 1,  $\alpha$ -unsaturated

aldehydes being excluded, (B) in presence of an acid. Examples are: (A) heptaldehyde with *n*-butylamine; *n*-butaldehyde with aniline, with *o*-tolylidguanidine, or with *n*-butylamine; propaldehyde with aniline; the mol. proportions being 2, 3, 5, 7, 10, or 15 : 1; (B) *n*-butaldehyde with aniline or *o*-toluidine, and heptaldehyde with *n*-butylamine, in presence of *n*-butyric, stearic, or *n*-valeric acids, AcOH, or H<sub>2</sub>SO<sub>4</sub>, the mol. ratio of aldehyde to amine being 3 or 5 : 1.

C. HOLLINS.

**Manufacture of substituted thiuram polysulphides [vulcanisation accelerators].** G. S. WHITBY, ASS. TO ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,780,545, 4.11.30. Appl., 28.4.28).—A disubstituted dithiocarbamate is treated with a S chloride, which may contain dissolved S; e.g., K dimethyldithiocarbamate and S<sub>2</sub>Cl<sub>2</sub> in Et<sub>2</sub>O give tetramethylthiuram disulphide, m.p. 108°.

C. HOLLINS.

**Manufacture of albumose-like degradation products.** A. CARPMAEL, FROM I. G. FARBENIND. A.-G. (B.P. 345,630, 24.12.29).—Wool, horn, hair, or other keratin-containing substance is converted into H<sub>2</sub>O-sol. degradation products containing thiol groups by hydrolysis (e.g., with dil. HCl) and reduction (e.g., with Sn and HCl), which may be simultaneous.

C. HOLLINS.

**Manufacture of thymol and its isomerides and homologues.** SCHERING-KAHLBAUM A.-G. (B.P. 344,970, 24.12.29. Ger., 31.12.28. Addn. to B.P. 273,685 and 279,855; B., 1930, 136; 1929, 237).—The condensation product obtained according to B.P. 273,684 (B., 1929, 236) from *m*- or *p*-cresol and an aliphatic ketone (COMe<sub>2</sub>) is heated in H<sub>2</sub> at 300°, and the vapours and H<sub>2</sub> are passed over reduced Ni on pumice at 200°.

C. HOLLINS.

**Purification of chloronaphthalenes.** HALOWAX CORP., ASSEES. OF S. BROWN and E. R. HANSON (B.P. 343,878, 18.10.29. U.S., 19.10.28).—Crude chlorinated C<sub>10</sub>H<sub>8</sub>, prepared by chlorinating molten C<sub>10</sub>H<sub>8</sub> until the desired sp. gr. is reached (*d* 1.41—1.43 at 150° for higher chlorination products, 1.20 for mono- and di-chloronaphthalenes), is fractionally distilled at reduced pressure (e.g., 100—150 mm.), preferably after neutralising with CaO, NaOH, etc. Mono- and di-chloro-derivatives are collected at 140—180°, higher derivatives at 200—225° and 225—250°, the last fraction being the most valuable. Suitable plant is figured.

C. HOLLINS.

**Manufacture of [derivatives of] hydrocarbons containing halogenomethyl [substituents].** I. G. FARBENIND. A.-G. (B.P. 345,146, 7.5.30. Ger., 8.5.29).—A polynuclear hydrocarbon is treated with aq. CH<sub>2</sub>O and H halide (HCl), without condensing agent or org. solvent, so as to introduce 2 or more halogenomethyl groups. The chloromethyl derivatives of naphthalene (m.p. 130—140°), anthracene, and tetrahydronaphthalene (m.p. 117.5°) are described.

C. HOLLINS.

**Production of acetyl-Cleve [acetyl 1:6(7)-naphthylaminesulphonic] acid.** W. M. RALPH, ASS. TO NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,778,914, 21.10.30. Appl., 9.7.25. See U.S.P. 1,566,425; B., 1926, 185).—Cleve acid is heated with AcOH and AcONa in substantial absence of H<sub>2</sub>O.

C. HOLLINS.

**Purification of anthracene.** C. J. THATCHER (U.S.P. 1,782,488, 25.11.30. Appl., 16.6.23).—The crude anthracene (30% pure) is ground in a ball mill with 1—2 times its wt. of a solvent, e.g., C<sub>6</sub>H<sub>6</sub>, CCl<sub>4</sub>, in which anthracene (and carbazole) is relatively insol., but which dissolves phenanthrene, oils, etc.; carbazole can be removed from the finely-divided product (45% anthracene) by solvents, e.g., COMe<sub>2</sub> or pyridine, or these may be used as the grinding solvent.

E. LEWKOWITSCH.

**Manufacture of [hydroxylated] anthraquinone derivatives.** BRIT. CELANESE, LTD., E. W. KIRK, H. C. OLPIN, and G. H. ELLIS (B.P. 346,355, 8.1.30).—In the nitrosylsulphuric acid method for introducing OH groups into anthraquinones, the amount of Hg catalyst should be 0.05—0.40%; H<sub>3</sub>BO<sub>3</sub> may be added. Anthraquinone is oxidised to quinizarin, 1:8-dihydroxyanthraquinone to 1:5:8-trihydroxyanthraquinone.

C. HOLLINS.

**Manufacture of halogenated products of anthraquinone derivatives [halogenated anthraquinone-acridones from anthraquinonylisatins].** J. Y. JOHNSON, FROM I. G. FARBENIND. A.-G. (B.P. 346,359, 30.10.29).—*N*-Anthraquinonylisatins are halogenated and converted into halogenated anthraquinoneacridones. Examples are: *N*- $\alpha$ -anthraquinonylisatin, dichlorinated, converted into the acridone (red vat dye) by vating; or chlorinated and brominated in presence of NaCl-AlCl<sub>3</sub> to give directly the chlorobromoacridone (blue-red); or brominated in presence of I and Fe in nitrobenzene at 160—170° for the bromoacridone (blue-red); 4(or 6)-chloro-1- $\alpha$ -anthraquinonylisatin, brominated for a chlorobromoacridone (blue-red), or chlorinated and vatted for the chloroacridone; 1- $\alpha$ -anthraquinonyl-5-methylisatin chlorinated for the chloroacridone (red); *N*- $\alpha$ -anthraquinonyl- $\beta$ -naphthisatin brominated in presence of S and I in nitrobenzene at 140—160° for the bromoacridone (yellow-red).

C. HOLLINS.

**Splitting of acetylene.**—See II. Alginate products.—See VII. EtOH.—See XVIII. Cineoles.—See XX.

#### IV.—DYESTUFFS.

**Hydrolysis of acetyloxycylic acid etc.**—See III.

See also A., June, Absorption of dye granules by hydrosols. 721, Azo chromophores. 722, Light-sensitive diazo compounds. 725, Nitro-dye from aminosulphosalicylic acid. 732, Insol. disazo dyes from 1:5-diaminoanthraquinone. 733, Plant colouring matters. 738, Dye of the corn poppy. Dye of acacia wood. Anthochlor of *Linarea vulgaris*. 741,  $\psi$ -Cyanine dyes. 748, Thioindigotins of the naphthalene and anthraquinone series. 777, Colouring matter of "Awobana."

PATENTS.

**Dye preparation.** M. P. PARRISH (U.S.P. 1,780,981, 11.11.30. Appl., 19.12.28).—An aniline dye is pre-digested with pancreatin, ox gall, NaCl, citric acid, AcOH, and COMe<sub>2</sub>, boiled with soap and tinctures of camomile flowers, hyssop, ground cedar wood, and myrrh, then fermented for 2 weeks with pancreatin, gelatin, COMe<sub>2</sub>, NaCl, ox gall, and AcOH, and finally

baked with dextrin until crisp. The product dyes cotton and wool directly. C. HOLLINS.

**[Manufacture of] indigoid dyes.** W. BAUER, W. NEELMEIER, and T. NOCKEN, Assrs. to GEN. ANILINE WORKS, INC. (U.S.P. 1,782,706, 25.11.30. Appl., 9.5.27. Ger., 11.6.26).— $\beta\beta$ -Naphthisatin  $\alpha$ -chlorides (etc.), having halogen in the 4-position, are condensed with a 4-alkoxy- $\alpha$ -naphthol, having the 2-position free, to give green vat dyes; e.g., brominated 4-chloro- $\beta\beta$ -naphthisatin, m.p. 313°, with 4-methoxy- $\alpha$ -naphthol. C. HOLLINS.

**Manufacture of indigoid vat dyes.** I. G. FARBENIND. A.-G. (B.P. 345,130, 16.4.30. Ger., 22.4.29).—6-Methylthioindoxyl is condensed with 5-chloro- or 5-bromo-isatin  $\alpha$ -chloride (etc.) to give fast grey vat dyes. C. HOLLINS.

**Manufacture of indigoid dyes.** SOC. CHEM. IND. IN BASLE (B.P. 345,743, 28.1.30. Switz., 2.2.29).—4 : 5-Dichloro-7-methylthioindoxyl is condensed, e.g., with 5 : 7-dichloroisatin  $\alpha$ -chloride (violet vat dye), acenaphthoquinone (red), isatin  $\alpha$ -anil (red-violet; brominated, violet), 9-chloro- $\beta\beta$ -naphthathioisatin  $\alpha$ -*p*-dimethylaminoanil (violet). C. HOLLINS.

**Manufacture of [thio]indigoid dyes.** I. G. FARBENIND. A.-G. (B.P. 345,349, 23.12.29. Ger., 22.12.28).—A pyrazolothioindoxyl is oxidised or is condensed with an isatin or thioisatin  $\alpha$ -chloride (etc.) to give vat dyes. 6-Aminoindazole, m.p. 210°, is converted by way of its 5- or 7-thiocyano-derivative, m.p. above 300°, into 6-aminoindazolyl-5- or -7-thioglycollic anhydride, m.p. above 300°, the  $\text{NH}_2$  group is exchanged for the CN group, and the product is heated with  $\text{Na}_2\text{S}$  solution to give, after treatment with  $\text{H}_2\text{SO}_4$ , 5 : 6- or 7 : 6-pyrazolothioindoxyl, m.p. 235°, which is oxidised to a grey vat dye. The *N*-Me compound from 6-amino-2-methylindazole (thiocyano-derivative, m.p. 280—285°; thioglycollic anhydride, m.p. 278—280°; thioindoxyl, m.p. 200°) dyes olive shades. 6-Amino-5-methylindazole, m.p. 240—242° (7-thiocyano-derivative and thioglycollic anhydride, m.p. above 300°; thioindoxyl, m.p. 285°) gives a grey vat dye. The pyrazolothioindoxyl first mentioned is condensed with  $\beta\beta$ -naphthathioisatin  $\alpha$ -anil (bluish-grey), 5-chloroisatin  $\alpha$ -chloride (bluish-grey),  $\beta$ -naphthisatin  $\alpha$ -chloride (olive-green), and  $\alpha$ -naphthathioisatin (grey). C. HOLLINS.

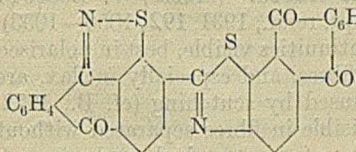
**Manufacture of vat dyes [from naphthalene-tetracarboxylic dianhydride and *o*-diamines].** I. G. FARBENIND. A.-G. (B.P. 344,779, 29.10.29. Ger., 29.10.28).—Vat dyes from naphthalenetetracarboxylic dianhydride and halogenated *o*-arylenediamines are condensed with suitable  $\text{NH}_2$  compounds. The product from 4-chloro- or 4-bromo-*o*-phenylenediamine is condensed with  $\alpha$ -aminoanthraquinone (corinth), 1-amino-2-methylanthraquinone (dull brown), 1-amino-5-benzamidoanthraquinone (dull brown), 1-amino-4-benzamidoanthraquinone (greenish-grey); that from 3 : 5-dibromo-*o*-phenylenediamine with  $\alpha$ -aminoanthraquinone (grey-brown). Similar products are obtained by condensing halogeno-compounds with diamino-bisimidazoles made, e.g., from nitro-*o*-phenylenediamine. C. HOLLINS.

**Manufacture of vat dyes [of the thiazolanthrone series].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 346,373, 30.9.29).—Thiazolanthrone-3-carboxylic

chloride etc. is condensed with an *o*-amino-phenol or -thiophenol or an *o*-diamine, the product, if desired, being halogenated, nitrated, etc. With 2-amino-3-hydroxyanthraquinone and with *o*-phenylenediamine yellow vat dyes are obtained. C. HOLLINS.

**Manufacture of vat dyes of the *N*-dihydro-1 : 2 : 2' : 1'-anthraquinoneazine [indanthrone] series.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 346,328 and 344,777, [A, B] 2.10.29, [A] 8.1.30).—(A) The condensation product from impure indanthrone and  $\text{CH}_2\text{O}$  is halogenated, or is first treated with P pentahalide (etc.) and then purified and/or halogenated by means of halogen. Fast greenish-blues are obtained. (B) The vat dyes obtained by oxidising the condensation products from indanthrones and  $\text{CH}_2\text{O}$  (B.P. 322,281; B., 1930, 316) are halogenated to give redder shades of increased fastness to bleach. C. HOLLINS.

**Vat colour of the 1 : 9-thiazolanthrone series.** R. N. LULEK, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,777,756, 7.10.30. Appl., 18.3.27).—A yellow vat dye (annexed formula) is obtained by condensing 2-amino-1-thiolanthraquinone in  $\text{PhNO}_2$  at 180° with thiazolanthrone-*o*-carboxylic chloride or the corresponding aldehyde. C. HOLLINS.



**Manufacture of [vat] dyes [chlorinated dibenzanthrone].** BRIT. ALZARINE Co., LTD., C. W. SOUTAR, and J. ANDERSON (B.P. 345,623, 24.12.29).—Indanthrone is heated with large excess (1.6—3.4 pts.) of  $\text{SO}_2\text{Cl}_2$  below 80° (65—75°) for about 24 hr. or for about 5—6 hr. in presence of a carrier, preferably in a large bulk of solvent ( $\text{PhNO}_2$ ), in which the dibenzanthrone may be preheated and cooled before chlorination. Blue, not violet, vat dyes result. C. HOLLINS.

**Manufacture of vat dyes [of the benzanthrone-pyrazolanthrone series].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 345,651, 14.10.29. Addn. to B.P. 341,884; B., 1931, 476).—All the Br in the brominated benzanthronepyrazolanthrone of the prior patent is exchanged for one or more of the groups  $\text{NHR}$ ,  $\text{OR}$ , and  $\text{SR}$ ; the products may be alkylated and/or further condensed, e.g., by alkaline fusion. The dibromo-compounds are condensed with 1-aminoanthraquinone (grey vat dye), 1-amino-4-benzamidoanthraquinone (green-grey), 2-aminoanthraquinone (blue-grey), benzamide (green-blue), and 2 : 4-dichloro-7-amino-8 : 9-phthaloylacridone (green-grey); the monobromo-compound is condensed with 1-aminoanthraquinone (blue-grey to blue-black), and  $\text{PhOK}$  (navy-blue). C. HOLLINS.

**Manufacture of vat dyes [of the benzanthrone-pyrazolanthrone series].** I. G. FARBENIND. A.-G., and A. G. BLOXAM (B.P. 345,728, 20.1.30. Addn. to B.P. 298,284; B., 1928, 923).—A substituted (especially anthraquinonyl) amino-derivative of an *N*-3-benzanthronylpyrazolanthrone is heated with (alcoholic) alkali to give a vat dye, usually bluish-grey. Examples of starting materials are the condensation products from *N*-(9-bromo-3-benzanthronyl)pyrazolanthrone with

1-amino-, 2-amino-, 1:4-diamino-, 1-amino-4-benzamido-, 1-amino-5-benzamido-, and 1-amino-2-aldehydo-antraquinones (grey to black), 3-aminobenzanthrone, aminobenzanthronylpyrazolanthrone, carbazole, or 1:2-diaminoanthraquinone; from 5-, 7-, or 10-chloro-*N*-3-benzanthronylpyrazolanthrone with 1-aminoanthraquinone, etc. C. HOLLINS.

**Disazo dye containing a diphenylcarbamide nucleus.** L. W. GELLER, Assr. to NAT. ANILINE & CHEM. Co., Inc. (U.S.P. 1,782,682, 25.11.30. Appl., 28.4.24).—The dye *p*-nitroaniline  $\rightarrow$  salicylic acid is reduced and phosgenated together with the dye *o*-toluidine-4-sulphonic acid  $\rightarrow$  cresidine. The product is a direct yellow dye for cotton. C. HOLLINS.

**Dye-works.**—See I. Blue sulphur dye. Halogenated dibenzpyrenequinones. Halogenated anthraquinoneacridones.—See III. Lakes etc.—See XIII. Colouring butter etc.—See XIX.

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

**Structure of textile fibres.** (MLE.) V. BOSSUYT and G. CHAUDRON (Compt. rend., 1931, 192, 1050—1053).—The transverse discontinuities visible, best in polarised light, in many textile fibres, and especially in flax, are not due to fracture caused by scutching (cf. B., 1922, 853 A), being equally visible in fibres separated without scutching. Comparison of a stretched and an unstretched fibre after treatment with 25% NaOH solution shows that they are merely folds of the external fibrils, and disappear on stretching. C. A. SILBERRAD.

**Mounting of textile fibre sections.** J. M. PRESTON (J. Soc. Dyers and Col., 1930, 46, 295—297).—When searching for foreign matter in a section it is preferred to use a mountant having the same *n* value as the section. Aq. mountants are not suitable for textile fibres and particularly for regenerated cellulose silks because of the consequent swelling. The most suitable mountants for fibre sections (other than cellulose acetate) are collodion of *n* 1.44 (for contrast) and Canada balsam of *n* 1.53 (for rendering invisible); for cellulose acetate fibre sections no mountant is available which does not cause swelling, but the most suitable are glycerin jelly of *n* 1.37 (for contrast) and Euparal of *n* 1.50 (for rendering invisible). A. J. HALL.

**Measurement of the colour of textile fabrics.**

**IV. The Ostwald colour system.** P. W. CUNLIFFE and P. N. LAMBERT (J. Soc. Dyers and Col., 1930, 46, 297—307).—The instruments used and the principles on which the Ostwald colour system is based are discussed critically in relation to actual colour measurements of the standards by the Guild colorimeter. It is concluded that the grey standards are satisfactory as regards their neutral colour and their brightness, but with "full" or "pure" colour standards the steps are too close together in the blue and blue-green regions and too widely separated in three other regions. Colours which are diametrically opposite in the colour standard circle deviate slightly from the requirement that they should be complementary. A. J. HALL.

**Determination of the quantity of silk in silk fabric.** D. ONGARO (Giorn. Chim. Ind. Appl., 1931, 13, 159—162).—Determination of silk by the Kjeldahl

method requires the preliminary removal of nitrogenous dyes and, like other suggested methods, occupies much time. The use of Van Slyke's method leads to variable results, owing to the influence of extraneous substances on the hydrolysis of the silk proteins and to the formation of artificial melanins or humin substances. The amino-N in fibroin may be determined by heating with alkaline  $\text{KMnO}_4$  for  $\frac{1}{2}$  hr. and titrating the  $\text{NH}_3$  distilled. The % N in dry de-gummed fibroin, thus determined, is 15.89, the ratio between the weights of fibroin and N varying, for cocoons of different races, from 6.26 to 6.32. With various silk tissues this method gives results agreeing closely with those of the determination of total N by Kjeldahl's method.

T. H. POPE.

**Decomposition of maize stalks by nitric acid.** E. HORVÁTH and G. ÉBER (Cellulosechem., 1931, 12, 85—95; cf. B., 1929, 937, 974).—The cell substance remaining after treatment of maize stalks with 2, 2.5, and 3%  $\text{HNO}_3$  at 70°, 90°, and 98.3° (bath temp.) during various periods has been determined. Most of the decomp. appears to take place while the temp. of the reaction mixture is rising to that of the bath, which temp. is never exceeded. It is concluded that the more conc. is the acid and the higher the bath temp., the more speedily the reaction is ended, little reaction taking place after the temp. of the reaction mixture is approx. that of the bath. For a given yield, the higher is the bath temp. and the more conc. the acid, the greater is the quantity of acid used up in the reaction. J. D. A. JOHNSON.

**Action of caustic soda on cellulose.** S. M. NEALE (J.S.C.I., 1931, 50, 177—182 T).—Recent theoretical developments are summarised and special attention is devoted to the characteristic properties of "mercerised cellulose." This material merely exhibits in greater degree the chemical properties of the original cellulose, and must be clearly distinguished from cellulose modified by oxidation or acid attack, which exhibits new chemical properties. Various methods may be used for estimating the "activation" of cellulose which persists after swelling; the results they yield are compared.

**Affinity of nitrocellulose for gelatinising agents.** J. DESMAROUX (J. Chim. phys., 1931, 28, 163—173).—A discussion of published work on the dissolution, swelling, and gelation of nitrocellulose in a number of solvents and solvent mixtures. E. S. HEDGES.

**A reaction of sulphite-cellulose.** C. KULLGREN (Svensk Kem. Tidskr., 1931, 43, 99—105).—Sulphite-cellulose removes Cu from a dil. solution in distilled  $\text{H}_2\text{O}$  filtered through it, the action being due to a base exchange similar to that operating in permutit filters; in one experiment 95% of the Cu present in solution was taken up by the cellulose. The rate of absorption of the Cu is practically instantaneous, but the total amount which can be taken up is small, sulphite-cellulose with 0.5% S taking up only 0.5% Cu. The presence of other cations, especially Ca and Na, in concentrations of more than 100 times that of the Cu, entirely prevents the absorption of the latter. The Cu taken up by sulphite-cellulose can be detected by treatment with KI and starch in the usual way. H. F. HARWOOD.

**Effect of swelling agents on the creasing properties of viscose silk.** A. J. HALL (J. Soc. Dyers and Col., 1930, 46, 293—294).—After swelling treatments with cold 5% NaOH,  $H_3PO_4$  ( $d$  1.55), and  $H_2SO_4$  ( $d$  1.44), viscose yarn has an increased affinity for direct dyes, but a slightly decreased elasticity and resistance to creasing as measured by the methods described previously (B., 1930, 1023), except in the case of  $H_3PO_4$ , when the swelled silk is somewhat less creasable than the untreated silk.  
A. J. HALL.

**Determination of the degree of decomposition of pulps.** K. BERNDT (Zellstoff u. Papier, 1931, 11, 257—258).—A summary (with references) of the methods in use for indicating or determining the extent of purification of pulps is given. The methods comprise staining, e.g., with malachite-green or a mixture of this with Congo-red, measurement of fluorescence, determination of lignin and of halogen consumption, and the use of oxidation methods, e.g., with saturated aq. quinone or with  $KMnO_4$ . There is an approx. linear relation between lignin content and Cl consumption.  
B. P. RIDGE.

**Recovery of acetate from the black liquors of soda-pulp manufacture.** H. SCHMIDT (Zellstoff u. Papier, 1931, 11, 259—260).—The liquor is used in continuous process and submitted to pressure and heat (about 350° and 150—200 atm.), whereby it is largely freed from organic impurities. Its alkalinity is also raised by addition of NaOH or by treatment with CaO and the process is continued until an acetate content of 10—15% is reached, when part of the liquor is drawn off for recovery of the acetate as such, or as MeOH or  $COMe_2$ . High yield of  $COMe_2$  is obtained by evaporating the solution and dry-distilling the residue.  $AcONa$  is obtained by evaporation and subsequent crystallisation; dissolved NaOH does not interfere with this process. A method is also described whereby soda-free acetate may be obtained. The yield of  $AcONa$  depends on the nature of the wood or other raw material; with pine wood about 13% on the wt. of the dry wood is recovered.  
B. P. RIDGE.

**Paper-making properties of New Zealand flax (*Phormium tenax*).** M. B. SHAW, G. W. BICKING, and M. J. O'LEARY (Bur. Stand. J. Res., 1931, 6, 411—420).—*P. tenax* should be a satisfactory material for the manufacture of wrapping and writing papers, the scutched material being the most promising. Prior to chemical treatment thorough mechanical cleaning by loosening the fibre, hand-sorting, and dusting is essential. The NaOH and two-stage sulphite-alkali processes of digestion are satisfactory as regards both the quality of the product and the yield. In semi-commercial-scale tests the yield of air-dried unbleached pulp from the fibre was 51—78%, the bleaching powder requirement was 12—20%, and the ratio of bursting strength to wt. for paper averaging 57 lb. per 500 sheets (25 in.  $\times$  40 in.) was 66.4—77.3% (91.3—104.5%) for bleached (unbleached) pulp, the sheet thickness being 0.004 in.  
H. F. GILLBE.

See also A., June, 683,  $pH$  stability region of insol. proteins. 716, Hydrolysis of cellulose. 717, Cellulose nitric esters.

## PATENTS.

**Felting [of fur and the like].** C. D. PARKS (U.S.P. 1,782,764—5, 25.11.30. Appl., 9.4.25. Renewed 14.2.30).—The material is treated with NaOH ( $d$  1.007—1.074) alone (B) or in admixture with  $H_2O_2$  (A). After drying, the fur fibres are removed from the skins, formed into hat shapes, and washed with dil. acid, e.g., 0.033% formic acid.  
D. J. NORMAN.

**Manufacture of felted fibrous products.** H. L. LEVIN, Assr. to FLINTKOTE Co. (U.S.P. 1,782,855, 25.11.30. Appl., 11.2.28).—An aq. dispersion of a bituminous waterproofing substance is added to an aq. fibrous stock and the whole treated with a fixing agent (Na resinate or silicate and alum), the  $pH$  of the mixture being adjusted to approx. that of the fixing agent at the isoelectric point; the mixture is then forced against a foraminous mould, and the formed article is removed and dried.  
F. R. ENNOS.

**Cellulosic product.** E. A. VAZQUEZ, Assr. to VAZCANE PROCESS, INC. (U.S.P. 1,782,751, 25.11.30. Appl., 3.4.28. Cuba, 2.6.27. Cf. B., 1928, 940).—Sugar cane is ground or abraded to separate the fibres and disrupt the sugar cells, and then repeatedly extracted with  $H_2O$  in countercurrent until all the sugar is removed. The fibrous pulp remaining is suitable for paper or fibre-board manufacture.  
F. R. ENNOS.

**Manufacture of plastic articles from artificial wood pulp.** K. KÜRSCHNER (B.P. 347,807, 4.3.30. Ger., 4.3.29).—A mixture of pulverised wood or like material with a dissolved compound of cellulose (cellulose xanthate, cuprammonium cellulose, nitrocellulose) is moulded under pressure and treated (e.g., with dil. alkali or alkali sulphide) to regenerate the cellulose.  
F. R. ENNOS.

**Manufacture of soluble cellulose esters.** I. G. FARBENIND. A.-G. (B.P. 347,451, 4.4.30. Ger., 4.4.29. Addn. to B.P. 283,181; B., 1928, 637).—The process of the prior patent is extended to include esters prepared from halides of saturated or unsaturated heterocyclic carboxylic acids or heterocyclic substituted fatty acids, e.g., the carboxylic acids of furan, coumarone, indole, pyridine, pyrazine, etc. and their derivatives. Esters containing N in the ring form salts with acids and additive products with, e.g., alkyl halides. By suitable selection of the heterocyclic radical, pharmaceutical or bactericidal properties may be imparted to the resulting ester. The preparation of cellulose isonicotinic acid is described.  
D. J. NORMAN.

**Manufacture and treatment of cellulose esters.** U.S. INDUSTRIAL ALCOHOL Co. (B.P. 346,430, 8.1.30. U.S., 30.4.29. Addn. to B.P. 306,531; B., 1930, 609).—Organic esters of cellulose are dissolved or dispersed in liquid  $SO_2$ , or esters of acids other than  $AcOH$  are produced in the liquid  $SO_2$  as medium; the liquid is then discharged into a precipitating medium so that the  $SO_2$  is suddenly gasified, causing disruption of the precipitated esters, to a light, fluffy, product.  
F. R. ENNOS.

**Manufacture of alkyl and aralkyl derivatives of cellulose.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 346,426, 4.1.30).—Cellulose, cellulose

hydrate, or hydrocellulose is treated in one stage with caustic alkali solution and an alkylating or aralkylating agent in the presence of solid NaOH, an indifferent salt (NaCl), and an indifferent solvent or diluent ( $C_6H_6$ ), the wt. of  $H_2O$  present in the reaction mixture not exceeding that of the cellulose. F. R. ENNOS.

**Manufacture of cellulose derivatives.** SOC. CHEM. IND. IN BASLE (B.P. 347,926, 29.5.30. Switz., 27.7.29. Addn. to B.P. 342,167; B., 1931, 343).—The treated material described in the prior patent is further treated with a compound containing an NH, OH, or SH group, or with a salt of such a compound. The product, while still immunised to direct cotton dyes, has its affinity for basic dyes more or less replaced by an affinity for acid dyes. F. R. ENNOS.

**Esterification of cellulosic materials.** BRIT. CELANESE, LTD. (B.P. 346,292, 6.1.30. U.S., 4.1.29).—Cellulose is agitated in a pretreating chamber, *e.g.*, with AcOH, and is then discharged directly and preferably gradually into the esterification vessel. F. R. ENNOS.

**Manufacture of cellulose nitro-acetates.** I. G. FARBENIND. A.-G. (B.P. 344,761, 11.7.30. Ger., 11.7.29. Addn. to B.P. 319,285; B., 1931, 343).—By adding urea in suitable amount to the acetylating mixture, nitro-acetates containing < 0.1% N may be produced. H. ROYAL-DAWSON.

**Manufacture of cellulose ether nitrates.** DEUTS. CELLULOID-FABR. (B.P. 347,423, 25.3.30. Ger., 25.3.29).—An alkyl ether of cellulose containing at least 2 ether groups per mol. of  $C_6H_{10}O_5$  and free OH groups, *e.g.*, diethylcellulose (100 pts.), is treated with diacetyl-orthonitric acid (400 pts.) and a dehydrating agent, *e.g.*, a mixture of  $Ac_2O$  (25 pts.) and glacial AcOH (25 pts.) at  $10^\circ$ . After 10 min. the product is precipitated with  $H_2O$  and washed. The resulting ether ester is stable, sol. in many org. solvents and solvent mixtures, and insol. in  $H_2O$ . D. J. NORMAN.

**Production of nitrocellulose.** H. C. HEIDE. FROM E. TSCHUDIN (B.P. 347,169, 14.12.29).—The nitrating acid used in the process of B.P. 323,019 (B., 1930, 184) may contain considerably less  $HNO_3$ , *e.g.*,  $HNO_3$  37%,  $H_2SO_4$  57%,  $H_2O$  6%, if the partly gelatinised cellulose is allowed to retain 10–20%  $H_2O$ . In this case the final washing is preferably effected at  $60^\circ$ . Alternatively, the material may be washed with  $HNO_3$  before nitration. D. J. NORMAN.

**Nitrocellulose compositions.** G. H. PADGHAM (B.P. 347,597, 23.1.30).—A stable film of plasticised nitrocellulose is produced by mixing a solution of collodion and amyl stearate with one consisting of "Neutroleum" (cyclohexyl acetate and 5% of a 1:1 mixture of  $AcOBu$  and butylcellulose) and pale mineral oil, and allowing to dry. F. R. ENNOS.

**Manufacture of mixed ethers of cellulose.** I. G. FARBENIND. A.-G. (B.P. 345,028, 30.1.30. Ger., 25.2.29).—Ethylene oxides accelerate the alkylation or benzylation of alkali-cellulose, and a mixed alkyl (or benzyl) hydroxyethyl ether of cellulose is produced. Examples are: 6 mols. of  $CH_2PhCl$  with 1 mol. of ethylene oxide, or 1.5 mols. of butylene oxide, heated with alkali-

cellulose at  $105$ – $110^\circ$ ; 20 mols. of  $PrCl$  and 3 mols. of ethylene oxide similarly at  $120^\circ$ . The products are sol. in  $C_6H_6$ . C. HOLLINS.

**Treatment of cellulose derivatives.** BRIT. CELANESE, LTD. (B.P. 347,257, 22.1.30. U.S., 22.1.29. Addn. to B.P. 341,890; B., 1931, 395).—Cellulose esters purified by the process of the prior patent may be used in the manufacture of films etc. D. J. NORMAN.

**Manufacture of films containing cellulose acetate.** H. J. HANDS (B.P. 346,339, 30.12.29).—A mixed aryl phosphate containing a single naphthyl group, *e.g.*, diphenyl  $\beta$ -naphthyl phosphate, phenyl *m*-tolyl  $\beta$ -naphthyl phosphate, is used as plasticiser. F. R. ENNOS.

**Apparatus for manufacture of artificial silk.** H. F. NIOGRET, ASSR. to DU PONT RAYON Co. (U.S.P. 1,782,581, 25.11.30. Appl., 14.4.28. Fr., 14.4.27).—Solutions of cellulose derivatives in a volatile solvent are dry-spun in a closed heated cell provided with one or more cooled condensers, each of which is directly connected to the cell by a single large aperture. F. R. ENNOS.

**Manufacture of artificial fibres.** HEBERLEIN & Co. A.-G. (B.P. 347,810, 7.3.30. Ger., 7.3.29).—Cuprammonium solutions of cellulose are spun, with or without preliminary coagulation in a known manner, in a precipitating bath of caustic alkali of mercerising concentration below  $0^\circ$ . F. R. ENNOS.

**Manufacture of artificial threads.** NOVASETA A.-G. ARBON (B.P. 347,878, 28.4.30. Switz., 11.7.29).—In the wet-stretch-spinning process the thread is passed substantially vertically first through a preliminary coagulating liquid, and then through one or more coagulating liquids of different composition from the first, each vessel containing coagulating liquid being so arranged with respect to the outlet of the preceding one that liquid flowing through this outlet does not enter the further vessels. F. R. ENNOS.

**Production of artificial filaments, yarns, or threads.** BRIT. CELANESE, LTD., H. DREYFUS, W. A. DICKIE, and W. I. TAYLOR (B.P. 346,354, 8.1.30. Addn. to B.P. 321,762; B., 1930, 98).—A systematic variation in denier is imparted to the threads by varying the degree of stretching, various mechanical means for which are described. F. R. ENNOS.

**Manufacture of diazotisable fibres.** C. GRÄNACHER (B.P. 346,385, 30.12.29. Switz., 28.12.28).—Fibres of cotton, regenerated cellulose, or partly acetylated cellulose, after being brought into a reactive condition if necessary, *e.g.*, by swelling with alkali and washing with water, are treated at about  $100^\circ$  with a nitrated benzyl halide, which may be undiluted, in an indifferent solvent or in aq. suspension, in the presence of an acid-binding agent other than caustic alkali, *e.g.*,  $CaCO_3$ ,  $Na_2CO_3$ ; the nitrobenzylated fibres are afterwards reduced to the  $NH_2$  derivative. F. R. ENNOS.

**Manufacture of paper.** MEAD RES. ENG. Co., Assees. of J. TRAQUAIR (B.P. 347,096, 7.12.29. U.S., 29.12.28).—Straw, bamboo, or similar cellulosic material is steeped for about  $\frac{1}{2}$  hr. in a large excess of a 1–6% solution of NaOH at about  $90^\circ$ , squeezed until it contains



3—4 pts. of liquor per 1 pt. of raw material, and digested, without further addition of treating liquor, for  $\frac{1}{2}$  hr. at 170—190°. The digester is heated by direct steam and the time taken to reach the cooking temp. should be about  $\frac{1}{2}$  hr. The resulting pulp is washed and chlorinated by the processes of B.P. 339,333—4 and 339,599 (B., 1931, 344). The process is continuous. Suitable apparatus is described. D. J. NORMAN.

**Wood-pulp material.** J. H. WALLACE, ASSR. to PINE WASTE PRODUCTS, INC. (U.S.P. 1,781,712, 18.11.30. Appl., 5.2.25).—A more uniform pulp is obtained if the chips are first treated with steam in the presence of hot waste furnace gases, to bring them to a substantially uniform moisture content. Suitable apparatus is described. D. J. NORMAN.

**Production of paper pulp.** R. B. WOLF (U.S.P. 1,780,638, 4.11.30. Appl., 23.12.29).—For the recovery of acid in the sulphite-pulp process, the gases and liquids from the digesters are cooled by passing through a relief line and an accumulator in which a constant pressure is maintained, whence they are discharged into an acid absorbent bath which is cooled to a predetermined temp. and maintained under pressure. F. R. ENNOS.

**Manufacture of fibrous sheet material.** J. C. PEABODY and A. BROWN, ASSRS. to F. B. HOPEWELL (U.S.P. 1,781,668, 11.11.30. Appl., 23.7.29).—Cellulosic material, particularly wood pulp, is partly xanthated so that it retains its fibrous structure and is then beaten either alone or in admixture with, *e.g.*, an equal quantity of untreated pulp and made into sheets. The resulting material is strong, does not disintegrate when immersed in H<sub>2</sub>O, and is suitable as a base for artificial leather. Softening agents such as glycerin may be incorporated. Xanthation is conveniently effected by impregnating the pulp (100 pts.) with 10% NaOH solution at 15—32° for 1 hr., pressing the mass to 300 pts., and treating it for about 1 hr. with 25 pts. of CS<sub>2</sub>. (Cf. B.P. 126,174; B., 1919, 458 A.) D. J. NORMAN.

**Manufacture of fibre board.** E. S. EDWARDS, ASSR. to PANELYTE CORP. (U.S.P. 1,778,147, 14.10.30. Appl., 16.5.29).—Kraft pulp, fusible asphaltic material (gilsonite), red gum or resin, calcium chloride, and borax are beaten together with water, and the mixture is pressed and dried. F. R. ENNOS.

**Manufacture of articles with surfaces having a pearl effect.** F. K. WICKEL (B.P. 347,888, 3.5.30).—Paper, leather, wood, etc. which has been coated with a suspension of powdered mica in an adhesive material and, if desired, with multicoloured patterns, is further coated with a transparent optically inactive layer (resin, albuminous substances, cellulose derivatives) and rubber with BiOCl or SbOCl to produce a "metallic mirror," through which the base material shows, thus producing the desired effect. F. R. ENNOS.

**Machines for decorticating fibre-containing foliate plants.** F. KRUPP GRUSONWERK A.-G. (B.P. 349,343, 19.6.30. Ger., 13.5.30).

**Manufacture of crêpe-like fabrics.** BRIT. CELANESE, LTD. (B.P. 349,121, 17.2.30. U.S., 15.2.29).

**[Wet-stretching] treatment of artificial silk threads.** F. J. GAHLERT (B.P. 349,232, 10.4.30. Ger., 10.4.29).

**Manufacture of [linen-surfaced] waxed papers.** WAXED-PAPERS, LTD., and W. T. CLOUT (B.P. 349,104, 10.1.30).

**Manufacture of [toilet] paper or paper-like material.** ROBINSON & SONS, LTD., and V. O. ROBINSON (B.P. 349,139, 28.2.30).

**Storing and mixing materials [paper pulp].—**See I. Glass. Transparent sheets.—See VIII. Treatment of surfaces. Fibrous compositions. Plastic masses. Adhesive.—See XIII. Absorbent for liquid O.—See XXII.

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

**Influence of electrolytes of different valencies on dyeing with substantive dyes.** P. P. VIKTOROV (Kolloid-Z., 1931, 55, 72—81).—The influence of electrolytes on the adsorption of the substantive dye "Benzoreinblau" has been studied. The electrolytes used can be arranged in the following order of effectiveness of cations, the sulphate being used in each case: NH<sub>4</sub><Na<K<Mg<Ni<Mn<Zn<Cd<Al. Greater amounts are adsorbed the higher is the valency of the cation. Little adsorption occurred with the dialysed dye, the addition of Na<sub>2</sub>SO<sub>4</sub> producing a tenfold increase, whilst Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> causes an increase of a further 30%. Anomalies were noted in the effects of Cu<sup>++</sup>, Cr<sup>+++</sup>, and Fe<sup>+++</sup> sulphates, where a different colour was given. The addition of gelatin increases the amount of dye taken up. E. S. HEDGES.

**Action of certain [organic] acids on cellulose.** E. HIBBERT (J. Soc. Dyers and Col., 1930, 46, 294—295).—The tendering effect of drying a solution of oxalic acid in cotton fabric is very much accelerated by simultaneous exposure of the fabric to sunlight; the tendering commences when the fabric becomes dry. If fabric be dried with oxalic acid under conditions such that inappreciable tendering occurs and the fabric be then exposed to sunlight or light from a Fadeometer, marked tendering occurs so that the fabric has a decreased affinity for direct dyes, an increased affinity for methylene-blue, and strong reducing power which can be detected by its high Cu number and reactivity towards *p*-nitrophenylhydrazine and Ermen's solution. A similar but less marked effect is obtained when other org. acids such as phthalic, tannic, and gallic acids are used; phthalic acid which is formed during the fading of cotton dyed with Purpurin (B., 1929, 715) produces definite tendering although less than that of oxalic acid. Tendering is also produced when an org. acid is dried into fabric which is then quickly ironed at 170°; the tendering is greatest with oxalic acid and less with tartaric, malic, citric, and phthalic acids, and viscose is much more readily tendered than cotton. A. J. HALL.

**Action of caustic soda on cellulose.—**See V. Laundry materials.—See VII.

See also A., June, 717, Action of CH<sub>2</sub>O on cellulose.

## PATENTS.

**Bleaching of [wood] pulp.** D. C. ANDREWS, ASSR. to NORTHWEST PAPER Co. (U.S.P. 1,782,800, 25.11.30. Appl., 23.6.26).—Wood pulp is bleached, in the form of a 10–20% slurry, in a wooden chamber provided with a false bottom. An alkaline solution of  $\text{CaOCl}_2$  is distributed over the slurry so that it percolates downwards and thereby displaces the  $\text{H}_2\text{O}$  present; the bleach liquor is then recirculated through the slurry until it is fully decolorised.

A. J. HALL.

**Immunisation of cotton and other cellulosic fibres.** CHEM. FABR. VORM. SANDOZ (B.P. 345,052, 19.2.30. Ger., 20.2.29).—In the alkalisation of cellulose etc. for immunising by esterification, the use of boiling dil. (12–14%) caustic alkali results in a more even product, especially in presence of phenols or cyclic alcohols, *e.g.*, cresol, cyclohexanol, which assist wetting.

C. HOLLINS.

**Dyeing or colouring of cellulose ethers and esters and materials containing them.** SOC. CHEM. IND. IN BASLE (B.P. 344,884, 9.12.29. Switz., 8.12.28).—Greenish-yellow shades are obtained on cellulose ethers or esters in textiles or varnishes by use of the dyes *p*-alkoxyaniline  $\rightarrow$  *p*-cresol. [Stat. ref.]

C. HOLLINS.

**Dyeing and printing with [soluble esters of leuco-]vat dyes.** DURAND & HUGENIN SOC. ANON. (B.P. 344,964, 23.12.29. Ger., 22.12.28).—Over-oxidation, even with excess of oxidant, in development of sol. esters of leuco-vat dyes, is prevented by addition of carboxylic or sulphonic acids of arylamines, *e.g.*, sulphanilic or diethylmetanilic acid, preferably as salts.

C. HOLLINS.

**Decorative treatment of textile fabrics.** CALICO PRINTERS' ASSOC., LTD., J. D. WEBSTER, and J. S. HEATON (B.P. 346,544, 13.3.30. Addn. to B.P. 326,823; B., 1930, 554).—Materials producing two or more different colours are used in the process of the prior patent. *E.g.*, fabric is prepared with Naphthol-AS, then printed "bang-through" with a mixture of Caledon Jade Green and Fast Scarlet R salt on one roller and a mixture of Indanthrene Brilliant Violet 4R and Fast Orange R salt on the other, and finally printed on one side with alkaline reducing paste. After steaming etc., the pattern appears in green and violet on one side of the fabric and in red and orange on the other.

C. HOLLINS.

**Manufacture of crimped artificial fibres and threads.** J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 346,799, 6.12.29).—Hardened threads or strips of the polymerisation products of butadiene hydrocarbons are crimped by immersion in organic swelling agents such as solutions or emulsions of cyclohexane,  $\text{C}_6\text{H}_6$ , ethers, esters, ketones, etc.

A. J. HALL.

**Degreasing of textile materials.** W. PFAFFENDORF, ASSR. to GEN. ANILINE WORKS, INC. (U.S.P. 1,780,885, 4.11.30. Appl., 26.5.27. Ger., 14.6.26).—The materials are washed with ethylene chloride at 0–80°.

H. ROYAL-DAWSON.

**Sizing of textile materials.** BRIT. CELANESE, LTD., H. DREYFUS, and W. I. TAYLOR (B.P. 346,267, 29.11.29).—Textiles, especially cellulose acetate materials, are impregnated with polymerised or partly polymerised

vinyl acetate (etc.) preferably dissolved in MeOH or  $\text{C}_6\text{H}_6$ . Polymerisation may also be effected on the fibre.

C. HOLLINS.

**Rubber-coated fabrics.** W. W. TRIGGS. FROM E. I. DU PONT DE NEMOURS & Co. (B.P. 347,826, 17.3.30).—Rubber-coated fabric suitable for automobile tops or upholstery is provided with an external protective layer by applying, before vulcanisation, a coat of asphalt varnish, after which vulcanisation is effected at a temp. which causes the asphalt to fuse to a smooth, chemically inert film. An improved result is obtained if a coat of oil varnish is applied before the asphalt varnish. The asphalt varnish itself may contain 2½–20 gal. of drying oil per 100 lb. of asphalt.

D. F. TWISS.

**Cloth fulling and washing machine.** D. GESSNER (B.P. 349,223, 7.4.30).

**Dye-works.**—See I. Detergents.—See III. Diazotisable fibres. Cellulose derivatives.—See V. Treatment of surfaces.—See XIII.

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

**Intensive [chamber] process for the manufacture of sulphuric acid.** E. OVSIANY (Chim. et Ind., 1931, 25, 832).—The author's apparatus, which is not described, effects an intimate contact of atomised liquid and gas under alternate pressure and expansion. Two forms of apparatus have been tested as an auxiliary in front of the two leading chambers of a set and gave a yield per unit space of 270–400 times the yield of an ordinary chamber. Applications to other gas-washing processes are suggested.

C. IRWIN.

**Production of phosphoric acid from phosphorites by volatilisation in an atmosphere of gaseous hydrocarbons.** C. PADOVANI and A. NARDELLA (Annali Chim. Appl., 1931, 21, 109–115).—A Land Pebble phosphorite containing  $\text{P}_2\text{O}_5$  30.59,  $\text{SiO}_2$  6.60,  $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$  2.36% was mixed with sufficient  $\text{SiO}_2$  to give the  $\text{P}_2\text{O}_5 : \text{SiO}_2$  ratio the value about 1 : 2 and heated at 1150–1200° in a current of natural gas composed largely of  $\text{CH}_4$ . More than 90% of the total P present was volatilised, all the H produced by the scission of the  $\text{CH}_4$  and about one half of the C (as CO) being recovered.

T. H. POPE.

**Ammoniation of superphosphate with anhydrous ammonia.** F. G. KEENEN (Amer. Fert., 1931, 74, [10], 20–22).—A description of the commercial process and of the material produced.

A. G. POLLARD.

**Limitation in capacity of a platinum catalyst for ammonia oxidation.** H. W. WEBB (J.S.C.I., 1931, 50, 128–131 r).—It is shown that the max. point in the curve connecting the yield of NO with the gas velocity in  $\text{NH}_3$  oxidation is mainly due to the reaction between  $\text{NH}_3$  and NO. Mixtures of these gases in equimol. proportions are found to explode in contact with activated Pt at 800°. The spacing apart of Pt gauze catalysts in  $\text{NH}_3$  oxidation is shown to be inadvisable.

**Value of volumetric methods for determining the basic constituents in quicklime and slaked**

**lime.** F. MACH and R. HERRMANN (*Z. anal. Chem.*, 1931, **84**, 1—14). The Mg and Ca silicates which are found in CaO and slaked lime are of very variable composition and dissolve to varying extents in all the solutions used in the analysis; in weak organic acids and solutions of sugars or of glycerin the solubility is relatively small but even conc. HCl does not decompose them completely. All attempts to effect a separation of the CaO and MgO from the silicates and carbonates by differential dissolution failed to give good results; decomp. by HCl followed by complete analysis gave results of sufficient accuracy for technical purposes.

A. R. POWELL.

**Commercial calcium nitrate [alleged fire risks in handling].** C. MATIGNON (*Chim. et Ind.*, 1931, **25**, 799—802, and *Compt. rend.*, 1931, **192**, 777—780).—Norwegian Ca nitrate, containing 13% N and 22—23% H<sub>2</sub>O, and the "ammoniacal" product of the Société Badoise, containing 5% NH<sub>4</sub>NO<sub>3</sub> and 14% H<sub>2</sub>O, have been examined. Both products are hygroscopic and their decomposition is endothermic, requiring a temp. above 500°. Intimate mixtures of Ca(NO<sub>3</sub>)<sub>2</sub> with powdered charcoal, sawdust, and jute from the bags in which it is packed failed to ignite below 300°. No record (since 1903) can be traced of a case of spontaneous ignition due to the commercial product. C. IRWIN.

**Detection of sodium silicate in laundry materials.** J. J. J. DINGEMANS (*Chem. Weekblad*, 1931, **28**, 229, 277—278).—A sample (1 g.), in aq. solution, after acidification with HCl is filtered and evaporated and after cooling, 10 c.c. of a 10% (NH<sub>4</sub>)<sub>2</sub>MoO<sub>4</sub> solution are added; the appearance of a yellow coloration indicates the presence of silicate, which is confirmed by the production of a blue coloration on addition of a strongly alkaline stannate solution. The presence of bleaching agents interferes, and in their presence SiO<sub>2</sub> must be sought in the ash. In laundry materials containing phosphate and bleaching agents, the phosphate is removed by treating the solution with magnesia mixture, and bleaching agents by boiling in alcoholic solution. SiO<sub>2</sub> may then be detected as described above.

H. F. GILLBE.

**Gravimetric determination of lead in solution of lead subacetate [U.S.P.].** J. L. MAYER (*J. Amer. Pharm. Assoc.*, 1931, **20**, 363—364).—10 c.c. of a solution of 5 g. of the subacetate in 50 c.c. of H<sub>2</sub>O and 5 drops of HNO<sub>3</sub> are diluted to 300 c.c. and heated to 90—95°; aq. NH<sub>3</sub> is added to incipient precipitation and then a slight excess of 10% HNO<sub>3</sub>. To the hot solution 50 c.c. of hot 10% K<sub>2</sub>CrO<sub>4</sub> are added with stirring over a period of 40 sec. The ppt. is filtered off and dried at 140—150°; wt.  $\times 0.6410 = \% \text{ Pb}$ .

E. H. SHARPLES.

**Lead in basic bismuth nitrate.** N. SCHOORL (*Pharm. Weekblad*, 1931, **68**, 277—279).—The usual pharmacopœia test for absence of Pb—a clear solution with 0.5 g. in 2 c.c. H<sub>2</sub>SO<sub>4</sub>—gives a positive result if carried out at room temp. if the Pb content exceeds 0.1%. At higher temp. a ppt. of basic Bi sulphate is formed at once; even in the cold, this ppt. forms after 24 hr.

S. I. LEVY.

**Alkali bismuth tartrates from the chemical and biological standpoint.** A. E. JURIST and W. G.

CHRISTANSEN (*J. Amer. Pharm. Assoc.*, 1931, **20**, 349—355).—The behaviour of these salts in aq. solution containing stabilising substances, and experiments on the toxicity, adsorption, and local action of the compounds when injected intramuscularly are described. The most stable solutions are prepared using glycerin or, preferably, for biological use, 25% sucrose solution suitably buffered. The alkali Bi tartrates when injected cause severe local reactions, including pain, swelling, and lump formation, and though they have low toxicity and are readily absorbed they are not satisfactory therapeutic agents for syphilis. The local reactions could not be materially reduced by modifying the aq. solvent. E. H. SHARPLES.

**Preparation of water-soluble sodium bismuth citrate.** W. F. VON OETTINGEN (*J. Amer. Pharm. Assoc.*, 1931, **20**, 426—429).—Freshly prepared, moist Bi(OH)<sub>3</sub> when shaken with 0.5*M*-citric acid forms dibismuthyl-citric acid, which is dissolved in NaOH and the pure monosodium dibismuthylcitrate precipitated by addition of EtOH. Details are given (cf. A., 1927, 1109).

E. H. SHARPLES.

**Compounds of titanium with glycol and phthalic acid, and ethyl ortho-silicate and -titanate as film-forming materials.** H. A. GARDNER and E. BIELOUSS (*Amer. Paint and Varnish Manufs.' Assoc.*, 1930, *Circ.* 366, 327—337).—Indefinite products, apparently of orthosilicate type, obtained by heating TiCl<sub>4</sub> with "pentasol," ethylene glycol, and glycerol until HCl ceases to be evolved, and by the action of phthalic anhydride in 50% EtOH on Ti(OEt)<sub>4</sub> followed by glycerol at the b.p., are described. The glycerol and glycol esters still contain Cl and are sol. in H<sub>2</sub>O, with hydrolysis in the latter case to a colloidal Ti(OH)<sub>4</sub> solution stable at 130—140°; the former forms films sol. in H<sub>2</sub>O, but the latter produces good films on a variety of surfaces and is compatible with cellulose esters. The glycol ester in contact with nitrocellulose in AcOBu produces a white Ti pigment in a finely-dispersed state, and gives a white ppt. with rubber latex, as also does TiCl<sub>4</sub>. Films of good adhesive strength were not obtained with Ti(OEt)<sub>4</sub> or Si(OEt)<sub>4</sub>. Ti(OEt)<sub>4</sub> is useless as an anti-knock agent.

H. A. PIGGOTT.

**Absorption and regeneration of hydrogen sulphide by alkaline liquids.** H. A. J. PIETERS and G. SMEETS (*Chem. Weekblad*, 1931, **28**, 246—249).—The efficacy of Na<sub>2</sub>CO<sub>3</sub> solution for the removal of H<sub>2</sub>S from coal gas increases with increase of concentration up to about 3%; 1 vol. of the solution is sufficient to reduce the sulphide content of 3 vols. of gas containing 0.5% H<sub>2</sub>S to 90% of its initial value. The resulting solution does not evolve H<sub>2</sub>S if CO<sub>2</sub>-free air is passed through it, but by treatment with a rapid current of air it is partly regenerated, most of the S being evolved as H<sub>2</sub>S and the remainder oxidised to thiosulphate. Bicarbonate solutions are not satisfactory for the washing process, saturated NaHCO<sub>3</sub> solution being only about one half as effective as 3% Na<sub>2</sub>CO<sub>3</sub> solution. There is no advantage in using K<sub>2</sub>CO<sub>3</sub>. HCN is absorbed, and is converted into M'CNO and M'CNS during regeneration.

H. F. GILLBE.

**Acid pumps.**—See I. Acetate from soda-pulp liquors.—See V. Anglesite.—See VIII. S-resistant

alloys.—See X. Photosensitised oxidation of  $\text{NH}_3$  and  $\text{NH}_4$  salts.—See XVI.

See also A., June, 677, Adsorption of H on charcoal. 678, Sorption of H by Pt metals. 695, Ca oxalate from Ca cyanamide. 696, Phosphorescent boric acid. 697, Iodine residues. 698, Specifications for reagents. 699, Determination of  $\text{H}_2\text{O}_2$ . Test for halogens. Bromo-iodometry. 701, Separation of Ca and Mg by molybdate. 718—9, Preparation of organo-Mg halides. 775, I in algæ.

## PATENTS.

**Manufacture of strontium oxide.** J. B. PIERCE, JUN., Assr. to BARIUM REDUCTION CORP. (U.S.P. 1,782,830, 25.11.30. Appl., 29.1.27).—Amorphous  $\text{SrCO}_3$ , prepared by precipitating a solution of an alkaline Sr compound with  $\text{CO}_2$  or a carbonate, is mixed with ashless C black, and the mixture calcined in a closed muffle at  $1200^\circ$ . The easily oxidisable SrO produced may be used for preparing  $\text{SrO}_2$ . W. J. WRIGHT.

**Production of anhydrous chlorides.** SOC. DE PROD. CHIM. DES TERRES RARES (B.P. 344,601, 18.1.30. Fr., 18.1.29).—Chlorides of Mg, Ca, Th, etc. which have been partly dehydrated and thereby rendered porous are dehydrated by treatment in an externally-heated vertical retort, in a countercurrent of HCl at a temp. below the softening point, e.g.,  $400$ — $450^\circ$  at the top of the tower and  $580$ — $620^\circ$  at the hottest point. A product is claimed containing  $\text{CaCl}_2$  0.05—0.5% and insol. material  $< 0.5\%$ . L. A. COLES.

**Cleansing agents [for metals etc.].** CHEM. FABR. J. A. BENCKISER G.M.B.H., and F. DRAISBACH (B.P. 347,072, 25.7.30. Ger., 23.4.30).—Up to 20% of  $\text{Al}_2(\text{SO}_4)_3$  is added to  $\text{Na}_3\text{PO}_4$  or to a mixture of that compound with soda. H. ROYAL-DAWSON.

**Production of compositions containing compounds of alginic acid and product thereof.** L. L. BURGESS, Assr. to PLASTIC, INC. (U.S.P. 1,782,887, 25.11.30. Appl., 14.7.28).—Alginic acid or a composition containing it, dissolved in an alkaline reagent, is treated with a solution of a hydrocarbon, e.g., paraffin wax in  $\text{CCl}_4$ , and the product is precipitated by addition of a mineral acid. Finely-divided Cu or Zn may be added to the reaction mixture, precipitation then occurring after several hr. without addition of acid. The washed and dried ppt. is extracted with  $\text{CCl}_4$ , and the product plasticised with aq.  $\text{NH}_3$  and rendered insol. by addition of a suitable metal oxide. W. J. WRIGHT.

**Treatment of dolomite and other natural carbonates of magnesium.** SOC. DE PROD. CHIM. DES TERRES RARES (B.P. 348,758, 10.3.30. Fr., 11.3.29).—See U.S.P. 1,778,659; B., 1931, 539.

**Manufacture of cakes of carbonic snow.** H. FIEVET (B.P. 348,581, 13.2.30).

**N-H mixture.**—See II. Lute.—See IX. Mixed fertilisers.—See XVI. Absorbent [for liquid O]. Liquid-O explosives.—See XXII.

## VIII.—GLASS; CERAMICS.

**Continuous kilns as applied to the pottery industry.** R. WHITFIELD (Trans. Ceram. Soc., 1931, 30, 150—168).—The gas-fired "Robertson" tunnel

kiln and an electrically heated decorating kiln are described in detail. The former has been applied to all types of ceramic ware, including that produced by salt-glazing. Close control of atm. heating and cooling rates etc. has been provided. The thermal efficiency is very high. J. A. SUGDEN.

**Continuous kilns for the burning of clay products.** J. WILLIAMSON (Trans. Ceram. Soc., 1931, 30, 21—32).—Descriptions are given of the different types of continuous kilns, including the "Manchester," "Staffordshire," "Ideal," "Simplex," and the various tunnel kilns, and their adaptability to the firing of clay wares is indicated. F. SALT.

**Alkali-lime-silica glasses.** G. KEPPELER and K. BÖHMERT (Keram. Rundsch., 1930, 38, 663—666, 697—700, 711—713, 738—742, 787—788; Chem. Zentr., 1931, i, 1341).—Devitrification limits, m.p., sp. gr., and extraction values of K glasses were determined. For similar mol. composition Na glasses are better; except with glasses high in K or Ca, for similar wt. composition K glasses are the better. Na-K-Ca-SiO<sub>2</sub> glasses showed no extraction min. contrary to Peddle's result, which is attributed to devitrification. A. A. ELDRIDGE.

**Mercury glasses.** F. H. ZSCHACKE (Glastech. Ber., 1930, 8, 519—525; Chem. Zentr., 1931, i, 1341).—When  $\text{HgCO}_3$  is added to the batch only a vanishingly small trace remains in the glass. The colour is golden-yellow to bluish-green, depending on the temp. and atm., and is attributed to colloidal Hg. Spectroscopic observations are recorded. A. A. ELDRIDGE.

**Sulphur trioxide content of glass. Clarification.** H. JEBSEN-MARWEDEL and A. BECKER (Glastech. Ber., 1930, 8, 525—529; Chem. Zentr., 1931, i, 1340—1341).—The effect of various furnace atm. on the removal of  $\text{SO}_3$  from window glass was examined. Vigorous reduction at  $1350^\circ$  removes the  $\text{SO}_3$ , whilst in an oxidising atm. a temp. of  $1500^\circ$  is necessary. The gradient of the removal is not const. for a particular temp., but appears to depend on the rate of heating. A. A. ELDRIDGE.

**Permeability of glasses to ultra-violet radiation.** II. P. GLARD, P. SWINGS, and A. HAUTOT (Bull. Acad. roy. Belg., 1931, [v], 17, 362—368; cf. B., 1930, 557).—The transparency of glasses to ultra-violet light is diminished by addition of As, Sb, Cr, Co, Mn, and Fe oxides. The effect of NiO is generally in the same sense, but depends on the composition of the glass. C. W. GIBBY.

**Plastic flow measurements [in clay] and their bearing on the plasticity problem.** G. W. S. BLAIR (Trans. Ceram. Soc., 1931, 30, 138—149).—Measurements of the rate of flow of slips through a capillary-tube type of plastometer were made. The curve showing the relationship between the rate of flow and pressure may be divided into four regions: (1) no flow; (2) linear flow—the paste flows through the tube as a solid plug; (3) mixed flow—the paste has started to flow telescopically, but there is still a plug in the centre; (4) telescopic or stream-line flow as a true fluid. The intercept of region (4) on the pressure axis,  $a$ , is Bingham's "yield value," and the point where region (3) begins,  $b$ , is the "critical shearing stress." Unsuccessful attempts were made in a series of clays

to correlate the values of  $a$  and  $b$  with the plasticity as measured by Bischof's "wire method" (plasticity is inversely proportional to diam. of the thinnest wire that can be rolled without breaking). All these attempts were made at the same arbitrarily chosen  $H_2O$  content for all the clays. The conception of "flow plasticity" is developed by the author. The underlying idea is to compare the values of  $b$  at the same arbitrarily chosen value of  $a$  for all the clays, thus making the comparison at corresponding (but not identical) moisture contents when the hydration phenomena mask the variations in plasticity. This new conception enabled many clays to be placed in correct order of plasticity and the observation that the combined action of  $NaOH$  and  $NaCl$  on a clay has a much greater plasticising effect than the action of either alone was confirmed. Certain plastic-flow constants are shown to be systematically affected by the addition of  $CaCO_3$  and  $CaO$ . It is pointed out, however, that the flow-plasticity method must be used with caution, since the constants are somewhat sensitive to the previous moisture-history of the material.

J. A. SUGDEN.

**Grog.** C. R. F. THRELFALL (Trans. Ceram. Soc., 1931, 30, 1—18).—Grog is defined as "any material present in a wet batch in a non-plastic or comparatively non-plastic form," and therefore includes both fired and unfired material. The term "green grog" is given to the nodules found in fired firebricks and caused by the coarser fractions in the green clay. The functions of grog, as thus defined, in refractory bodies are discussed in detail in relation to porosity, shrinkage, etc., and to the various processes of manufacture.

F. SALT.

**Effects of various kiln gases on the burning of bright gold for gilding ceramic ware.** A. NAKATSUCHI (J. Soc. Chem. Ind., Japan, 1931, 34, 67—69B).—The results of burning "bright gold" in atm. of  $CO$ ,  $H_2$ ,  $SO_2$ ,  $H_2S$ ,  $CO_2$ ,  $O_2$ ,  $N_2$ , and  $H_2O$  vapour are reported. The first five gases are injurious; the best atm. is one of  $N_2$  with a little  $O_2$ , but this is impracticable. The suggested burning process for bright gold is to remove the injurious gases from the kiln at about  $300^\circ$  before raising the temp. to  $750^\circ$ .

F. SALT.

**Moisture expansion of glazes and other ceramic finishes.** H. G. SCHURECHT and G. R. POLE (Bur. Stand. J. Res., 1931, 6, 457—463).—When subjected to artificial weathering by the action of steam at  $150\text{ lb./in.}^2$  for 1 hr., the average expansion of slip finishes was 0.033%, of matt glazes 0.011%, of vitreous slip 0.005%, and of lustrous glazes 0.004%. The necessity of considering this type of expansion when adapting a glaze or finish to a ceramic body is emphasised, since it is often of greater importance as regards the crazing of the glaze than is the elasticity or tensile strength. The ring test may be employed for comparative moisture expansion tests of ceramic finishes if the rings are all made from the same body and are treated in the same manner.

H. F. GILLBE.

**Works' tests on refractories and service conditions.** R. J. SARJANT (Trans. Ceram. Soc., 1931, 30, 46—65).—Various tests for refractories are discussed in relation to their serviceability in steel works' practice. The refractoriness-under-load test has definite value in

the case of firebricks and monolithic refractories; results with this test, using a load of  $50\text{ lb./in.}^2$ , are given and discussed. Useful data are obtainable from the change in porosity and density on firing refractories to a moderately high temp. ( $1450^\circ$ ). Simulative slag-attack tests are useful, provided that they are classified according to the type of refractory and use. Spalling tests are of doubtful value, whereas thermal conductivity tests provide important information in relation to industrial practice. The service test remains the ultimate arbiter in the selection of refractories.

F. SALT.

**Mode of formation of anglesite.** V. L. BILLET [with M. BEULCKE] (Natuurwetensch. Tijds., 1931, 13, 67—68).—Anglesite, identical with the natural mineral, has been found deposited on the walls of ovens in which Pb-glazed articles have been fired.

H. F. GILLBE.

See also A., June, 700, **Silicate analysis.** 707, **Doubrovka kaolin. Clay from Tshasov-Jar deposits.**

#### PATENTS.

**Synthetic oriental emerald or emerald-green sapphire.** E. G. SANDMEIER, ASSR. to SWISS JEWEL CO. SOC. ANON. (U.S.P. 1,775,867, 16.9.30. Appl., 28.2.28. Switz., 19.1.28).—A mixture of oxides or salts to give the composition  $Al_2O_3$  98.6%,  $CoO$  0.986%,  $V_2O_5$  0.119%,  $NiO$  0.295%, when fused according to the Verneuil process (cf. U.S.P. 1,004,505; B., 1911, 1254), gives a synthetic gem resembling the oriental emerald.

M. PARKIN.

**Synthetic spinels.** E. G. SANDMEIER, ASSR. to SWISS JEWEL CO. SOC. ANON. (U.S.P. 1,775,868—1,775,870, 16.9.30. Appl., [A, B] 28.2.28, [C] 3.5.28. Switz., [A, B] 19.1.28).—(A) Green spinels are produced synthetically by fusing according to the Verneuil process (see preceding abstract) a mixture of oxides or salts to give the composition  $Al_2O_3$  82.53%,  $MgO$  16.506%,  $TiO_2$  0.066%,  $Cr_2O_3$  0.099%. (B) A spinel like Brazilian emerald is similarly produced by melting a batch to give the composition  $Al_2O_3$  82.6%,  $MgO$  16.5%,  $CoO$  0.004%,  $V_2O_5$  0.04%,  $MnO$  0.83%. (C) A spinel resembling blue zircon results from a similar fusion giving the composition  $Al_2O_3$  86%,  $MgO$  10%,  $MnO$  3%, and traces of  $CoO$  and  $TiO_2$ .

M. PARKIN.

**Splinterless glass.** BRIT. CELANESE, LTD. (B.P. 347,219, 22.1.30. U.S., 22.1.29).—The glass sheets and reinforcing layer comprising a cellulose derivative are heated at above  $50^\circ$  (preferably at  $100$ — $200^\circ$ ), separately or after assemblage, before they are united by compression.

L. A. COLES.

**Manufacture of compound transparent [glass] sheets.** SOC. DES USINES CHIM. RHÔNE-POULENC (B.P. 345,176, 21.6.30. Fr., 22.6.29. Addn. to B.P. 316,955; B., 1930, 1029).—Safety glass assembled as described in the prior process without the use of volatile solvents is subsequently heated to improve the adhesion of the glass to the core, e.g., for 42 hr. at  $45^\circ$ , or 52 at  $65^\circ$  for materials assembled without de-gassing *in vacuo*, or for 4 hr. at  $85^\circ$  when they had been so de-gassed.

M. PARKIN.

**Compound transparent sheets.** DUPLATE CORP., Asses. of J. H. SHERTS and R. E. HAMILL (B.P. 347,455,

7.4.30. U.S., 9.4.29).—Edge defects in composite sheets comprising glass and a pyroxylin plastic are cured or prevented by subjecting the finished sheets to further compression in the presence of a heated liquid, preferably a high-boiling solvent of the plastic.

L. A. COLES.

**Ceramic materials.** W. W. TRIGGS. From R. POLLAK (B.P. 347,486, 30.4.30).—Mixtures of hydrated Mg silicates free from alkali compounds (serpentine, chlorite, soapstone, etc., or artificial products) with not more than 10% of alkaline-earth compounds and  $\text{Al}_2\text{O}_3$ , with the addition of graphite if desired, are calcined for the production of refractory linings, stills, emery wheels, etc.

L. A. COLES.

**Manufacture of ceramic products.** V. M. GOLDSCHMIDT (U.S.P. 1,782,295, 18.11.30. Appl., 11.12.28. Ger., 18.8.27).—The raw materials include Mg hydro-silicates, e.g., serpentine, with the exception of talcum, and other substances rich in Mg, e.g., asbestos waste, these being heated to a temp. at which Mg orthosilicate is formed without fusion.

W. J. WRIGHT.

**Manufacture or burning of bricks and the like, and fuel for use therewith.** J. ONIONS (B.P. 347,266, 24.1.30).—Perforated tiles or blocks of fuel are interspaced with the bricks as they are set up in the kiln for burning.

L. A. COLES.

**Refractory structure.** F. L. ARENSBERG and A. J. JACKMAN, Assis. to VESUVIUS CRUCIBLE Co. (U.S.P. 1,782,023, 18.11.30. Appl., 8.2.29).—The tendency of refractory structures to spall is reduced if materials of different thermal conductivity are used, one predominating near one face and extending to a considerable depth, and the other, with a lower thermal conductivity and consequent greater susceptibility to spalling, forming the remainder of the structure and being protected by the former.

W. J. WRIGHT.

**Lehrs for annealing glassware.** UNITED GLASS BOTTLE MANUFRS., LTD., and T. C. MOORSHEAD (B.P. 348,872, 10.5.30).

**Manufacture of glass tubes and rods.** J. R. C. JORGENSEN, Assec. of L. BOSCH (B.P. 348,857, 28.4.30. Austr., 6.11.29).

**Brick-making machinery.** C. WHITTAKER & Co., LTD., and N. WHITTAKER (B.P. 349,187, 15.3.30).

Lute.—See IX. Treatment of surfaces.—See XIII.

## IX.—BUILDING MATERIALS.

**Dissolution velocity of Portland cements.** T. YOSHIOKA and K. KUMAGAE (J. Soc. Chem. Ind., Japan, 1931, 34, 89B).—Dyckerhoff Doppel, Rapid-hardening, Super, Special, Velo, and Japanese normal cements were extracted with  $\text{H}_2\text{O}$  in the absence of  $\text{CO}_2$  and determinations of the electrical conductivity and alkalinity of the extracts were made. Cements of the Portland type show a common and characteristic time-reaction tendency of hydration. Cements of similar chemical composition are not necessarily similar in velocity of hydration, and marked differences are observed in the rate of increase of alkalinity of the solution. The concentration of CaO in the aq. phase increases with time to a saturation limit, but the time curve shows a

point of arrest which is common to all the cements examined.

E. S. HEDGES.

**Effect of free lime in Portland cement clinker on the solubility of lime and alumina.** K. KOYANAGI (J. Soc. Chem. Ind., Japan, 1931, 34, 56—58B).—In well-burned clinker, low in free CaO, the solubility of  $\text{Al}_2\text{O}_3$  is comparatively high, that of CaO low. With increasing free CaO content, the solubility of the  $\text{Al}_2\text{O}_3$  decreases, whilst that of the CaO increases rapidly. Addition of  $\text{H}_2\text{O}$  to clinker containing much free CaO produces a large quantity of a flocky gel substance, and formation of  $\text{Ca}(\text{OH})_2$  takes place much more quickly than with well-burned clinker.

F. SALT.

**Rôle of calcium hydroxide in the hardening of Portland cement.** F. F. TIPPMMANN (Kolloid-Z., 1931, 55, 85—107).—The setting and hardening processes of Portland cement are illustrated by photomicrographs. The needle-like crystals which are observed in the presence of an excess of  $\text{H}_2\text{O}$  are not Ca hydrogen silicate, as was formerly supposed, but consist of  $\text{Ca}(\text{OH})_2$ . They can be produced by the hydration of pure CaO in the presence of gypsum, which stimulates the crystallisation. Gypsum also assists in the transformation of amorphous into cryst.  $\text{Ca}(\text{OH})_2$ , and the hitherto unexplained effect of gypsum on the hardening of Portland cement is attributed to this cause. Crystalloidal and colloidal theories of the hardening of cement are reviewed. It is claimed that the process can be understood only by considering both the cryst. and colloidal hydrated products.

E. S. HEDGES.

**Hydration phenomena of cements.** T. YOSHIOKA, K. KUMAGAE, and H. TAKATA (J. Soc. Chem. Ind., Japan, 1931, 34, 87—88B).—Microscopical observations of the slow hydration of 5 types of cement in a limited excess of  $\text{H}_2\text{O}$  are recorded. Velo cement and blast-furnace cement were rich in needle crystals, whilst Special, Super, and normal Portland cements formed needle-like crystals during the initial course of the process, which later disappeared.

E. S. HEDGES.

**Swelling of cement grains on hydration.** T. YOSHIOKA and H. TAKATA (J. Soc. Chem. Ind., Japan, 1931, 34, 88B).—Grains of normal Portland cement undergo marked swelling during hydration, the original linear dimensions increasing by as much as 45%. Special, Super, and Velo cements undergo a total decrease of vol. when mixed with  $\text{H}_2\text{O}$ . Probably swelling occurs with these cements also, but vol. increase due to this cause is more than counterbalanced by the decrease due to dissolution of certain constituents of the grains.

E. S. HEDGES.

**Durability and strength of bond between mortar and brick.** L. A. PALMER and J. V. HALL, JUN. (Bur. Stand. J. Res., 1931, 6, 473—492).—The porosity of the brick has but little influence on the strength of the mortar bond. The principal factors affecting the durability, assuming sufficient wetting of the brick, are the pressure on the joint, freedom of movement during shrinkage of the mortar, and the type of mortar used. The ratio strength of bond : tensile strength is greater for a 1 : 1 : 6 CaO-cement-sand mortar than for a 1 : 3 cement-sand mortar, but the percentage of

failures is rather greater with the former. Metal lugs in the mortar, by preventing movement as the mortar contracts on ageing, reduce the durability of the joint. Disintegration of the mortar or of the brick is not a common cause of failure of a joint. H. F. GILLBE.

**Retrogression of porous concretes.** G. NATTA and C. G. FONTANA (*Giorn. Chim. Ind. Appl.*, 1931, 13, 173—178).—Investigation of the slow falling-off in the mechanical properties of the concrete in a reinforced-concrete structure revealed insufficient gravel or coarse sand, excess of fine sand, and excess of  $H_2O$ . The very high porosity of the concrete and the large sp. area of the mortar, due mainly to the faulty granulometric composition of the sand, led to pronounced hydrolysis, with carbonatation of the CaO produced. In slightly porous concretes rich in cement, the gradual increase in vol. accompanying such hydrolysis and carbonatation results in greater compactness and mechanical strength; hydrolysis is slow, since the increase in vol. gives lessened porosity. Poor, porous cements undergo rapid hydrolysis and carbonatation and show negligible increase in strength with time. T. H. POPE.

**Nature of the building stone employed at Salamanca; the patina and its artificial production.** I. RIBAS and (SRTA.) P. DE PRADA (*Anal. Fis. Quím.*, 1931, 29, 211—220).—The stone quarried near Salamanca consists principally of quartz and felspar. The patina is of practically the same chemical composition as the underlying stone, and its formation is ascribed to dehydration and consequent change of structure or orientation of the  $Fe_2O_3$  in the surface layer; the Ti present (0.4—0.5%  $TiO_2$ ) does not take part in the change. The patina may be produced artificially by applying to the stone an aq. suspension of precipitated  $Fe_2O_3$  and powdered stone. H. F. GILLBE.

**Durability of Philippine woods against fungi.** O. A. REINKING and C. J. HUMPHREY (*Philippine J. Sci.*, 1931, 45, 77—89).—The relative durability of Philippine woods against fungi, as determined by laboratory tests, agrees with that obtained under service conditions. D. K. MOORE.

**Thermal conductivity.**—See I.

#### PATENTS.

**Rotary [cement] kilns.** J. S. FASTING (B.P. 347,665, 22.11.29).—A portion of the length of a rotary kiln is subdivided into a number of cylindrical sections grouped round the axis and spaced apart so that air can circulate around them; they may either be fixed to the main tube of the kiln or be separate from it and driven at a different speed. [Stat. ref.] B. M. VENABLES.

**Manufacture of Portland cement.** F. W. HUBER (U.S.P. 1,781,232, 11.11.30. Appl., 19.3.30).—In making cement from raw materials containing  $Ca_3(PO_4)_2$ ,  $Fe_2O_3$  is added in greater mol. ratio than the  $P_2O_5$  radical. During calcination all the  $P_2O_5$  is converted into  $FePO_4$  and the residual Fe becomes effective as a mineralising agent. C. A. KING.

**Hydraulic cement and other calcareous plastics.** W. A. COLLINGS, Assr. to SILICA PRODUCTS Co. (U.S.P. 1,755,502, 22.4.30. Appl., 17.11.27).—The rate of ageing and the final mechanical strength of concrete is

increased if 5—10% of a naturally swelling clay (bentonite) and NaCl are added to Portland cement. The proportion of NaCl required depends on the amount occurring naturally in the bentonite, but is usually about 10% of the wt. of the clay. C. A. KING.

**Asbestos mortar.** I. KOTANI (B.P. 345,727, 20.1.30).—A mixture of crushed, air-blown asbestos fibre with Portland cement and sand is stirred and kneaded with  $H_2O$ . H. ROYAL-DAWSON.

**Rendering porous structural materials [stone-work] water- or weather-proof.** ELEKTROCHEM. WERKE MÜNCHEN A.-G. (B.P. 347,514, 29.5.30. Ger., 22.7.29).—The materials are treated with aq. emulsions of fatty oils (linseed oil) prepared with the smallest quantity of an emulsifier necessary to yield a stable emulsion which coagulates irreversibly on drying. L. A. COLES.

**Making a compound for mixing with cements.** R. BÜHMAN (U.S.P. 1,781,267, 11.11.30. Appl., 5.4.24).—A mixture, used especially for pumping into the cases of oil wells, consists of a cement slurry to which about 1.5% of  $CaCl_2$  and 1% of  $FeCl_3$  have been added. The  $FeCl_3$  is thereby converted into  $Fe(OH)_3$ , which retards the initial set of the slurry, the final set being accelerated. C. A. KING.

**Utilisation of blast-furnace slags.** P. P. BUDNIKOV (B.P. 347,357, 17.2.30).—The slag is ground with anhydrite and/or gypsum cement, insol. anhydrite or Estrich gypsum, or dolomite burned at 500—800°;  $CaCO_3$ , dolomite, fluorspar, phosphorite, or mixtures of these, may also be added. Alternatively, the slag is ground wet and the other constituents are added during the drying. L. A. COLES.

**Asphalt emulsions.** P. M. TRAVIS and A. L. HALVORSEN, Assrs. to EMULSION PROCESS CORP. (U.S.P. 1,757,102—3, 6.5.30. Appl., [A] 10.1.28, [B] 29.12.28).—(A) An emulsion of asphalt with  $H_2O$  contains not more than 1% (0.1—0.4%) of Na silicate and not more than 0.5% of a fatty acid, preferably oleic acid. (B) As the emulsifying agent, not more than 0.75% of  $Na_3PO_4$  by wt. on the emulsion is used. C. A. KING.

**Manufacture of an emulsion of tar for road-making.** UNION CHIM. BELGE, SOC. ANON. (B.P. 344,490, 2.12.29. Belg., 9.7.29).—Coal tar, fractionally distilled up to 270° to remove  $H_2O$ , light oils, and  $C_{10}H_8$ , is mixed at above room temp. with soap solution and potato fecula. H. ROYAL-DAWSON.

**Manufacture of road-building material.** S. S. SADTLER, Assr. to AMESITE ASPHALT Co. (U.S.P. 1,758,913—4, 13.5.30. Appl., [A] 28.5.29, [B] 28.6.29).—(A) Suitable aggregate is treated in a pug mill with kerosene, light coal tar, or other asphalt solvent and an aq. dispersion of rubber latex. Later a substantial coating of bitumen of 70—130 penetration is applied and 0.2%—1% of CaO is added to toughen the asphalt. The proportion of rubber is 0.2—2% of the bitumen used. (B) To ensure a satisfactory coating of the aggregate with rubber before applying bitumen, 0.25—1 pt. of rubber in 8—12 pts. of solvent is used for 1900 pts. of aggregate, which is then treated with 35—140 pts. of bitumen. C. A. KING.

**Production of premixed asphalt composition.** J. S. DOWNARD (U.S.P. 1,781,105, 11.11.30. Appl., 6.3.26).—Asphalted aggregate is coated with  $\text{CaCl}_2$  solution to keep the surface wet and prevent adhesion. At any subsequent time the mass is treated with a precipitating agent, e.g.,  $(\text{NH}_4)_2\text{C}_2\text{O}_4$ , which renders the mass adhesive and capable of being consolidated.

C. A. KING.

**Floor coverings.** N. KENYON, and SPEN RUBBER WORKS, LTD. (B.P. 345,700, 8.1.30).—Rubber, cork powder, S, ZnO, whitening, French chalk, and paraffin wax are mixed together and vulcanised.

H. ROYAL-DAWSON.

**Lute.** R. B. McCARTY, Assr. to GULF REFINING CO. (U.S.P. 1,782,932, 25.11.30. Appl., 6.9.27).—A luting material for withstanding the action of  $\text{Cl}_2$  at high temp. is prepared by mixing 65% of finely-ground, burnt fireclay, preferably obtained from the lining of  $\text{AlCl}_3$  chambers, with 35% of NaOH solution (*d* 1.384).

W. J. WRIGHT.

**Cementing together two surfaces of identical or different substances.** T. GOLDSCHMIDT A.-G. (B.P. 347,242, 20.12.29. Ger., 2.2.29).—A porous carrier, e.g., tissue paper, impregnated with a liquid artificial resin or intermediate condensation product, to which glycerin may be added, is interposed between the surfaces of, e.g., sheets of wood, and the whole is united by heat and pressure.

L. A. COLES.

**Colouring of concrete [by spraying with water paints].** J. F. BARR (B.P. 347,523, 5.1.30).

**Production of artificial marble.** A. BAUMANN (B.P. 349,348, 26.6.30).

Workshops. Mixing of materials.—See I.

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

**Surface pyrometers [for metals].** W. CLAUS and R. HASE (Z. Metallk., 1931, 23, 120—123).—Methods of measuring the temp. of metal surfaces by means of special pyrometers are described and the sources of error discussed.

A. R. POWELL.

**Detection of sulphur in steel sections by the impression method.** H. J. VAN ROYEN and E. AMMERMANN (Arch. Eisenhüttenw., 1930—1, 4, 435—438; Stahl u. Eisen, 1931, 51, 462—463).—The yellow colour of  $\text{HgCl}_2$  paper prints obtained from steel sections is invariably due to S and not to P, but the test is not sufficiently delicate as white or black Hg compounds containing S may also be formed under certain conditions. A more certain test comprises covering the surface of the steel with a gelatin paper impregnated with a solution containing 1.25%  $\text{Cd}(\text{OAc})_2$ , 8%  $\text{AcOH}$ , and 2.5%  $\text{H}_2\text{SO}_4$ , whereby  $\text{CdS}$  is formed in the paper covering the sulphide inclusions. The paper is washed in running  $\text{H}_2\text{O}$  and developed by immersion in a solution containing 120 g.  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  and 120 c.c. conc.  $\text{H}_2\text{SO}_4$  per litre, which converts the  $\text{CdS}$  into black  $\text{CuS}$ .

A. R. POWELL.

**Factors affecting corrosion of buried steel.** F. L. BASSETT (J.S.C.I., 1931, 50, 161—166 T).—The

wt. losses of 480 specimens of two brands of mild steel in 80 samples of damp soils were recorded after periods of 1, 2, and 3 months. The soils were marls and clays from the Iraq-Syrian desert, with variable proportions of sand, and chloride salinity from zero to 27.8% NaCl equiv. The  $p_{\text{H}}$  values were all in the sub-alkaline range, and corrosion took the usual course of localised anodic pitting. Soils with very low chloride, up to 0.2% NaCl equiv., showed in each series a higher average attack than salt-free soils; above about 0.2%, however, the attack is diminished, and in soils containing more than 1% NaCl equiv. the repression of corrosion becomes strongly marked. Diminished corrosive attack in the salted soils is also found to be correlated on the average with increased water retentivity, and is ascribed to hindrance of O diffusion. Hygroscopicity of salted soils therefore becomes a controlling factor which at quite low concentrations may reverse the normal stimulation of Cl ion for corrosion, by reducing the O supply to the cathodic areas of the plate.

**[Tensile] resistance of ordinary steels at high temperatures.** L. GUILLET, J. GALIBOURG, and M. SAMSOEN (Compt. rend., 1931, 192, 861—863).—Tensile tests at 225°, 325°, 425°, and 525° carried out as previously (cf. B., 1929, 601, 752) are detailed regarding four ordinary steels containing, respectively, (in %) C 0.15, 0.17, 0.18, 0.35; Si 0.07, 0.10, 0.11, 0.0008; Mn 0.32, 0.53, 0.58, 0.60; S 0.028, 0.027, 0.022, 0.020; P 0.017, 0.038, 0.022, 0.027, and having breaking stresses at room temp. of 37.3, 44.2, 46.8, 55.2 kg./mm.<sup>2</sup> The results strengthen the conclusion that there is, properly speaking, no true elastic limit for steel even at room temp., apparent absence of change in dimensions being due only to imperfect means of measurement.

C. A. SILBERRAD.

**Resistance of chromium steel to alkali solutions.** A. KRÜGER (Chem.-Ztg., 1931, 55, 335).—Cr steel, although attacked by hot, dil. acid solutions, is quite resistant to boiling solutions of NaOH. No trace of Fe could be detected in the solution after treatment under various conditions. Vessels of Cr steel should therefore prove useful in quantitative analysis involving the use of alkaline solutions. The material is also resistant to  $\text{H}_2\text{O}_2$  and  $\text{Na}_2\text{S}$  solutions.

**Manganese-free zirconium-treated steels.** F. M. BECKET (Min. and Met., 1931, 12, 234—236).—Steels with a high content of S and P and with < 0.1% Mn cannot be rolled without cracking, but if 0.4% Zr is added to the ladle just before casting, the resulting ingots work well and have a higher ductility but lower yield point and ultimate strength than Mn steels. It is suggested that addition of Zr to steels with a moderate Mn content and relatively low in P and S would increase the ductility and confer good deep-drawing properties on the sheet.

A. R. POWELL.

**Influence of occluded gases on the mechanical properties of metals.** L. GUILLET and J. COURNOT (Compt. rend., 1931, 192, 787—789).—The conclusions arrived at by Guichard etc. (B., 1931, 592) are questioned in view of the results of Hugues (B., 1928, 159), Bogitch (B., 1928, 126), and others.

C. A. SILBERRAD.



**Outdoor corrosion of zinc and effect of rainfall and atmospheric pollution.** W. S. PATTERSON (J.S.C.I., 1931, 50, 120—123 τ).—The deposit formed on Zn during outdoor corrosion in an urban atm. does not offer complete protection to the metal. There is appreciable evidence, however, to show that the deposit has some retarding effect on the corrosion, probably by preventing the direct access of acid rain-water to the metal. In the winter period, when the air of London contains appreciable amounts of SO<sub>2</sub>, the curves for rainfall and corrosion are divergent; in the summer period, when atm. pollution is low, the curves are parallel. The velocity of the outdoor corrosion of Zn is largely governed by the character of the corrosion products and the manner in which the solubility of these in rain-water is affected by atm. pollution.

**Initial corrosion rates of metals.** R. H. BROWN, B. E. ROETHELI, and H. O. FORREST (Ind. Eng. Chem., 1931, 23, 350—352).—The initial rates of corrosion in oxygenated water have finite values and have been measured by rotating an oxide-free metal cylinder in oxygenated water and determining the decrease of O concentration during the test. The initial rates of corrosion of the metals examined decrease in the following order: Al, Zn, Fe, Cu, Ni, Sn, Ag. In the case of the first three metals the corrosion rate appears to depend on the rate of diffusion of O to the specimen, but with the remaining metals the rate of oxidation of the metal is the controlling factor. The initial corrosion rates of all the metals decrease after a relatively short time, indicating the formation of partly or completely protective films in all cases. Metals such as Ni, Sn, Cu, and Ag do not seriously deteriorate on exposure to oxygenated water, not because of a negative corrosion tendency, but because of the formation of an impermeable corrosion product which prevents further deterioration. The electromotive series is not considered a proper criterion for judging the probability of corrosion of a metal in oxygenated water, and the free energy decreases, assuming O and H<sub>2</sub>O to react with the metals to form hydroxides in saturated solution are more exact measures of the tendencies to corrode.

E. S. HEDGES.

**Method of testing the local-element theory of corrosion.** W. GUERTLER and B. BLUMENTHAL (Z. Metallk., 1931, 23, 118—119).—According to the theory of Palmaer and others the rate of dissolution of Zn in dil. acids should be proportional to the concentration of the acid. An apparatus is illustrated for alternately immersing Zn strips in 0.1N- and 0.2N-HCl and measuring the vol. of gas evolved; results obtained in this apparatus show that the rate of dissolution of Zn in 0.2N-HCl is only 1.5—1.8 times as great as in 0.1N-HCl. The bearing of these results on Palmaer's theory (cf. B., 1929, 921) is discussed briefly.

A. R. POWELL.

**Attack of insects on metals.** II. O. BAUER and O. VOLLENBRUCK (Z. Metallk., 1931, 23, 117; cf. B., 1930, 1071).—Specimens of Zn roofing perforated by beetles of the *Cerambycides* family or possibly by wood-wasps of the *Sirex* family are illustrated.

A. R. POWELL.

**Alloys resistant to heat and sulphur.** H. GRUBER (Z. Metallk., 1931, 23, 151—157).—The behaviour of 29 Fe, Co, and Ni alloys containing varying quantities of one or more of the metals Cr, Mn, Al, Mo, and W has been investigated in an atm. of H<sub>2</sub>S at 700—1000°. The rate of corrosion by the gas depends on the fusibility of the sulphides formed, but in alloys containing Al a resistant S film is formed which is generally highly impervious to H<sub>2</sub>S. An alloy of 61% Ni, 15% Cr, 20% Fe, and 4% Mn to which 10% of Al has been added is permanent in atm. containing S compounds and has sufficient strength and good casting properties for use in the manufacture of furnace parts and annealing boxes. Addition of 10% Al to pure Ni reduces the rate of absorption of S at 1000° to  $\frac{1}{3.5}$ th that of pure Ni and the resulting sulphide layer is fairly adherent; with 15% Al even better resistance is obtained. Similar results are obtained with Co-Al alloys. Although Mo itself is the most resistant of the metals mentioned above to the action of S at 1000°, addition of Mo to alloys of Fe, Co, Ni, and Cr tends to reduce their resistance to S at high temp.

A. R. POWELL.

**Röntgenographic investigation of the ageing of duralumin at room temperature.** J. HENGSTENBERG and G. WASSERMANN (Z. Metallk., 1931, 23, 114—117).—During the ageing of duralumin at room temp. slight broadening of the interference lines in the Debye-Scherrer diagrams takes place and their intensity increases, whilst the intensity of the diffused radiation diminishes slightly. These facts are interpreted as indicating that, during ageing, an enrichment in Cu atoms occurs in small regions of the Al lattice without the simultaneous separation of a new crystal phase.

A. R. POWELL.

**Chemical and physical phenomena in autogenous welding.** L. KNEZ (Arch. Hemiju, 1931, 5, 15—25).—A theoretical treatment of the chemical and physical processes which occur during welding of Fe and Cu with the C<sub>2</sub>H<sub>2</sub> flame. The effects of impurities in the gas, such as PH<sub>3</sub> and H<sub>2</sub>S, are summarised. H. F. GILLBE.

**Theory of addition agents [in electroplating].** S. KANEKO (J. Soc. Chem. Ind., Japan, 1931, 34, 103B).—The effect of addition agents on electroplating baths is ascribed to (1) reduction of surface tension of the solution, favouring the formation of a larger number of nuclei; and (2) positive adsorption of the addition agent, producing a smaller grain size. E. S. HEDGES.

**Buffer effect in nickel-plating baths.** J. BARBAUDY and A. PETIT (Compt. rend., 1931, 192, 834—837).—Titration curves and, therefrom, Van Slyke's (cf. A., 1922, i, 893) buffer value,  $\beta = dB/dp_H$ , have been determined at 20° and 50° for (1) a rapid Ni-plating bath (hot) of composition (per litre) NiSO<sub>4</sub>·7H<sub>2</sub>O 450 g., NiCl<sub>2</sub> 12 g., and H<sub>3</sub>BO<sub>3</sub> 22 g.; (2) the same bath modified by use and additions of acid and NiCO<sub>3</sub>; and (3) bath (1) without H<sub>3</sub>BO<sub>3</sub>. The buffer effect of the H<sub>3</sub>BO<sub>3</sub> in maintaining the  $p_H$  below that of the precipitation of Ni(OH)<sub>2</sub> is clearly shown, and also the greater similarity *inter se* of the three  $\beta/p_H$  curves at the higher temp. Curves relating, at 20—80°, the working  $p_H$  to the  $p_H$  of H<sub>2</sub>O at the same temp. show an almost constant relative acidity for (1) and (2), but a

rapid increase with temp. for (3). This is attributed to the unbuffered hydrolysis of  $\text{NiSO}_4$ . C. A. SILBERRAD.

**Protection of alloys and steel against atmospheric attack.** E. DE WINTWARTER (Bull. Soc. chim. Belg., 1931, 40, 129—143).—A lecture on Ni-, Co-, and Cr-plating. R. CUTHILL.

**Electrodeposition of chromium from ammonium chromioxalate.** A. MAZZUCHELLI (Atti R. Accad. Lincei, 1930, [vi], 12, 587—588).—A satisfactory deposit of Cr may be obtained on a brass cathode by electrolysis with a high c.d. (25—150 amp./dm.<sup>2</sup>) a conc. solution of  $\text{NH}_4$  chromioxalate. The current efficiency is very low, but rises somewhat with increase of c.d.; the coherence of the deposit decreases under the same conditions. F. G. TRYHORN.

**Hardness and hydrogen content of electrolytic metals.** GUICHARD, CLAUSMANN, BILLON, and LANTHONY (Compt. rend., 1931, 192, 1096—1098; cf. B., 1931, 592, 680).—Five samples of electrolytic Fe gave the following figures for hardness and H content (in c.c. per g. of Fe): 365, 2.1; 338, 1.7; 265, 1.5; 247, 8.3; 202, 3.0. A similar absence of parallelism between hardness and H content is observed in unannealed Ni and Co. The error in the determination of H in the authors' previous results cannot exceed 0.1 c.c. C. A. SILBERRAD.

**Refractories.**—See VIII. **Protection of Fe.**—See XI. **Cd and foodstuffs.**—See XIX.

See also A., June, 676, **Pb-Bi and Sn-Bi alloys.** **Al bronze.** **Al-Ti, Cu-Sn, and W-Re alloys.** 691, **Electrochemistry of corrosion.** 693, **Ferrite from rusting of iron.** 701, **Test for Ag in minerals.** 702, **Analysis of Cu-Ag and Cu-Zr alloys.** **Determination of Cu in presence of Pt.**

#### PATENTS.

**Production of malleable-iron castings.** L. H. MARSHALL, Assr. to A. A. GRUBB (U.S.P. 1,781,119, 11.11.30. Appl., 27.4.28).—White-iron castings are annealed above 360° in closed containers which are packed with a mixture of refractory material and a compound, e.g.,  $\text{FeCl}_3$ , which will evolve a non-oxidising and etching atm. when heated. C. A. KING.

**Forming [ferrous] alloys.** P. A. E. ARMSTRONG (U.S.P. 1,781,490, 11.11.30. Appl., 8.5.28).—Ferrous alloys are formed into articles, e.g., valves and tappets, by melting the constituents of the alloy by means of an electric arc in a mould crucible made of a metal of relatively low m.p., but of high conductivity. Thus an Fe electrode is inserted into the base of a Cu crucible the bottom of which is of the desired shape. The metal alloy and slag constituents are packed round the crucible walls and a second electrode down the central open space causes an arc between the two electrodes. As metal melts, the slag rises and forms a protective layer on the crucible walls, which cool it. The electrodes themselves may form part of the alloying metals. C. A. KING.

**Heating metallic and other electrically conducting bodies.** S. E. KUSNETZOV (B.P. 347,163, 17.10.29).—Two articles or groups of articles embedded in carbon-

aceous material mixed with, if desired, fireclay, asbestos, etc., are directly connected with the respective supply leads from a source of current. J. S. G. THOMAS.

**[Salt-bath] heat-treatment of metals.** A. E. BELLIS (B.P. 347,238, 16.12.29. U.S., 14.12.28).—Fusion of the salt-bath materials is initiated by combustion of an Fe compound, e.g., thermit, on the surface of the bath, and a low-voltage current is passed between an electrode immersed in the bath and the metal wall of the container. J. S. G. THOMAS.

**Carbonising of metal surfaces [used in electron-discharge devices].** WESTINGHOUSE LAMP Co., Assees. of C. V. IREDEL (B.P. 347,267, 24.1.30. U.S., 25.1.29).—The oxidised surface of the metal, e.g., Ni, is exposed at 800—1000° to a hydrocarbon gas, e.g.,  $\text{C}_2\text{H}_2$ , containing a vaporised liquid hydrocarbon, e.g., light petroleum or kerosene, and is degassed by heating *in vacuo*. J. S. G. THOMAS.

**Hg-vapour generators.**—See I. **Cleaning metals.**—See VII. **Blast-furnace slag.**—See IX. **Treatment of surfaces.** **Coating compound.**—See XIII.

#### XI.—ELECTROTECHNICS.

**Protection of iron in aerated saline solutions: an Evans pile.** E. HERZOG and G. CHAUDRON (Compt. rend., 1931, 192, 837—839).—The pile, depending on differential aeration (cf. Evans, B., 1923, 983A), consists of two precisely similar Fe plates. The cathode, horizontal, is immersed in NaOH solution and is aerated by a current of air impinging on it; the anode is vertical and below the cathode in a solution of  $\text{FeCl}_2$  or  $\text{FeSO}_4$ , the two solutions being separated by a horizontal diaphragm. The p.d. increases in a few hr. from 0.15 to 0.43 volt, then becoming steady. Time-potential curves show the results of adding NaCl without (giving a p.d. of 0.4 volt), or with, simultaneous addition of an acetate, citrate, or phosphate (to illustrate their buffer effects). Citrates, oxalates, tartrates, phosphates, and arsenates all sooner or later cause the formation of a film reducing the p.d. to about 0.02 volt. With sea-water the p.d. is steady at 0.15 volt, a deposit of MgO forming. C. A. SILBERRAD.

**Positive electrode with gas circulation for air depolarisation.** C. FÉRY (Compt. rend., 1931, 192, 881).—To assist depolarisation by atm.  $\text{O}_2$  where the polarising gases (e.g.,  $\text{H}_2$  or  $\text{NH}_3$ ) are lighter than air, the positive electrode is made hollow and air brought to the bottom of the cavity, whilst the polarising gases escape by a vent at the top. C. A. SILBERRAD.

**Efficient accumulator not liable to sulphation.** C. FÉRY and REYNAUD-BONIN (Compt. rend., 1931, 192, 1035—1037; cf. B., 1925, 44).—To prevent access of  $\text{O}_2$  to the negative plates these are each surrounded by a protecting medium, permeable to the ions but not to the gases, and covered by a cap 15—20 mm. high, which completely prevents access of air from above. These caps at the end of charging are full of  $\text{H}_2$ , production of which continues slowly through the action of the electrolyte on the spongy Pb. Actual comparison of precisely similar commercial-type, 30AH-type batteries, the one thus protected, the other not, shows the very marked

superiority of the protected type, and confirms the authors' views on the cause of sulphation (cf. A., 1919, ii, 263).

C. A. SILBERRAD.

**Continuous kilns.**—See VIII. Electroplating. Ni-plating baths. Protection of alloys etc. Cr. Hardness etc. of electrolytic metals.—See X. Photoelectric control in waterworks' practice.—See XXIII.

See also A., June, 686, Conductivity of liquids. Potential of high-resistance cells. 702, Determination of Cu in presence of Pt. 703, Self-regulating induction furnace.

#### PATENTS.

**[Gas-filled] photoelectric cells.** N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 347,544, 5.7.30. Holl., 24.7.29).—A grid raised to a suitable potential is arranged within the photoelectric cell at a distance from the photo-active electrode which is equal to or less than the free path of an electron in the gas-filling.

J. S. G. THOMAS.

**Cathodes of electron-discharge tubes or thermionic valves.** N.V. PHILIPS' GLOEILAMPENFABR. (B.P. 347,500, 16.5.30. Holl., 9.10.29).—A highly refractory metal core, e.g., of W, covered, at least partly, with a layer of metal having a lower m.p., e.g., Ni, is helically wound with metal wire, e.g., Ni, at least partly covered with material of high electron-emitting capacity, e.g., an alkaline-earth metal or oxide.

J. S. G. THOMAS.

**[Indirectly heated] cathodes of vacuum-electric tube devices.** E. Y. ROBINSON, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 347,311, 31.1.30).—A filamentary heater, e.g., of W, is, prior to assembly within an insulating sheath, e.g., of SiO<sub>2</sub> or porcelain, coated with an adherent protective coating consisting of refractory metallic oxide or other non-injurious refractory insulating material.

J. S. G. THOMAS.

**[Partition walls for] electrical accumulators.** H. WEHRLIN (B.P. 347,012, 1.5.30. Ger., 29.5.29).—The edges of the walls (composed of glass wool, asbestos, or cellulose) are united by substances, e.g., asphalt, artificial resins, or rubber, which are not attacked by the electrolyte.

H. ROYAL-DAWSON.

**Precipitation electrode for electric gas purification.** SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 347,501, 16.5.30. Ger., 18.5.29).—The electrode, made in the form of a box with slot-like openings in its sides serving as precipitation surfaces, has the parts lying behind the trap-pockets and protected from the flow of gas entirely or partly cut out, and vertical or oblique stays are arranged in the inner trap chamber to prevent cross-currents.

J. S. G. THOMAS.

**Mercury-vapour rectifiers.** A.-G. BROWN, BOVERI & Co. (B.P. 349,411 and 349,413, [A] 14.10.30, [B] 23.10.30. Ger., [A] 5.12.29, [B] 19.4.30).

**[Switching means for the heating device in] luminous electric-discharge tubes.** GEN. ELECTRIC Co., LTD., Assees. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 347,559, 18.8.30. Ger., 19.10.29. Addn. to B.P. 316,611; B., 1930, 775).

**Electric-discharge devices for producing ultra-violet rays [in foodstuffs etc.].** F. REYNOLDS (B.P. 349,294, 20.5.30).

**Treatment of hydrocarbons.**—See II. Forming alloys. Heating of metals. Carbonising metal surfaces.—See X.

#### XII.—FATS; OILS; WAXES.

**Influence of the so-called isooleic acid on fat hardening from the viewpoint of the phase rule.** W. KOCZY and F. GRIENGL (Monatsh., 1931, 57, 253—290).—The m.p.—composition relationships are studied for the possible binary systems comprising isooleic (prepared by Schmidt's method; A., 1890, 863), palmitic, oleic, linoleic, and stearic acids, and for the ternary systems comprising stearic and the above unsaturated acids, both liquidus and solidus values being determined. The system formed by an American cottonseed oil and a technical hydrogenated oil derived from it is also investigated. As the result of a study of "pseudo-binary" mixtures, chosen to represent the possible products obtainable in the course of hydrogenation of the free acids in such an oil, it is concluded that: (1) during the hydrogenation of a mixture of 40.5% of oleic and 59.5% of linoleic acids the formation of isooleic acid is at first without influence, but beyond a concentration of about 7% has a marked effect in increasing the m.p.; (2) the presence of a const. amount (1.8%) of stearic acid considerably reduces the hardening effect of the isooleic acid; (3) an increase in the content of stearic acid to 6.6%, as the result of hydrogenation, has a much greater hardening effect than has isooleic acid alone; and (4) the presence of a const. proportion (20.9%) of palmitic acid prevents any increase in m.p. as the result of hydrogenation. The last effect is also observed in the system formed by linoleic and oleic with 1.8% of stearic and 20.9% of palmitic acids.

H. A. PIGGOTT.

**Carbohydrates affect mill values of cottonseed.** G. S. MELOY (Oil & Fat Ind., 1931, 8, 187—189).—Tables are given connecting oil, protein, and NH<sub>3</sub> contents with the proportion of meats in the whole seed: for a given % of oil in whole seed, the increase of NH<sub>3</sub> content (due to increased % of meats) coincides with increase of material other than fat or protein. The connexion between composition and growing conditions is illustrated.

E. LEWKOWITSCH.

**Splitting of castor oil.** H. M. LANGTON (J.S.C.I., 1931, 50, 213—214 T).—After pointing out the unique character of castor oil, which is attributable to its containing upwards of 80% of the triglyceride of ricinoleic acid, the author enumerates the results of saponifying "firsts" castor oil in an autoclave using 2.5% CaO as the splitting base under a steam pressure of 120 lb./in.<sup>2</sup> for 6 hr. The course of the reaction was followed by hourly withdrawal of samples which were tested for free fatty acids. The const. of the oil are given and the results are exhibited both numerically and graphically, and are compared with those of Jones, who operated at 200 lb./in.<sup>2</sup> (J.S.C.I., 1917, 36, 359). The author's results lie along a curve of the exponential type, and are thus in accord with his published records of

the splitting of other fatty oils. The average split obtained was apparently 75.2%, but a glycerin determination on the final product showed that the split was almost complete. The reduction in Ac value from 135.6 to 115.4 is evidence that some polymerisation has taken place during the splitting, but not to such a great extent as in the anomalous results obtained by Jones. The lower pressure used in the present case is given as the reason for absence of abnormality in the results.

**Composition of commercial palm oils. II. Fatty acids of some palm oils of high free acidity.** T. P. HILDITCH and (Miss) E. E. JONES (J.S.C.I., 1931, 50, 171—176 t).—Four commercial palm oils of high free acidity from various sources have been investigated with regard to the quantitative composition of the fatty acids present in (a) the crude oils and (b) the neutral portions of the oils; the results have been discussed in conjunction with those for four oils of low free acidity (cf. B., 1930, 956). Six of the eight crude oils were closely similar in composition, namely, myristic (1.2—5.9%), palmitic (37.5—43.0%), stearic (2.2—5.9%), oleic (40.2—45.2%), and linoleic (6.5—11.2%) acids; the remaining two, both from Drewin (Gold Coast) but of widely different free acidity, had myristic (2.2—2.3%), palmitic (34.3—35.3%), stearic (5.2—5.6%), oleic (49.5—52.3%), and linoleic (5.5—8.3%) acids. Broadly speaking, increase in unsaturated acids is compensated for by diminution in palmitic acid, and *vice versa*, the proportions of myristic and stearic acids being small and somewhat variable. Plantation oils, in addition to very low free acidity, tend towards const. fatty acid composition, probably in consequence of standardisation of the variety of oil palm cultivated. Except in one case (Niger), there was little difference between the component fatty acids of the crude oils and of the neutral portions prepared by alkali-refining the crude oils; it would appear that hydrolytic changes during rancidity affect the different fatty acids indiscriminately and non-selectively. The neutralised Niger oil was about 4% lower in palmitic, and higher in oleic, acid than the crude oil. The acetyl values of the neutralised oils indicated relatively little di- or mono-glycerides and correspondingly extensive hydrolysis to glycerol and fatty acids. On the other hand, the plantation palm oils of lowest free acidity had acetyl values of 11—12, indicating the presence of some mono- or di-glycerides in the fresh fat as matured in the palm fruit; the amount may correspond to 4—8% of the oil, expressed as diglycerides.

**Laboratory bleaching technique for fatty oils.** J. T. R. ANDREWS and R. G. FOLZENLOGEN (Oil & Fat Ind., 1931, 8, 183—185).—For laboratory bleaching tests the use of 3% of English earth and 1% of H<sub>2</sub>O (at 135° for 5 min. or 105° for 15 min.) gives results only slightly inferior to those by the official A.O.C.S. method (6% of earth) and comparable with plant practice; moisture control is essential when comparing bleaching-earth efficiencies. E. LEWKOWITSCH.

**Oils for chronometers.**—See II. **Litharge-linseed oil.**—See XIII. **Oleum ricini.**—See XX.

See also A., June, 679, **Soap solutions.** 691, **Oxidation of fats by air.** 711, **Hydrogenation of fatty**

**acid esters.** 712, **Hydrogenation of linolenic acid esters.** 773, **Autoxidation of fats.**

## PATENTS.

**Margarine and other manufactured fatty-predominating substances.** J. L. KERR (B.P. 346,276, 3.1.30).—The melted mixed fats are sprayed upwards into a cooling chamber, from which the solidified granules are subsequently collected and amalgamated.

E. LEWKOWITSCH.

**Production of saturated fatty acids.** H. P. KAUFMANN (B.P. 345,626, 24.12.29. Ger., 27.12.28).—Catalytic hydrogenation (preferably with Ni) is conducted simultaneously with hydrolysis of the fat by any usual method (*e.g.*, in the presence of MgO, with alkali). A working pressure of about 10 atm. is cited. The relative extent of hydrolysis and hydrogenation is controlled by the amounts of the catalysts used, the temp. and duration of the treatment, and the H pressure. [Stat. ref.]

E. LEWKOWITSCH.

**Manufacture of sulphonation products of fats, fatty oils, fatty acids, or aliphatic hydrocarbons of high mol. wt.** I. G. FARBENIND. A.-G. (B.P. 346,945, 17.3.30. Ger., 15.3.29).—The fats or oils (C<sub>8</sub> or over), especially mineral lubricating fractions (saturated or unsaturated compounds, with or without OH groups), are dissolved in liquid SO<sub>2</sub> and treated with SO<sub>3</sub>, oleum, or chlorosulphonic acid at about —12° to —15°. After evaporating the solvent at 0° the product is worked up in the usual manner. E. LEWKOWITSCH.

**Manufacture of water-soluble condensation products of unsaturated fatty acids.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 346,425, 4.1.30).—Higher (poly-)unsaturated fatty acids (*e.g.*, wood oil fatty acids) are treated with sulphonic acid derivatives of aromatic (polynuclear) hydroxy-compounds, *e.g.*,  $\alpha$ - or  $\beta$ -naphtholsulphonic acids, in the presence of dil. H<sub>2</sub>SO<sub>4</sub> at 120—140°. The neutralised products are H<sub>2</sub>O-sol. and stable to mineral acids and alkali, but can be salted out by NaCl. E. LEWKOWITSCH.

**Manufacture of soft soap.** K. HENTSCHEL (B.P. 346,389, 3.1.30. Ger., 29.1.29).—Coconut oil, palm-kernel oil, etc., or their mixtures with other fats (not over 35%), are saponified with KOH containing not more than 2.4% of constituents such as chlorides etc. with salting-out properties; the amount of H<sub>2</sub>O is adjusted to give a yield of 200—300% of the transparent stable product. E. LEWKOWITSCH.

**[Paste] soaps and cleansers [for paint etc.].** F. J. LOVELACE (B.P. 347,433, 29.3.30).—Na silicate or glycerin, borax solution, light mineral oil containing petroleum jelly, powdered pumice, benzene, and perfume are added successively to a NaOH-tallow-vegetable oil soap. E. LEWKOWITSCH.

**Apparatus for treating oleaginous material.** J. DAVIDSON, Assr. to DAVIDSON-KENNEDY Co. (U.S.P. 1,782,714, 25.11.30. Appl., 15.4.29).—Plant for cooking oleaginous materials with direct superheated steam at 206—372° is detailed: an automatically controlled measuring kettle, a cooker, and receiver are superimposed concentrically. E. LEWKOWITSCH.

**Manufacture of waxes.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 346,438, 10.1.30).—Natural or artificial waxes (*e.g.*, montan or shellac wax) are heated with alkylene oxides (*e.g.*,  $C_2H_4O$ ) at, *e.g.*, 120–170° in a closed vessel. Hydrocarbon waxes, and/or organic acids such as phthalic acid or anhydride, resinic acids, etc., may be included if desired. The products, which resemble beeswax, Japan wax, etc., are readily emulsifiable in alkaline solutions.

E. LEWKOWITSCH.

**Lubricating compositions.**—See II. **Polymerised alkylene oxides. Detergents. Basic products.**—See III. **Drying agents for paints.**—See XIII.

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

**Viscous and elastic flow of paints.** H. WOLFF (Kolloid-Z., 1931, 55, 81–85).—A “turboviscosimeter” for use with very viscous liquids is described. For liquids showing viscous flow (glycerol, mineral oil, sucrose solutions) the product of the time of fall of the wt. and the load is const., but for the paints examined the product decreases with increasing load. The ratio of the product under a small load to the product under a heavy load gives a measure of the plasticity. E. S. HEDGES.

**Reaction between litharge and linseed oil at room temperature.** K. CHARISIUS and E. KINDSCHER (Farben-Ztg., 1931, 36, 1297–1300).—A mixture of litharge (3 pts.) and linseed oil (2 pts.) was stored for 7 weeks, in the absence of air, with vigorous daily shaking. The colour of the supernatant oil gradually changed to a dark red-brown and the vol. of sediment increased until, after 5 weeks, no sedimentation occurred during 24 hr. After storage it was found by progressive extractions that 29% of the litharge had reacted with the oil, comprising oil-sol. Pb, and  $Et_2O$ - and  $C_6H_6$ - $EtOH$ -sol. organic Pb compounds containing 46 and 50.4% Pb, respectively. The quantity of fatty acids isolated from the Pb compounds (greatly in excess of the fatty acid content of the original oil) indicates that definite splitting of the oil must have occurred. Analyses of the raw materials and of the various products isolated are given. S. S. WOOLF.

**Application of low-temperature tar in the production of phenol-formaldehyde resins.** G. T. MORGAN and N. J. L. MEGSON (J.S.C.I., 1931, 50, 191–193 r).—Hardening resins have been prepared from crude phenolic fractions of a typical low-temp. tar and purified sufficiently for use in electrical tests. These resin preps. when tested either on single impregnated sheets or in laminated boards have breakdown voltages at least equal to those of trade resins and of resins made under comparable laboratory conditions from pure phenol. Soluble non-hardening resins of Novolak type accompany hardening resins in  $CH_2O$  condensations with crude phenols.

**Machinery for nitrocellulose lacquer manufacture.** F. ZIMMER (Farben-Ztg., 1931, 36, 1342–1344).—Centrifuges used for clarification of clear nitrocellulose lacquers and refining of pigmented lacquers, and of suitable grinding mills for the latter, are briefly described. S. S. WOOLF.

**Nitrocellulose.**—See V. **Ti film-forming materials.**—See VII.

### PATENTS.

**Coating compound.** S. I. CHARLESWORTH and A. W. HARRIS (U.S.P. 1,782,341, 18.11.30. Appl., 4.2.27).—The molten resinous residue from the distillation up to 300° *in vacuo* of the “pure still residue” remaining after the distillation of the light oils recovered from by-product coke-oven gas is mixed with approx. 30 wt.-% of benzol etc., forming a coating composition for nails etc. S. S. WOOLF.

**Compositions of cellulose compounds containing softeners of the acetal type.** IMPERIAL CHEM. INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & Co. (B.P. 318,980, 13.9.29).—Compositions of cellulose derivatives, *e.g.*, cellulose acetate, and, as softener therefor, an acetal of relatively low v.p. having outside the acetal group more than one ether group, *e.g.*, an acetal of an alkyl ether of diethylene glycol, together with a solvent mixture are claimed. S. S. WOOLF.

[Coating composition for] **treatment of surfaces.** BRIT. CELANESE, LTD. (B.P. 346,269, 4.12.29. U.S., 4.12.28).—An ester or ether of cellulose is precipitated from a solution in a mixed solvent, the higher-boiling constituents, *e.g.*, amyl alcohol, “cellosolve,”  $PhMe$ ,  $H_2O$  being non-solvents for the cellulose derivative, and the lower-boiling constituents, *e.g.*,  $COMe_2$ ,  $AcOEt$ ,  $CHCl_3$ , being solvents or “latent” solvents therefor. Clouded or opaque coatings are formed. S. S. WOOLF.

**Coating compositions.** E. I. DU PONT DE NEMOURS & Co. (B.P. 319,590, 23.9.29. U.S., 22.9.28).—Styrene is polymerised by heating in the presence of a catalyst, *e.g.*,  $Bz_2O_2$ , and an inert solvent, *e.g.*, aromatic hydrocarbons, at below 140° (*e.g.*, 5 hr. at 115–120°). The resulting vinyl polymeride is used as the base of lacquers free from “cobwebbing” tendency when sprayed. S. S. WOOLF.

**Preventing spontaneous combustion [of spray dust from coating compositions].** E. C. PITMAN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,782,126, 18.11.30. Appl., 13.7.28).—The deposits of spray dust containing oxidisable matter, *e.g.*, linseed oil, are treated with anti-oxidants, *e.g.*, quinol, tannic acid. S. S. WOOLF.

**Production of drying agents with a high metal content for use in paints.** GEBRÜDER BORCHERS A.-G. (B.P. 346,812, 3.1.30. Ger., 23.1.29).—Metals are substituted in the OH and  $CO_2H$  groups of mono- or polybasic hydroxy-fatty acids or their glyceryl esters (*e.g.*, ricinoleic acid, glycollic acid, castor oil) by reaction with metal oxides, preferably in the presence of saturated or unsaturated non-hydroxylated fatty acids or fatty oils. Such products are sol. in turpentine, oils, etc. E. LEWKOWITSCH.

**Manufacture of litharge.** E. KNAPP and C. G. ALLGRUNN, Assrs. to NIAGARA SPRAYER Co. (U.S.P. 1,779,003, 21.10.30. Appl., 5.5.25).—Air is forced upwards through molten Pb maintained at approx. 550°, and the  $PbO$  floating to the surface is skimmed off continuously and conveyed to a finishing furnace, where it is kept in const. agitation in contact with air for 3–4 hr. at approx. 600°. S. S. WOOLF.

**[White] pigment.** E. C. HOLTON, Assr. to SHERWIN-WILLIAMS Co. (U.S.P. 1,778,975, 21.10.30. Appl., 19.10.27).—White pigments comprising a Zn-Pb fume pigment and a smaller proportion of a Ti-O compound and substantially free from non-opaque extenders, *e.g.*, BaSO<sub>4</sub>, are claimed. Such pigments have high opacity and show little tendency to "chalk." S. S. WOOLF.

**Metal-oxide pigments.** GOODLASS WALL & LEAD INDUSTRIES, LTD., and N. J. READ (B.P. 347,129, 18.1.30).—A metal salt, inert towards paint vehicles and capable of combining with S compounds to form a white sulphide and liberate an inert white oxide, *e.g.*, Zn sesquitanate or orthosilicate, is incorporated as colour-preserving agent for white pigments, particularly Sb oxide, containing the oxide of a metal forming a coloured sulphide. S. S. WOOLF.

**Manufacture of colour lakes from petroleum.** G. ALLEMAN, Assr. to SUN OIL Co. (U.S.P. 1,781,772, 18.11.30. Appl., 24.8.26).—The H<sub>2</sub>O-sol. soaps in the soda-sludge from petroleum refining are separated in an oil-free condition (resins may also be removed, if desired) and precipitated with an aq. solution of a sol. salt of a suitable metal, *e.g.*, CaCl<sub>2</sub>, in the presence of a H<sub>2</sub>O-sol. dye. The lakes produced are insol. in H<sub>2</sub>O, but sol. in AcOEt, petroleum spirit, etc., and may be used in paints, wall-paper inks, etc. E. LEWKOWITSCH.

**Manufacture of colour lakes.** J. Y. JOHNSON From I. G. FARBENIND. A.-G. (B.P. 346,263, 7.10.29).—Basic dyes which may contain acid groups are precipitated by solutions of salts of complex inorganic acids, *e.g.*, phospho-molybdates or tungstates, the  $p_H$  values of which (whilst definitely above 7) have been predetermined by the addition of basic substances, *e.g.*, caustic alkalis, carbonates, acetates, to solutions of the complex acids, or their acid salts, or by other suitable means. Strong acids, *e.g.*, HCl, chloroacetic acid, may, if desired, be added before or after the precipitation. S. S. WOOLF.

**Printing processes.** H. WADE. From AMER. MULTIGRAPH Co. (B.P. 345,263, 17.12.29).—The surface of a printing plate prepared with an image is treated with a substance, *e.g.*, metal or NH<sub>4</sub> nitrates and/or sulphates, which will render the plate initially ink-repellent on the non-image portions thereof without injuring the image. Printing is then carried out using an ink which will adhere to the image only and which has been treated with a similar substance to the above and will continue the surface-treatment, maintaining the non-image portions ink-repellent. If a Zn plate be used, suitable material, *e.g.*, NH<sub>4</sub> phosphate, is added to wash and ink, forming a protective film on the plate. S. S. WOOLF.

**Manufacture of lacquers, varnishes, or the like.** BAKELITE GES.M.B.H. (B.P. 347,611, 28.1.30. Ger., 29.1.29. Addn. to B.P. 295,335; B., 1930, 469).—The "A" resins ("resols") of the prior patent are converted into "B" resins ("resitols") by heating in suitable solvents, *e.g.*, hydrogenated phenols, with or without hardening agents, the H<sub>2</sub>O of reaction being removed by distillation or desiccation. Org. non-solvents, *e.g.*, Tetralin or PhBr, may be added. S. S. WOOLF.

**Production of rosin oil.** J. N. BORGLIN, Assr. to HERCULES POWDER Co. (U.S.P. 1,782,401, 25.11.30.

Appl., 2.1.29. Renewed 29.3.30).—Wood rosin is heated at 215–250° in the presence of 0.05–1.0% of a B compound, *e.g.*, H<sub>3</sub>BO<sub>3</sub>, and then destructively distilled under reduced pressure. S. S. WOOLF.

**Removing colour from rosin.** W. B. LOGAN, Assr. to NEWPORT Co. (U.S.P. 1,782,267, 18.11.30. Appl., 2.8.28).—A solution of gum or wood rosin in a light petroleum solvent (petroleum naphtha) is treated with a metal halide, *e.g.*, SnCl<sub>4</sub>, capable of forming an insol. compound with the colour substances in the rosin, and the ppt. thus formed is removed by filtration etc. The metal halide remaining in the solution is removed therefrom, *e.g.*, by precipitation with H<sub>2</sub>O. S. S. WOOLF.

**Mixed esters of colophony etc.** H. A. BRUSON, Assr. to RESINOUS PRODUCTS & CHEM. Co. (U.S.P. 1,783,165, 25.11.30. Appl., 8.10.28).—Glycerol (etc.) is esterified with a mixture of resin acid and a dibasic aliphatic acid between C<sub>6</sub> and C<sub>10</sub>, *e.g.*, colophony (30 pts.), sebacic acid (15 pts.), and glycerin (5 pts.) are heated at 195–260° to give a C<sub>6</sub>H<sub>6</sub>-sol. mixed ester gum which remains flexible at –5°. C. HOLLINS.

**Manufacture of synthetic resins.** BRIT. THOMSON-HOUSTON Co., LTD., H. W. H. WARREN, R. NEWBOUND, and A. T. WARD (B.P. 346,333, 29.11.29).—Resins of the polyhydric alcohol-polybasic acid type, *e.g.*, glycerol-phthalate, are acylated by treatment in a fusible state with an aliphatic or aromatic acyl chloride or acid anhydride, *e.g.*, BzCl, Ac<sub>2</sub>O, excess of the latter substances being subsequently removed by distillation under normal or reduced pressure. Plasticisers and natural or other synthetic resins may, if desired, be incorporated. The time taken to polymerise the resin is reduced and the product is free from cavities or blisters. S. S. WOOLF.

**Synthetic resin varnishes and synthetic resins.** IMPERIAL CHEM. INDUSTRIES, LTD., A. A. DRUMMOND, and H. H. MORGAN (B.P. 345,276 and 345,310, 13.9.29).—(A) CH<sub>2</sub>O and *m*-xylenol (or its isomerides) or coal-tar distillates (b.p. 216–223°) are heated under normal or increased pressure with or without an acid catalyst, in the absence of alkali, and, if desired, in the presence of excess of a solvent or diluent, *e.g.*, industrial methylated spirit, which is distilled off after the initial reaction is complete. The temp. is then allowed to rise to 120° and maintained thereat until a suitable resinous product is obtained on cooling. This is sol. in a wide range of solvents and may be heated at 170° for 1–2 hr. with a drying oil and converted into varnish by the addition of driers, thinners, etc. (B) Homogeneous synthetic resin products derived from phenols, CH<sub>2</sub>O, and fatty acid esters (> 20% of the final product; cf. B.P. 329,313 [B., 1930, 780]), sol. in alcohol, ketone, or ester solvents but substantially insol. in hydrocarbon solvents, are heated at 110–160°, preferably with continuous removal of solvent vapours liberated, until gelation is imminent (1–2 hr.). The product is then insol. in the original solvents, but sol. in hydrocarbon solvents, *e.g.*, heavy naphtha and, for compositions containing large proportions of oils, white spirit, giving air-drying varnishes with the addition, if desired, of driers. S. S. WOOLF.

**Production of urea-formaldehyde condensation products.** E. G. BUDD MANUFG. CO., Assees. of G. L. KELLEY and M. W. DEISLEY (B.P. 319,687, 11.9.29. U.S., 26.9.28).—A mixture of urea,  $\text{CH}_2\text{O}$ , and  $\text{NH}_3$  solution, of  $p_H$  5.2—7.0 is heated under reflux for a short time; thiourea is added to increase the  $\text{H}_2\text{O}$ -repellent properties of the final product. The gelation of the resulting homogeneous fluid is retarded by addition of a salt having a strong basic ion and a weak acid ion, e.g.,  $\text{AcONa}$ , and  $\text{H}_2\text{O}$  is removed, an acid or acid-yielding substance, e.g., formic acid,  $\text{KH}_2\text{PO}_4$ , or  $\text{AlCl}_3$ , being added during removal of  $\text{H}_2\text{O}$  and after the removal of free  $\text{CH}_2\text{O}$ . Heat is applied, if desired, to free the product from bubbles. [Stat. ref.] S. S. WOOLF.

**Phenolic resinous products and compositions containing them.** BAKELITE CORP., Assees. of V. H. TURKINGTON and W. H. BUTLER (B.P. 347,737, 28.1.30. U.S., 29.1.29).—Hydroxydiphenyls are condensed with methylene-containing substances, e.g.,  $\text{CH}_2\text{O}$ , in the presence, if desired, of fatty oils, e.g., tung oil, giving oil-sol. resins. S. S. WOOLF.

**Fibrous resinous compositions.** BAKELITE CORP., Assees. of G. E. WIGHTMAN (B.P. 345,332, 17.12.29. U.S., 18.12.28).—Mouldable compositions comprising wood fibres containing approx. 90% (preferably 94% or more) of  $\alpha$ -cellulose, and a potentially reactive resinoid binder, e.g., a phenol-fatty oil-methylene reaction product, are claimed. S. S. WOOLF.

**Manufacture of [resinous] condensation products.** KUNSTHARZFABR. DR. F. POLLAK GES.M.B.H. (B.P. 347,228, 24.1.30. Austr., 21.12.29).—Thiodicyanodiamidine ready-formed, or prepared *in situ* by acting on cyanamide or dicyanodiamide with  $\text{H}_2\text{S}$ , is condensed with  $\text{CH}_2\text{O}$  in alkaline solution. The product is neutralised or acidified and further heated, and, if desired, a urea- $\text{CH}_2\text{O}$  or other condensation product is incorporated. The solution is then thickened *in vacuo*, and mixed with fillers etc. S. S. WOOLF.

**[Synthetic resin-]bonded materials.** BAKELITE CORP., Assees. of V. H. TURKINGTON (B.P. 345,228, 8.11.29. U.S., 10.11.28).—A resinoid binder to be incorporated with a filler, e.g., a woven fabric, and subsequently set by heat without the aid of a polymerising catalyst, has imparted to it a reticulated structure by dispersing in it, before setting, a material, e.g.,  $\text{H}_2\text{O}$ , that is liquid and insol. in the binder or gaseous at the temp. of setting, the volatilisation of this material being controlled so that a small proportion, e.g., 2—6%, is left in the bonded material when set. A hygroscopic substance, e.g.,  $\text{CaCl}_2$  or glycerin, may be added to the  $\text{H}_2\text{O}$  to retard volatilisation until the binder sets. The reticulated structure may be imparted by incorporating with the binder a resinous material immiscible therewith, e.g., a flexible resinous product of the phenol-tung oil type. High impact strength and a minimum of swelling are claimed for the products. S. S. WOOLF.

**Resin plasticiser.** H. A. BRUSON, Assr. to RESINOUS PRODUCTS & CHEM. CO. (U.S.P. 1,783,166, 25.11.30. Appl., 5.7.29).—A condensation product of a polyhydric alcohol, e.g., glycerol, and pimelic, suberic, azelaic, or

sebacic acid is incorporated with a  $\text{PhOH-CH}_2\text{O}$  resin as plasticiser. S. S. WOOLF.

**Preparation of resin-like plastic masses.** J. BAER (B.P. 345,894, 20.6.30. Ger., 24.6.29).—To a solution of Ca (or other) polysulphide ( $d$  1.07) is added "sulphite-turpentine" or "Tallöl," and the mixture is heated to the b.p. H. ROYAL-DAWSON.

**Moulding powder.** W. H. BOORNE (B.P. 345,472, 10.3.30).—A mixture of rosin (4 pts.),  $\text{CaO}$  (1 pt.), and formalin (4 pts.) is heated in an open vessel and ground. The powdered mixture (450 pts.) is admixed with a cellulose filler or waste, e.g., wood flour (300 pts.), Portland cement (4 pts.), and heavy mineral oil (10 pts.) which has been mixed to paste with  $\text{CaCO}_3$  (5 pts.). For increased hardness and brilliance of surface, hexamethylenetetramine (< 8%) is added, and synthetic phenolic resins may be incorporated with the rosin. S. S. WOOLF.

**Mouldable compositions containing mica.** BAKELITE CORP., Assees. of C. A. NASH (B.P. 346,349, 8.1.30. U.S., 17.1.29).—Compositions of reactive phenolic resinoids (2 pts.) and a filler (more than 3 pts.), the latter containing 5% or more of finely-divided mica (passing 80-mesh), are claimed. S. S. WOOLF.

**[Liquid] adhesives.** BRIT. CELANESE, LTD. (B.P. 347,445, 1.4.30. U.S., 2.4.29).—A liquid adhesive containing a cellulose derivative, e.g., cellulose acetate, a synthetic resin, e.g., a  $\text{PhOH-CH}_2\text{O}$  condensation product, suitable solvents, and, if desired, a plasticiser, is applied to surfaces to be united. When this coating has become tacky, a finely-divided powder comprising a cellulose derivative, e.g., the acetate, and a plasticiser, e.g., ethyl phthalate, is sprinkled on the surface and the articles are pressed together under increased temp. S. S. WOOLF.

**Manufacture of inlaid linoleum or the like.** ARMSTRONG CORK CO., Assees. of J. A. PHELAN (B.P. 348,892, 21.5.30. U.S., 22.6.29).

**Nitrocellulose compositions.**—See V. Colouring cellulose esters etc.—See VI. Cementing of surfaces.—See IX. Soaps for paints.—See XII. Nitro-starch.—See XXII.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

See A., June 683, Degradation of rubber solutions.

##### PATENTS.

**Manufacture of ribbon, sheet, etc. from aqueous dispersions of organic substances, particularly rubber latex.** J. T. SHEVLIN. From SOC. ITAL. PIRELLI, and U. PESTALOZZA (B.P. 347,691, 31.1.30).—Heat-stabilised dispersions are forced through a space of the required thickness formed between two plate-like elements heated to the coagulation temp. Fibrous material such as woven or cord fabric may be impregnated by passage together with the dispersion between the plates. D. F. TWISS.

**Treatment of rubber.** NAUGATUCK CHEM. CO., Assees. of S. M. CADWELL (B.P. 347,955, 23.6.30. U.S., 2.7.29).—The mixture of aliphatic bases of the empirical composition  $\text{C}_{2n+2}\text{H}_{5n+8}\text{N}_{n+2}$  obtainable by the

interaction of an ethylene dihalide and  $\text{NH}_3$ , is used as an accelerator of vulcanisation. The dihalide of a homologue of ethylene, e.g., a propylene dihalide, may be used as an alternative in the primary reaction.

D. F. TWISS.

**Treatment [prevention of scorching] of rubber.** S. M. CADWELL, ASSR. to NAUGATUCK CHEM. CO. (U.S.P. 1,777,874, 7.10.30. Appl., 2.3.28).—Scorching is prevented by the use of acid (chloroacetic, sulphanilic, oxalic, trichloroacetic acids,  $\text{H}_3\text{PO}_4$ , toluene-*p*-sulphonyl chloride, pine tar) in conjunction with a dithiocarbamate derivative hydrolysable by alkali, e.g., 2:4-dinitrophenyl dimethylthiocarbamate, triphenylmethyl ethyl xanthate, 4-chloro-2:6-dinitrophenyl dithiobenzoate, 2:4:6-trinitrophenyl 2-benzthiazyl sulphide, benzylidene dimethylthiocarbamate, etc.

C. HOLLINS.

**Vulcanisation of rubber and accelerator therefor.**

D. H. POWERS, ASSR. to E. I. DU PONT de NEMOURS & Co. (U.S.P. 1,780,149, 28.10.30. Appl., 26.1.26).—Products obtained by the action of an aliphatic aldehyde, particularly  $\text{CH}_2\text{O}$ , on the condensation product of an aliphatic amine and an aliphatic aldehyde, such as  $\text{CH}_2\text{O}$ -ethylamine or heptaldehyde-methylamine, are capable of so aiding vulcanisation that this will occur at  $120^\circ$ ; below  $100^\circ$  their effect is only slight, so that they are free from scorching tendencies.

D. F. TWISS.

**Vulcanisation accelerators.**—See III. **Rubber-coated fabrics.**—See VI. **Road-building material.** **Floor coverings.**—See IX.

## XV.—LEATHER; GLUE.

**Vegetable tanning materials of Erythrea.** G. A. BRAVO (Annali Chim. Appl., 1931, 21, 141—146).—The leaves of *Osyris Abissinica* (Hochst) or Erythrean sumac contain 18.43% of tans of the pyrocatechol group and 16.72% of sol. non-tans; the pods of *Acacia nilotica* (Del) 35.25% of tans; and the pods with (without) seeds of *Cesalpinia tinctoria* (Dombey, Benth) tans 45.62 (51.88—55.75), sol. non-tans 20.45 (20.45—22.38), insol. matter 24.85 (14.02—14.12), and  $\text{H}_2\text{O}$  9.08 (9.68)%. Leather tanned with the last of these is very light in colour, resembling that obtained with sumac, and when mordanted with Fe (Ti) assumes a bright black (red) colour.

T. H. POPE.

**Biochemistry of tan-liquor fermentation.** P. D. DALVI (J. Indian Inst. Sci., 1930, 13A, 173—192).—Microflora from tan liquors of avaram bark (*Cassia auriculata*), divi-divi (*Cesalpinia coriaria*), and myrobalans (*Terminalia chebula*) were isolated and their physiological characteristics studied. Of the moulds, *Aspergillus niger*, *Penicillium glaucum*, and an unknown *Penicillium* developed strongly even in presence of 10% of tannic acid. Tannase was prepared from *A. niger* grown in an extract of myrobalan powder together with Freudenberg's medium, and purified by precipitation with EtOH and dialysis. The preparation, which was protein-free, hydrolysed Me gallate, the hydrolysis being hastened by addition of  $\text{KH}_2\text{PO}_4$ . The optimum temp. for the enzyme is  $60^\circ$ . Hydrolysis of tannic to gallic acid by tannase is accelerated by presence of  $\text{K}_2\text{HPO}_4$  and by decrease in concentration of substrate and is inhibited by very acid reactions. In checking either the growth

of *A. niger*, *A. luchuensis*, and *P. glaucum* or the loss of tannic acid,  $\text{CS}_2$ ,  $\text{CH}_2\text{O}$ , and paraffin are ineffective, whilst thymol and camphor are highly efficient.

F. O. HOWITT.

See also A., June, 752, **Determination of tannic and gallic acids.** 777, **Tannin of barley husk.**

## PATENTS.

**Leather-stretching test.**—See I. **Albumose-like products.**—See III. **Articles having pearl effect.**—See V.

## XVI.—AGRICULTURE.

**Soil structure.** M. KRAUSE (Landw. Jahrb., 1931, 73, 603—690).—A comprehensive description and discussion of Russian investigations in connexion with pore space,  $\text{H}_2\text{O}$  relationships, and the physical condition of soils and their effects on crop production.

A. G. POLLARD.

**Elutriation, water capacity, and compression tests in the characterisation of soils.** VON NOSTITZ (Landw. Jahrb., 1931, 73, 407—429).—In a discussion of soil survey results, mechanical analyses are shown to be valuable in soil classification.  $\text{H}_2\text{O}$ -capacity measurements are of little value for this purpose. Compression tests with Puchner's apparatus were made with soil blocks prepared by kneading the sieved soil (2 mm.) with just sufficient moisture to form a compact mass. The blocks were dried at room temp. for 24 hr. and then at  $50^\circ$  until of constant wt. The crushing pressure of the blocks increased with the clay content of the soils. Freezing did not appreciably affect the crushing strength of surface soils, but markedly increased that of subsoils.

A. G. POLLARD.

**Study of the flocculation of a sodium clay soil with the objects of reclaiming sodium clay soils and the amelioration of gault, Kimmeridge and Oxford clays.** E. M. TAYLOR and R. M. WOODMAN (J.S.C.I., 1931, 50, 203—206 T).—The flocculating concentrations of solutions of  $\text{CaSO}_4$  and  $\text{Ca}(\text{OH})_2$  for a Na clay soil have been determined. When flocculation of a suspension of this soil has once been obtained by solutions of these substances, replacement of the liquid medium of the suspension by water, followed by reshaking, is accompanied by reflocculation, the number of such reflocculations depending on the concentration of the original flocculating solution. It is also demonstrated that flocculation can be maintained so long as any water in contact with the sediment contains a small percentage—much less than would flocculate the original soil—of these substances. The bearing of these two findings on the reclamation of "black alkali" soils is discussed.

**Effect of water on the reaction of clay soils.** G. ROTHE (Arb. biol. Reichsanstalt Land- u. Forstwirts., 1931, 18, 425—429).—Liming practice is discussed in relation to fruit tree culture, with special reference to blue clay soils. Fruit trees may be grown successfully in acid soils of good permeability. The compact nature of acid blue clays is unfavourable to root development, but carefully regulated liming is essential. An alkaline reaction in these soils involves the coagulation of colloidal Fe and Al compounds, but other colloids may be dispersed and washed out by  $\text{H}_2\text{O}$ .

A. G. POLLARD.



**Soil acidification by ammonium sulphate.** R. W. BELING (Landw. Jahrb., 1931, 73, 491—501).—In soils treated with dicyandiamide to prevent nitrification, the acidifying effect of  $(\text{NH}_4)_2\text{SO}_4$  is small. The definite increase in acidity of normal soils resulting from treatment with  $\text{NH}_4$  salts results directly from the nitrification process. A. G. POLLARD.

**Soil acidification and superphosphate.** C. KRÜGEL (Superphosphate, 1931, 4, 117—124).—Experimental evidence is quoted to show that the supposed acidity produced in soil by superphosphate treatment is insignificant. A. G. POLLARD.

**Interrelationships of certain single-valued soil properties.** G. B. BODMAN and E. P. PERRY (Soil Sci., 1931, 31, 365—378).— $\text{H}_2\text{O}$ -vapour adsorption methods indicated a higher proportion of colloid in soils than that represented by the clay fraction ( $< 2 \mu$  diam.), after  $\text{H}_2\text{O}_2$ -HCl pretreatment. Of the clay fraction nearly 90% of the particles were of  $< 1 \mu$  diam. Pretreatment of soil with  $\text{NH}_3$  yielded much smaller amounts of clay with diam.  $< 1 \mu$  than did the  $\text{H}_2\text{O}_2$ -HCl treatment. Close correlation was found between the following values, arranged in descending order of correlation coeff.: moisture equiv. and sticky point, colloid and moisture equiv., colloid and sticky point, moisture equiv. and non-sticky plastic range, colloid and air-dry moisture content, colloid and clay  $< 2 \mu$ , moisture equiv. and rolling-out limit. Correlations of a lower order were obtained for clay and sticky point, clay and moisture equiv., colloid and rolling-out limit. The sticky point and rolling-out limit should be valuable in characterising field soils. In soils having a moisture equiv. of 9.8% approx., an inversion or coincidence of values for the sticky point and rolling-out limit (normally the lower) may be anticipated. A. G. POLLARD.

**Soil solution experiments of Wrangell and the views of Němec.** L. MEYER (Forts. Landw., 1930, 5, 745—748; Bied. Zentr., 1931, 60A, 70).—Determinations of the available P in soil cannot be based on a single extraction with  $\text{H}_2\text{O}$  (Němec) since the proportion of the total P thus removed varies with the adsorptive power of the soil. Moreover, more P is removed if the extraction is prolonged beyond the stipulated 30 min. A. G. POLLARD.

**Solubility of the adsorptively bound bases in soils in relation to their lime condition and degree of saturation.** II. A. GEHRING, U. CREUZBURG, E. POMMER, O. WEHRMANN, A. WOLTER, and H. VON STOCKHAUSEN (Z. Pflanz. Düng., 1931, 20A, 183—217; cf. B., 1931, 455).—The action of Mg salts on crop yields varies with the degree of saturation of the soil with Ca, being most marked where the Ca saturation is 20—25% and decreasing as the value is raised. The solubility of soil potash also depends on the degree of Ca saturation. This effect is masked in pot cultures, but is apparent in the field. Difficulties attending potash-requirement determinations by means of pot cultures are discussed. A. G. POLLARD.

**Composition of the soil solution and the growth of plants in very dilute solutions.** M. VON WRANGELL (Ergeb. Agrik.-Chem., 1930, 2, 9—20; Bied. Zentr., 1931, 60A, 81).—Interrelationships between the com-

position of soil solutions and plant growth are examined and discussed. A. G. POLLARD.

**Determinations of  $p_{\text{H}}$  in soil suspensions.** C. DREYSPRING and F. HEINRICH (Z. Pflanz. Düng., 1931, 20A, 155—169).—Details of the technique for determining  $p_{\text{H}}$  values in aq. and KCl soil suspensions are recommended. The sample (10 g. passing 2-mm.) is shaken in a hard glass tube with 10 c.c. of  $\text{H}_2\text{O}$  in a rotary apparatus (40 r.p.m.) for 15 min. Quinhydrone (50 mg.) is added to the tube and the measurements are made 3—5 min. later. A. G. POLLARD.

**Application of soil analyses.** I. J. BEHR and R. KÖHLER (Mitt. Lab. Preuss. Geol. Landesanst., 1930, No. 11, 15—34; Chem. Zentr., 1931, i, 1346).—A discussion of the results of elutriation analyses and the differentiation of soil classes and constituents. A. A. ELDRIDGE.

**Nutrient content of soils.** K. NAUMANN (Diss., T.H., Dresden; Bied. Zentr., 1931, 60A, 66—67).—The logarithmic nature of Mitscherlich's growth curves and the constancy of growth factors in respect of K for various crops and under different climatic conditions are confirmed. The effect factor for K based on the K intake of plants agreed with that calc. from the K applied, but the value was lower than that adopted by Mitscherlich. A logarithmic relationship in respect of P was not observed and the effect factors from different series of experiments showed poor agreement. The effect factor for P, calc. from the amount of P applied, was less than that indicated by the P intake of plants. In Neubauer tests the whole of the available P and K is not removed by one crop of barley. In a comparison of methods the values for K were in the order: Neubauer  $>$  Weissmann  $>$  Mitscherlich. Values for P were less divergent, except in Neubauer tests, where values were markedly low. A. G. POLLARD.

**Biochemical determination of the nutrient content of soils.** H. NIKLAS (Ergeb. Agrik.-Chem., 1930, 2, 21—34; Bied. Zentr., 1931, 60A, 67).—Christensen's *Azotobacter* method and Neubauer's seedling method for determining nutrient contents show close agreement in soils of low P content, diverged somewhat in soils of moderate P content, and were reasonably concordant in rich soils. In very heavy soils the *Azotobacter* test was less satisfactory. A method described for determining the assimilable P and K in soil by means of *Aspergillus* gave more const. results for K than for P. A. G. POLLARD.

**The *Aspergillus* method [of determining soil nutrients].** H. SÖDING (Z. Pflanz. Düng., 1931, 20A, 129—130).—The method of Niklas and co-workers (B., 1931, 37) is criticised. The preparation of the samples for examination and the  $p_{\text{H}}$  changes involved are discussed. A. G. POLLARD.

**Determination of easily soluble nutrients in soils by electro-ultrafiltration.** R. DIEHL (Ernähr. Pflanze, 1930, 26, 453—456; Bied. Zentr., 1931, 60A, 124).—Electro-ultrafiltration methods offer a satisfactory means of determining the proportion of sol. nutrients in soils and also yield information as to the rate at which nutrients become sol. The "easily-sol." portion of the soil reserves may thus be differentiated. A. G. POLLARD.

**Photosensitised oxidation of ammonia and ammonium salts and the problem of nitrification in soils.** G. G. RAO and N. R. DHAR (Soil Sci., 1931, 31, 379—384).—Photochemical oxidation of  $\text{NH}_3$  to nitrate occurs in the presence of sensitizers. The activity of sensitizers examined was in the order  $\text{TiO}_2 > \text{ZnO}, \text{CdO} > \text{Na}_2\text{U}_2\text{O}_7 > \text{Al}_2\text{O}_3 > \text{SiO}_2$ . Alkalinity favours oxidation and *vice versa*. A. G. POLLARD.

**Determination of the assimilable nitrogen in soils by means of grass seedlings.** R. WAGNER (Wiss. Arch. Landw., 1930, 5A, 166; Bied. Zentr., 1931, 60A, 80—81).—Western ryegrass may be used for determining the available N content of soils under conditions similar to those of Neubauer tests. Utilisation of nitrates and  $\text{NH}_4$  salts by ryegrass is dependent on the reaction of the medium. Fresh, moist soil samples must be used, since drying under any conditions leads to changes in the N content of the soil. Results do not agree with those obtained by König's method.

A. G. POLLARD.

**Soils. VIII. Assimilable potassium. IX. Calcium and the soil.** J. CLARENS and H. MARGULIS (Bull. Soc. chim., 1931, [iv], 49, 380—384, 384—389; cf. B., 1930, 294).—VIII. It is shown that solubilisation of K in soils occurs in definite stages, and an attempt is made to correlate these with the requirements of the plant during the stages of its growth. The total alkali metals were determined as chlorides in the usual way in extracts of 4 samples of the soil in solutions containing suitably increasing quantities of acid (*e.g.*, for a soil containing 3%  $\text{CaCO}_3$ , 0.25, 0.5, 0.75, and 1.0 c.c. of 0.1N-HCl per g. respectively).

IX. On the basis of previous work,  $\text{CaCO}_3$  in a soil poor in humus may be determined from the change in level of a Hg manometer attached to a closed vessel containing 5 g. of soil with an excess of dil.  $\text{HNO}_3$ . Details are also given for the determinations of acidic functions actually saturated by so-called displaceable bases, and free acidic functions. The quantity of  $\text{CaCO}_3$  necessary for recalcification is found by agitation of the soil with saturated  $\text{CaSO}_4$  solution, or with an equiv. solution of  $\text{CaCl}_2$ . Solid  $\text{CaCO}_3$  recalcifies only very slowly on account of its insolubility (cf. B., 1930, 474, 629); examples are quoted. Acidic functions are liberated and appreciable quantities of bases displaced by treatment with HCl in concentrations less than that required for saturation of the  $\text{CaCO}_3$ , the whole of the bases being displaced by excess of acid. J. GRANT.

**Determination of the lime requirement of soils on the basis of their hydrolytic acidity.** J. VON CSIKY (Z. Pflanz. Düng., 1931, 20A, 142—155).—Addition to soils of the CaO required to neutralise hydrolytic acidity as calc. by Woltersdorf (B., 1930, 734) does not remove acidity completely. Relationships between the author's lime-water method for determining CaO requirement (B., 1930, 256); the limiting values of Vageler and Woltersdorf (B., 1930, 295); the (*T*—*S*) value, of Hissink; and the presence of free  $\text{CaCO}_3$  in soils, are discussed. A. G. POLLARD.

**Phosphate requirement of soils in relation to soil type.** E. LAUTERBERG (Z. Pflanz. Düng., 1931,

20A, 302—356).—Discrepancies in the results of soil-nutrient determinations by various methods may depend, in part, on the physical composition of the soil, and such results should be considered in conjunction with mechanical analyses. The phosphate content of soils is largely concentrated in the finer particles, there being practically none in particles of  $>0.05$  mm. diam. The adsorptive power of soils for phosphate varies with the nature of the phosphate applied. Soils containing much assimilable phosphate (Neubauer) have a low adsorptive power and *vice versa*. A. G. POLLARD.

**Soil examination and the use of artificial fertilisers.** H. VAGELER (Z. Pflanz. Düng., 1931, 10B, 238—253).—Field-trial results are utilised in a general discussion of the rational use of fertilisers.

A. G. POLLARD.

**Effect of artificial nitrogenous fertilisers on the occurrence and development of *Azotobacter chroococcum* in soils.** E. SCHNEIDER (Arch. Pflanz. enb., 1931, 5, 304—373; Bied. Zentr., 1931, 60A, 70).—Application to soil of nitrogenous fertilisers, especially  $\text{NaN}_3$  and, to a smaller extent, of urea, favours the growth of *A. chroococcum*.  $(\text{NH}_4)_2\text{SO}_4$  is not effective until nitrified. Continuous dressings of  $(\text{NH}_4)_2\text{SO}_4$  reduce the development of *Azotobacter*. A. G. POLLARD.

**Effect of increasing applications of nitrogen on pure and mixed herbage plants of meadows and pastures.** J. VASTERS with T. REMY (Landw. Jahrb., 1931, 73, 521—602).—The growth of grasses, clovers, and weeds was affected to different proportional extents by increasing applications of nitrogenous fertilisers. No relationships were apparent between the effects on plants of similar families. The yield and chemical composition of hay was mainly the result of the different botanical composition of the herbage brought about by fertiliser treatment. Nitrogenous fertilisers increased the crude protein content of all non-legumes, but simultaneously depressed the yield of legumes. The protein content of the mixed herbage depended on the balance of these two effects and, in general, increased with the heavier applications of fertiliser. The proportion of root to top growth increased with fertilisation, but the N content of the root was lower than that of the green matter. A. G. POLLARD.

**Effect of potash on the stiffness of cereal straws.** F. BERKNER and W. SCHLIMM (Landw. Jahrb., 1931, 73, 503—520).—Applications of potash fertilisers to soils had no consistent effects on the potash content, diam., and tensile strength of oat and barley straws grown in them. No relationship between the  $\text{SiO}_2$  content and bending strength of straw was observed. Applications of potash fertilisers reduced the  $\text{SiO}_2$  content of straw. CaO applied with small dressings of potash fertilisers increased the bending strength of straw, but phosphatic fertilisers had no very definite effect in this respect. In most cases potash manures increased the length and thickness of the vascular bundles and thickened the sclerenchymatous tissue. Kainit produced a considerable increase in the number of vascular bundles.

A. G. POLLARD.

**Hay and silage.** E. BROUWER (Vereen. Exploit. Proofzuivel. Hoorn, 1929, 1—34; Chem. Zentr., 1931,

i, 1378).—Losses are recorded and feeding experiments described. A. A. ELDRIDGE.

**Characteristics of various methods of utilising peat in manure making in the Schenkursk district of the northern area.** A. A. KUPRIANOV and N. S. ROSANOV (Arb. Zentr. Torfstat., 1930, 2, 41—60; Bied. Zentr., 1931, 60A, 74).—Where peat is trampled in cattle pens to a depth of about 1 m. the exclusion of air from the lower layers has little effect on the properties of the final product since only the surface layer undergoes change. Where aeration of the peat is maintained, the penetration of dung and urine is more thorough and the product has a lower  $H_2O$ -holding capacity. The best peat manure is obtained by filling successive layers of mossy peat into the pens. Good-quality manure is produced by composting moor soil with dung from the stalls. Peat manuring increases the bacterial numbers of soils to a greater extent than does straw manuring. A. G. POLLARD.

**Conservation and availability of the nitrogen in farmyard manure.** A. F. HECK (Soil Sci., 1931, 31, 335—363).—During the storage of farmyard manure under anaerobic conditions  $H_2O$ -sol. N compounds are transformed into  $NH_3$ , which combines with the org. acids produced by acid fermentation. Losses of N from manure during handling and storage may amount to 50% of the total, or the whole of the liquid-manure N, and are due to the volatilisation of free  $NH_3$  in the early drying period and of  $NH_4$  salts in the later stages. Changes in  $p_H$  during acid fermentation are directly proportional to the amount of cellulosic (energy-producing) material present. When liquid manure is stored its N compounds are steadily transformed into  $(NH_4)_2CO_3$ , which may be lost by volatilisation. Covering the liquid with oil prevents this loss even in open cisterns. The use of straw litter in manure reduces N losses during drying, but also reduces its availability. Available N in manure may be expressed as the total  $H_2O$ -sol. N less the loss during handling and storage. A. G. POLLARD.

**Nitrogen losses and action of cold- and hot-fermented manures.** D. MEYER (Z. Pflanz. Düng., 1931, 10B, 253).—A correction (cf. B., 1931, 558).

A. G. POLLARD.

**Manuring of meadows.** EICHINGER (Superphosphat, 1931, 7, 83—91).—The proportion of mineral nutrients applied to soil which are recovered in successive cuttings of meadow herbage is examined. No appreciable loss of nutrient is incurred by applying the whole dressing of phosphates and potash in the spring. Nitrogenous fertilisers are better applied in two portions, one in spring and the other following the first cutting. The value of applications of farmyard manure and of  $CaO$  are discussed. Superphosphate is superior to basic slag on low-moor soils. A. G. POLLARD.

**Effect of phosphate manuring on the yield and quality of malting barley.** C. DREYSPRING, H. KURTH, and F. HEINRICH (Z. Pflanz. Düng., 1931, 10B, 217—237).—In field trials with numerous varieties of barley, applications of superphosphate increased the grain : straw ratio and the starch content of the grain, decreased the protein content of the grain, and accel-

ated ripening. The extent of these effects varied considerably with the variety examined.

A. G. POLLARD.

**Increased yield of starch from potatoes by phosphate manuring.** OSKIERSKI (Superphosphat, 1931, 7, 102—103).—Numerous field trials are recorded in which applications of superphosphate increased the yield and starch content of potatoes and, more consistently, the yield of starch per acre. A. G. POLLARD.

**Analogy between the course of growth and nutrient intake of oats and physico-chemical laws.** H. WAGNER (Landw. Jahrb., 1931, 73, 453—490).—Pot cultures of barley in which varying depths of soil, but the same nutrients, were used showed that the dry matter yields increased with the depth of soil, gradually approaching a maximum value. The percentage nutrient content of all plants remained practically identical. The ratio of nutrients ( $K_2O : P_2O_5 : N = 1$ ) in the green matter of the plants widened as the flowering stage was approached. Assimilation of potash, phosphate, and N by the plant increase the rate of production of organic matter; Ca had but little effect. Mathematical expression of growth processes must be based on an exponential junction. The S-form of growth curve observed, e.g., in the K intake, results from a temporary limitation of the growth process. Various mathematical treatments of growth curves are examined.

A. G. POLLARD.

**Premature heading of cauliflower as associated with the chemical composition of the plant.** W. R. ROBBINS, G. T. NIGHTINGALE, and L. G. SCHERMERHORN (New Jersey Agric. Exp. Sta. Bull., 1931, No. 509, 14 pp.).—Premature "heading" of cauliflower was produced in sand cultures by restricting the supply of N in the nutrient. In comparison with vigorous plants those with "button" heads had high carbohydrate content and low N contents of which the proportion present as protein was abnormally high.

A. G. POLLARD.

**Biology of the potato. F. MERKENSCHLAGER. VI. Ecological deterioration of the potato.** H. WARTENBERG (Arb. biol. Reichsanstalt Land- u. Forstwirts., 1931, 18, 405—423).—Symptoms typical of ecological deterioration in potatoes (leaf curl, mosaic, etc.) are associated with a high sol. salt concentration in the soil and in particular with heavy potash manuring. Affected plants frequently have high ash contents and a high potash content in the ash. The bearing of these results on the ultimate cause of deterioration is discussed.

A. G. POLLARD.

**Protecting stored grain from insects by the use of oils.** W. P. FLINT (J. Econ. Entom., 1931, 24, 425—427).—Successful results obtained by the use of standard and proprietary oil emulsions are described. A thin film of oil over the surface of the grain prevents the entry of newly hatched larvæ.

A. G. POLLARD.

**Lethal power of certain insecticides against the European corn borer.** F. L. SIMANTON, F. F. DICKE, and G. T. BOTTGER (J. Econ. Entom., 1931, 24, 395—404).—Trials with arsenates, pyrethrum, nicotine, fluosilicates and numerous other F compounds are described. F compounds proved the most satisfactory,

although Na and Ba fluosilicates caused serious injury to the grain. The use of carriers, adhesives, spreaders, activators, etc. did not appreciably improve the insecticides. A. G. POLLARD.

**Summer oil [insecticide] for the control of European elm scale (*Gossyparia almi*, L.).** C. R. CLEVELAND (J. Econ. Entom., 1931, 24, 349—355).—Successful trials with 2% white-oil emulsions are described. No foliage injury was observed. A. G. POLLARD.

**Spray-residue removal.** D. F. FISHER (J. Econ. Entom., 1931, 24, 526—531).—A discussion of recent developments. A. G. POLLARD.

**Eradication of prickly-pear by chemicals, with particular reference to emulsions in the systems gelatin-aqueous arsenic acid.** R. M. WOODMAN and W. J. WILEY (J.S.C.I., 1931, 50, 187—189 T).—It is argued that when spraying for the eradication of prickly-pear, emulsions containing aq.  $H_3AsO_4$  solutions and the cresols as liquid phases with gelatin or glue as the emulsifier, should preferably be of the aq.  $H_3AsO_4$ -cresols type. The  $H_3AsO_4$  present is demonstrated to favour this desirable type, any tendency to formation of the opposite type possessed in the absence of this acid being thus suppressed.

**Cottonseed.**—See XII. **Sugar beet.**—See XVII. **Sewage sludge as manure. Control of the house fly.**—See XXIII.

See also A., June, 699, **Glass electrode for  $p_H$  determinations.** The "pehameter." 705, **Kjeldahl N distillation.** 708, **Air in low-moor soils. Hungarian lowland soils. Soil structure in N. Germany.** 769, **Nitrifying bacteria. N cycle in forest soils. Decomposition of carbamide in soil.** 774, **Petroleum oils and the respiration of bean leaves. Permeability to iodine of vegetable plants.**

#### PATENTS.

**Manufacture of mixed fertilisers.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 346,991, 14.4.30. Addn. to B.P. 326,529; B., 1930, 475).—Similar results to those claimed in the prior patent are obtained by adding up to 6% of  $H_2O$ -sol. Mg salts to the liquid mixture containing  $NH_4NO_3$ . H. ROYAL-DAWSON.

**Non-hygroscopic seed disinfectant.** M. ENGELMANN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,776,423, 23.9.30. Appl., 6.2.28).—A mercuriated phenol (*o*-chloro- or *o*-nitro-phenol) or acid etc. (benzoic anhydride) is mixed with an alkaline-earth oxide or hydroxide and an alkali salt which can react with the latter to give an insol. salt (Na oxalate, phosphate, sulphate, fluoride). C. HOLLINS.

**Weed-destroying compositions.** G. LITSCHÉ (B.P. 344,701, 22.4.30).—Cryst.  $CuSO_4$  (1 pt., powdered) is made into a paste with  $H_2SO_4$  (*d* 1.70, 3 pts.). H. ROYAL-DAWSON.

**Sol. cellulose esters.**—See V.

### XVII.—SUGARS; STARCHES; GUMS.

**Sampling, analysis, and composition of sugar beet.** G. MILNE, H. T. JONES, and J. S. WILLCOX (J.S.C.I., 1931, 50, 155—160 T).—Experiments were made to

investigate the accuracy of various methods for the sampling and analysis of sugar beet, and the effect of such factors as size, exposure of the upper part of the root above the soil, clamping, and fanginess on the composition of beet. The following conclusions are reached. The standard error calculated from 212 duplicate determinations of sugar in beet by the Pellet hot-water process is  $\pm 0.136$ . Except in the case of small crops, a sample of sugar beet to be representative should contain at least 50 roots. The use of a boring tool for obtaining material for pulping from a field sample is unreliable. The juice-and-factor method leads to higher results than those obtained by the pulp method. The possible causes are discussed. "Bolters" are significantly lower in sugar and in dry matter contents than are normal roots. "Crowns" constituting about 13% of the entire root are much inferior in sugar and richer in N, fibre, and ash than is the rest of the root. Small beet are definitely richer in sugar and dry matter than larger beet grown under identical conditions. Evidence is adduced to show that the factory method of sampling a bulk is inaccurate. Greening of the upper part of the root due to exposure above the soil does not materially affect the composition. Development of fangs on beet is not accompanied by a significant alteration in the sugar content.

**Rapid determination of sugar in exhausted [beet] slices.** V. STANEK and P. PAVLAS (Z. Zuckerind. Czechoslov., 1930, 55, 175—182).—By passing 0.5—0.75 kg. of the slices through a modified hand fruit-press ("Cosmos"), rejecting the first 50 c.c. of juice, and polarising the next 100 c.c. after clarification with 0.75 g. of a well-ground mixture of 1 pt. of fine CaO with 25 pts. of neutral  $Pb(OAc)_2$  (cheap substitute for dry basic Pb acetates), results are obtained in good agreement with those by official methods and in a much shorter time, viz., 9 min. J. H. LANE.

(A) **Conductometric formula for determining ash in both raw and refinery syrups and molasses.**  
(B) **Ash and electrical conductivity of refined cane sugars.** F. W. ZERBAN and L. SATTLER (Ind. Eng. Chem. [Anal.], 1931, 3, 38—40, 41—43. Cf. B., 1928, 422; 1930, 387, 925).—(A) The formula proposed is: ash (%) =  $0.0191369K - 0.002249K_2 - 0.001210K_3 + 3.07$ , in which  $K$  is the sp. conductivity of a solution containing 0.5 g. of the sample plus 4.5 g. of pure sucrose per 100 c.c., and  $K_2$  and  $K_3$  are those of similar solutions treated with 5 c.c. of 0.25*N*-KOH and 5 c.c. of *N*- $H_3PO_4$  respectively, per 200 c.c. With 133 samples, including one of beet molasses, of ash contents 4.84—14.23%, this formula gave a max. deviation from the chemical ash (sulphated minus  $H_2O$ ) of 0.3 and an average deviation of 0.134. A nomograph for simplifying calculations is illustrated.

(B) On the basis of 34 samples tested, from 4 refineries, it is concluded that for granulated and remelt cane sugars containing up to 0.3% of ash, in solutions of 25 g./100 c.c., the sp. conductivity, corr. for that of the  $H_2O$  and multiplied by the  $C$ -value 530, agrees satisfactorily with the chemical ash content. With soft sugars (41 samples containing 0.18—2.31% of ash were tested, from 3 refineries) at a concentration of

5 g. (or 2.5 g. plus 2.5 g. of sucrose) per 100 c.c. it is necessary to use different *C*-values for different refineries or districts, or the following general formula may be used: ash (%) =  $0.001695(0.913K + 193.5 - 0.1K_1)$ , in which *K* is the sp. conductivity of a solution of 5 g. of the sample in 100 c.c., and *K*<sub>1</sub> that of a similar solution treated with 5 c.c. of 0.25*N*-HCl per 200 c.c. The authors conclude with some practical recommendations for equipment and methods to be used in routine work on all types of cane products. J. H. LANE.

**Effect of amino-acids on rotation of glucose [dextrose] and fructose [lævulose] and its significance to determination of sucrose by double-polarisation methods.** D. T. ENGLIS and F. A. DYKINS (Ind. Eng. Chem. [Anal.], 1931, 3, 17—21).—The combination between dextrose and amino-acids in alkaline solutions (A., 1926, 151, 714, 1061) may occur to some extent in solutions of sugar products clarified with basic Pb acetate, and affect the determination of sucrose. Mixtures of equal vols. of 0.0666*M*-dextrose and glycine solutions buffered at *p*<sub>H</sub> 8.11, 9.52, and 10.3 lost, respectively, 2.77, 16.66, and 22.22% of their rotatory power in 48 hr. at 25°, and their iodometric values decreased to about the same extent. With asparagine in place of glycine similar slow changes were observed, depending on the alkalinity, but the falls in rotation and iodometric value did not correspond. The changes produced in solutions of low alkalinity are very slowly reversed on acidification, but at higher alkalinities the reversibility becomes incomplete. At very low temp. the interaction of dextrose and glycine is much restricted, whilst at 50° it is relatively rapid and non-reversible and marked discoloration occurs. Lævulose showed no optical change in presence of glycine and asparagine with increasing *p*<sub>H</sub> values, which could not be attributed to the alkalinity alone. J. H. LANE.

**Determination of glucose [dextrose] in presence of fructose [lævulose] and glycine by the iodometric method.** F. A. DYKINS and D. T. ENGLIS (Ind. Eng. Chem. [Anal.], 1931, 3, 21—23).—In the iodometric determination of dextrose in presence of amino-acids the oxidation of the sugar is too rapid for any appreciable condensation with the amino-acid to occur (cf. preceding abstract). Low results from this cause are likely only if the sugar solution has remained alkaline for some time before the analysis. Amino-acids and lævulose both tend to produce positive errors, which are greatest when the alkalinity is highest and due to NaOH alone. Of the various alkaline agents employed with I, phosphate buffer is preferred. The authors add 10 c.c. of 25% Na<sub>2</sub>HPO<sub>4</sub>·12H<sub>2</sub>O and 5 c.c. of 0.5*N*-NaOH to a mixture of 25 c.c. of sugar solution and 25 c.c. of 0.1*N*-I, and leave in a stoppered bottle for 1 hr. before acidifying. J. H. LANE.

**Measurement of consistency of starch solutions.** J. C. RIPERTON (Ind. Eng. Chem. [Anal.], 1931, 3, 152—154).—To measure the swelling of starch without rupturing the granules, nearly boiling H<sub>2</sub>O is allowed to drop gently on to a suspension of the starch grains in cold H<sub>2</sub>O. The degree of swelling cannot be taken as a direct measure of the viscosity, but the viscosity is

proportional to the swelling power multiplied by the concentration, and a given starch may therefore be evaluated without the use of a viscosimeter. The method is particularly applicable to tuber starches having a high swelling power, such as those of potato, sago, and arrowroot. E. S. HEDGES.

**Starch in cereals. Fodder for milk goats.**—See XIX.

See also A., June, 691, Oxidation of carbohydrates by air. 703, Sodium light for refractometry. 776, Carbohydrate in root of *Arctium lappa*. New sources of inulin. Sorbitol in Norwegian rowan berries. 699, Glass electrode for *p*<sub>H</sub> determinations.

#### PATENTS.

Cellulosic product.—See V. Yeast.—See XVIII.

### XVIII.—FERMENTATION INDUSTRIES.

**Proteolytic enzymes of malt preparations.** K. A. TISSUE and C. H. BAILEY (Cereal Chem., 1931, 8, 217—226).—Removal of the proteolytic enzymes from diastatic malt extracts by treatment with safranin increases their bread-improving qualities.

E. B. HUGHES.

**Fermentation of millet.** A. HEIDUSCHKA and P. DANTSCHOFF (Pharm. Zentr., 1931, 72, 337—339).—A very thick mash of finely-ground millet is boiled and cooled several times and inoculated with an infusion of ground chick-pea and air-dried embryos of millet. After keeping overnight at room temp., the diluted mash is filtered through wire gauze and allowed to ferment further. The resulting yellowish-brown beverage in which are suspended the finest particles of the millet is termed "Bosa" and contains at the end of 13 days 0.515 wt.-% of EtOH, 0.493% of lactic acid, and 0.0975% of AcOH. C. RANKEN.

**Formation, presence, and detection of βγ-butylene glycol in wine and cider.** J. PRITZKER and R. JUNGKUNZ (Ann. Falsif., 1931, 24, 139—144).—βγ-Butylene glycol is present as a by-product in alcoholic fermentation. When oxidised by Br in Na<sub>2</sub>CO<sub>3</sub> solution, βγ-butylene glycol forms diacetyl, which may be distilled off in presence of FeCl<sub>3</sub>. A sensitive test for diacetyl is to distil into a mixture of NH<sub>2</sub>OH, HCl, NaOAc, and NiCl<sub>2</sub>, when a ring of Ni dimethylglyoxime is formed. The reaction may be made quantitative, but only one third of the glycol is oxidised to diacetyl. The production of βγ-butylene glycol is considered to be characteristic of an alcoholic fermentation.

E. B. HUGHES.

**"Casse" of white wines.** R. MARCILLE (Ann. Falsif., 1931, 24, 167—168).—The presence of Fe is not the usual cause of "casse" in white wine, as is usually supposed, but it is due to H<sub>3</sub>PO<sub>4</sub> present. The ppt. is shown to consist of Ca<sub>3</sub>PO<sub>4</sub> and protein matter.

E. B. HUGHES.

**Fluorescent indicators for the determination of free acidity of red wines.** Y. VOLMAR and J. M. CLAVERA (Anal. Fis. Quím., 1931, 29, 247—254).—Comparative experiments have been made with umbelliferone and acridine and with ordinary indicators for the

determination of the free acidity of wines of which the colour obscures the end-point, the potentiometric method being used to establish the true end-point. Titration to  $p_{\text{H}}$  7 may be carried out by the use of 5–6 drops of a 0.001% alcoholic solution of umbelliferone in 100 c.c. of a solution containing 4 c.c. of the wine; the error is 0–3%. With phenolphthalein the results are high (5–30%), and with acridine low (7–15%).

H. F. GILLBE.

**Composition of Morocco wines.** L. CHAUVEAU and A. VASSEUR (Ann. Falsif., 1931, 24, 152–164).—Analyses are given. It is concluded that in 1930 26% of the samples did not conform to the usual standards. Analyses of samples taken in 1926–1929 are also discussed.

E. B. HUGHES.

**Tan liquor fermentation.** See XV.

See also A., June, 759, Urease.

#### PATENTS.

**Production of yeast.** A. BRAUN, J. and E. FISCHL (M. FISCHL'S SÖHNE), and F. ROSENBERG (B.P. 346,361, 4.12.29. Austr., 18.12.28).—Yeast is propagated in a series of fermentations directly following one another. Apart from the first phase in which seed yeast is used, the subsequent phases are seeded with the yeast crop from the preceding phase. The dilution of the molasses solution and the volume of the fermentation liquid increase with each phase, together with a corresponding increase in the amount of aeration. Each phase is supplied with sufficient nutrients and the propagation carried on until the sugar has been consumed as completely as possible. The quantities of sugar and nutrient required for the amount of yeast yielded by any phase are calc. by means of a growth factor, which is constant for the second and following phases, but slightly lower for the first phase.

C. RANKEN.

**Increasing the fermenting power of yeast.** H. WINDESHEIM and F. W. THIELE (B.P. 347,542, 2.7.30).—Cholesterol or a similar compound in the form of an emulsion with fat is added with phosphates to yeast. Brewer's yeast is stored before treatment in dil. malt extract and purified with charcoal.

C. RANKEN.

**Production of absolute alcohol from unpurified spirit.** G. B. ELLIS. From E. MERCK (B.P. 347,268, 24.1.30).—Raw spirit and an entraining liquid are distilled with or without pressure in a dehydrating column. The entraining liquid is separated from the aq. EtOH and low-boiling impurities in the azeotropic distillate and is returned to the dehydrating column with the EtOH obtained by rectification of the alcoholic layer of the distillate. The abs. alcohol and high-boiling impurities are removed from the dehydrating column and separated from each other and from traces of the entraining liquid in two further columns.

C. RANKEN.

**Rectification of alcohol.** Soc. ANON. DES DISTILLERIES DES DEUX-SÈVRES, E. RICARD, and H. M. GUINOT (B.P. 347,122, 18.11.29).—Liquid low in EtOH content is heated in a small auxiliary column so that less than 3% of the feed liquid is vaporised. Impurities insol. or only slightly sol. in  $\text{H}_2\text{O}$  are thus eliminated before the "refined wort" enters the main distillation column.

C. RANKEN.

**Treatment [concentration] of ethyl alcohol.** C. HANER, Assr. to PUBLICKER, INC. (U.S.P. 1,783,086, 25.11.30. Appl., 3.10.28).—NaOH or KOH (e.g., 0.8 lb./gal.) is added to the aq. EtOH, which is distilled; 98% or more conc. EtOH is recovered in the first stages. Distillation is discontinued at a point (90–98% of total EtOH recovered) when the still residue remains liquid when hot, but solidifies on cooling to room temp.

E. LEWKOWITSCHE.

**Dye preparation.**—See IV. Cheese.—See XIX.

#### XIX.—FOODS.

**Determination of starch in cereal products.** C. W. HERD and D. W. KENT-JONES (J.S.C.I., 1931, 50, 15–22 T).—Methods of starch determinations are classified broadly into two classes: (1) hydrolytic method; (2) non-hydrolytic method. Many of the methods in each group are examined and discussed and it is suggested that a modification of Rask's method is very suitable for cereal work. Results of application to commercial starches, commercial flours and mill stocks, wheat offals, and whole wheats are given. Reference is made to the uncertainty of the constituents of starch, and it is suggested that discrepancies in various methods are due to the inclusion or exclusion of one or other of these constituents.

**Hygroscopicity of wheat.** L. PAP (Cereal Chem., 1931, 8, 200–206).—A tabulation of the hygroscopicity of Hungarian and other foreign wheats, showing the deviations in moisture content of samples stored under the same conditions.

E. B. HUGHES.

**Fermentation period of Northwest and Pacific Northwest flours as indicated by carbon dioxide production and dough expansion.** J. L. ST. JOHN and M. HATCH (Cereal Chem., 1931, 8, 207–216).—Owing to complicating factors, the application of the Bailey-Johnson method (B., 1925, 111) is limited to determining the effect of added ingredients on fermentation.

E. B. HUGHES.

**Relation of wheat protein to baking quality. II. Saskatchewan hard red spring wheat crop of 1929.** R. K. LARMOUR (Cereal Chem., 1931, 8, 179–189; cf. B., 1930, 583).—A statistical study of results of baking tests on wheats of greatly varying protein content. The correlation factor between protein content and loaf vol. is high for the  $\text{KBrO}_3$  and blend-bromate methods, the latter giving better results with high-protein flours.

E. B. HUGHES.

**Wheat proteins in relation to peptisation and baking strength.** R. H. HARRIS (Cereal Chem., 1931, 8, 190–200).—Peptisation of wheat protein by inorg. salt solutions gives results comparable with those obtained on flour.

E. B. HUGHES.

**Mildly toasted wheat germ—a possible merchandisable table cereal.** R. HERTWIG (Cereal Chem., 1931, 8, 226–228).—Wheat germ roasted at 120–130° to golden-brown keeps well and has a pleasant flavour.

E. B. HUGHES.

**Toasted wheat germ self-rising flour—a possible merchandisable cereal.** R. HERTWIG (Cereal Chem., 1931, 8, 229–231).—Toasted wheat germ is

blended with flour, baking powder, salt, and cerelose (dextrose) for use in cakes and biscuits.

E. B. HUGHES.

**Use of oxygen in the quick determination of ash [in flour].** R. B. POTTS (Cereal Chem., 1931, 8, 232—233).

—The flour is burned in an open furnace at 427° and transferred to an airtight furnace at 593° into which O<sub>2</sub> is introduced. The total time occupied is 1½—2½ hr.

E. B. HUGHES.

**Fermentation and proofing cabinet [for baking tests] giving low temperature variability.** R. K. LARMOUR, F. D. MACHON, and S. F. BROCKINGTON (Cereal Chem., 1931, 8, 233—241).—A cabinet of the rotating-shelf type with a temp. variation of not more than ± 0.5° is described and scale diagrams are given.

E. B. HUGHES.

**F.p. of milk and its applications.** G. D. ELSDON and J. R. STUBBS (J.S.C.I., 1931, 50, 135—141 τ).—The authors review the published literature on this subject, showing that the f.p. is the most const. of all the properties of cows' milk and therefore the most useful for detecting added water. The theoretical and practical aspects of the subject are discussed and a detailed description of the Hortvet apparatus is given. From their own results the authors show that up to the present the indications obtained by the process have not once been in error. An extensive bibliography is given.

**Acido-proteolytes and thermophiles in the pasteurisation of milk.** C. GORINI (Atti R. Accad. Lincei, 1931, [vi], 12, 519—593).—Milk pasteurised for 30 min. at 63° and at once cooled to 5°, bottled, and stoppered mostly kept good at room temp. (in Milan) for 42—48 hr., but during the summer of 1930, with air temp. reaching 28—30°, this period was sometimes reduced to 28—34 hr. Many of the bacterial forms, like *B. acidificans presamigenes casei*, developing were capable of acido-proteolytic action.

T. H. POPE.

**Nitrogen distribution in Kingston cheese-ripening.** B. A. EAGLES and W. SADLER (Nature, 1931, 127, 705—706).—The N distribution indicates that after the first few hr. of ripening, the proteolytic breakdown in the ripening of Kingston cheese is of a peptic-tryptic-like nature.

L. S. THEOBALD.

**Ammonium hydrogen carbonate given with acid slices of sugar beet [from the diffusion process of sugar manufacture] as a substitute for protein in fodder for milk goats.** F. H. ZIEMER (Biochem. Z., 1931, 232, 352—422).—Provided that the fodder which they receive is otherwise appropriate and sufficient, up to 50% of the digestible protein of such fodder can be replaced by NH<sub>4</sub>HCO<sub>3</sub> without detrimental effect on the health of the animals or on their capacity to yield satisfactory milk.

W. MCCARTNEY.

**Determination of diacetyl in butter.** G. TESTONI and W. CIUSA (Annali Chim. Appl., 1931, 21, 147—150).—The butter (200 g.) is slowly distilled in a current of steam, 10 c.c. of distillate are collected, and these are heated gently with excess of hydroxylamine carbonate, 10 drops of 10% NiSO<sub>4</sub> solution, and aq. NH<sub>3</sub>, a red precipitate of Ni dioximine being formed if diacetyl be present. The precipitate may be either weighed or

converted, without separating it, into the sol. complex containing Ni<sup>++</sup> by heating it with PbO<sub>2</sub> and a few drops of alcoholic KOH. The ppt. is then filtered off and the clear red filtrate made up to a definite vol. and compared in a colorimeter with a similar solution obtained from a known wt. of freshly prepared Ni dioximine. Diacetyl was not detected in fresh milk, edible cacao butter, or molten or pasteurised butters, but about 0.0005% was found in various samples of fresh butter (cf. A., 1928, 924; 1929, 1099).

T. H. POPE.

**Digestibility and fodder value of tapioca and fish meal.** H. EDIN, G. KIHLEN, and A. GUSTAFSSON (Kungl. Landbruks-Akad. Handl. Tidskr., 1930, 69, 882—904; Chem. Zentr., 1931, i, 1378—1379).—Digestion coefficients of tapioca constituents are: crude protein 26, crude fat 80, crude fibre 47, N-free extract 99, carbohydrate 98. Coarsely-powdered material is less digestible (2%) than finely powdered.

A. A. ELDRIDGE.

**Adulteration of fish meal with bone meal.** CLAUSSEN (Z. Fleisch- Milch-hyg., 1931, 41, 137—139; Chem. Zentr., 1931, i, 1379).—On addition of 10% of bone meal to fish meal the number of bone particles in 0.01 g. of the finest powder rose from 4—36 to 98.5.

A. A. ELDRIDGE.

**Cadmium in contact with foodstuffs.** O. C. FORMENTI (Boll. Chim. Farm., 1931, 70, 313—315).—Cd produces chronic poisoning and sometimes death when ingested in small amounts as salts. As regards the possibility of using Cd as a coating for food containers and utensils, the metal is appreciably dissolved in a few hr. by 1% AcOH, tartaric, citric, or lactic acid solution, and by preserved peaches or tomatoes.

T. H. POPE.

**Malt preparations.**—See XVIII.

See also A., June, 757, **Determination of Ca, Mg, and acid-sol. P of milk. Residual N of cows' milk. 774. Ripening of Japanese persimmon. 776. Ether extractives of white leaves of cabbage. Carotene of lettuce. Lipoids from spinach and cabbage. Active substance from raw coffee.**

#### PATENTS.

**Production of bakery products.** POMOSIN-WERKE GES.M.B.H. (B.P. 347,512, 27.5.30. Ger., 1.11.29).—Liquid or powdered pectin (Pomosin) added to the mix for cakes and other bakery products assists in keeping the crumb moist and delaying staling.

E. B. HUGHES.

**Baking of bread or the like and baking powders therefor.** C. F. H. SCHOTT (B.P. 348,509, 2.12.29).—K salts, particularly KHCO<sub>3</sub>, may be used in baking powder if a moisture-absorbing material such as kieselguhr or lycopodium be added.

E. B. HUGHES.

**Colouring material for oleomargarine, butter, and the like.** J. SCHAUB, ASSR. to BEST FOODS, INC. (U.S.P. 1,783,154, 25.11.30. Appl., 6.7.25).—Starch grains are coated with Yellow-OB or -AB by mixing with a solution of the colour in a volatile solvent (COMe<sub>2</sub>). The solvent is evaporated and recovered.

E. B. HUGHES.

**Egg products.** A. K. EPSTEIN (B.P. 346,794, 12.11.29).—The viscosity of liquid egg materials is

modified by the addition of a hydrophillic colloid, *e.g.*, 0.2% of gum tragacanth for whole egg or 1% for yolk. The colloid is dispersed first in a non-aq. medium such as glycerin or edible oil. The product may then be frozen without impairing the viscosity on subsequent thawing. The gum may be used in conjunction with NaCl or sugars as preservatives. E. B. HUGHES.

**Manufacture of cheese.** A. AXELROD (B.P. 347,244, 24.12.29).—Milk is fermented with lactic acid-producing and peptonising bacteria, partly condensed under reduced pressure, and treated with acid and rennet as for normal cheese. E. B. HUGHES.

**Treatment of coffee beans.** K. LENDRICH (B.P. 346,784, 17.1.30).—The excess of chlorogenic acid, which gives rise to the astringent flavour in coffee, may be removed by decomposition into caffeic and quinic acids by treatment of the raw beans with steam under pressure, with or without addition of mineral acid or alkali. E. B. HUGHES.

**Chocolate material.** E. B. WORKING (U.S.P. 1,781,672, 11.11.30. Appl., 16.7.30).—It is claimed that the addition to chocolate of up to 1% of lecithin (usually 0.2%) retards or prevents "greying" or bloom, gives better gloss, increases the temp. range for coating, and renders the material less affected by moisture. The fat content of the chocolate can be reduced (*e.g.*, from 35% to 30%) without decreasing fluidity, and the fat is rendered more digestible. E. B. HUGHES.

**Production of chocolate and chocolate goods.** H. BOLLMANN and B. REWALD (B.P. 347,088, 17.10.30. Ger., 14.5.30. Addn. to B.P. 330,450; B., 1930, 792).—By incorporating 0.25–1% of lecithin in chocolate, 10–25% less cacao butter is required and the tendency for the chocolate to deteriorate is diminished. E. B. HUGHES.

**Manufacture of chewing-gum material.** W. W. TRIGGS. FROM SWEETS LABORATORIES, INC. (B.P. 347,376, 25.2.30).—Chewing-gum base is prepared by drying a mixture of rubber latex, powdered coumarone resin, and hard hydrogenated oil above 100° in a mixer. Flavouring, sugar, and filling materials may be added subsequently. E. B. HUGHES.

**Manufacture of dried food products from raw meat.** INTERNAT. PRODUCTS PROPRIETARY, LTD. (B.P. 346,200, 12.5.30. Austral., 11.5.29).—Raw meat is trimmed of fat, minced, immersed in H<sub>2</sub>O (15–26°), and stirred. The blood matters separate and are strained from the meat tissue, which is pressed dry, again placed in H<sub>2</sub>O, stirred into a spongy mass, and heated to 55–65°, when the water substance soon comes away freely. The meat protein is pressed to a dry cake, granulated or powdered, and "conditioned" by exposure for a few hr. to the atm. The product is practically flavourless, but the liquid expressed at 55–65°, containing the meat flavours, may be concentrated, powdered, and diffused over the meat proteins. E. B. HUGHES.

**Preservation of foodstuffs [meat].** G. M. CLARK. FROM J. F. STELLA and L. TESSIER (B.P. 346,520, 21.2.30).—Meat is preserved (*e.g.*, in the preparation of canned beef) by passing it, by means of a screw conveyor, first through water at 100° for 25 min. and then through

pickling solution at 77° for 1 hr. Apparatus is described. E. B. HUGHES.

**Preservation of [edible] emulsions.** A. MUSER (U.S.P. 1,782,011, 18.11.30. Appl., 26.10.28).—Paraffin wax is used to fill the air space usually found in bottles containing mayonnaise and salad cream. E. B. HUGHES.

**[Capsules for] flavouring foods.** P. LINDNER (B.P. 348,698, 17.2.30. Ger., 20.2.29).

**Margarine.**—See XII.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Chemical examination of *p*-hydroxyphenylmethylaminoethanol hydrochloride.** S. M. GORDON (J. Amer. Pharm. Assoc., 1931, 20, 437–443).—Chemical and physical characteristics of the compound are described and compared with those of related substances. Standards are suggested. E. H. SHARPLES.

**Colour reactions of some medicaments.** J. SIVADJIAN (J. Pharm. Chim., 1931, [viii], 13, 528–529).—Plasmoquin forms a blue colour when its epichlorohydrin solution is boiled with chloranil; percaïne, under similar conditions, forms an emerald-green colour. Na 5-iodo-2-hydroxypyridine acetate ("Uroselectan") in aq. solution (1 c.c.) gives a blue colour when heated with 6–7 drops of Br water; addition of conc. H<sub>2</sub>SO<sub>4</sub> causes its disappearance and the formation of a ppt., but the colour reappears on adding NH<sub>3</sub> solution. An ammoniacal solution of pyrocatechol forms a cherry-red colour when boiled with a little nitroprusside. E. H. SHARPLES.

**Absorptive power of medicinal carbon.** G. FRERICHS (Apoth.-Ztg., 1930, 45, 913–914; Chem. Zentr., 1930, ii, 2416).—The accepted method of determination is criticised and a modification proposed. L. S. THEOBALD.

**Application of the analytical quartz lamp to the examination of perfumes.** E. EKMAN and A. SAMY-SCHLAXEVA (Riechstoffind., 1930, 5, 164–165, 182–184; Chem. Zentr., 1931, i, 1375).—Data for alcohols, aldehydes, ketones, phenols, acids, and esters are tabulated. When pure, few alcohols fluoresce. A. A. ELDRIDGE.

**Sterilisation of cocaine solutions.** W. HÖK (Dansk Tidsskr. Farm., 1931, 5, 37–42).—During the technical sterilisation of local anæsthetic solutions containing cocaine, H<sub>2</sub>O, EtOH, and phenylurethane are lost and the residual solution becomes richer in cocaine and NaCl. Experiments with a 1% aq. solution of pure cocaine hydrochloride showed that no appreciable decomp. of the alkaloid took place during 15 min. sterilisation at 125°, but by more prolonged heating at this temp. or by sterilisation at higher temp., partial decomp. resulted with production of BzOH; in one experiment 30% of the cocaine was decomp. during sterilisation for 30 min. at 150°. H. F. HARWOOD.

**Ash structures of important drug-containing leaves. Recognition of drugs by means of their ash structures.** II. K. OHARA and Y. KONDO (Arch. Pharm., 1931, 269, 292–306; cf. J. Pharm. Soc. Japan, 1929, 49, 573).—The distribution and shape of the CaC<sub>2</sub>O<sub>4</sub> crystals in the ash of leaves prepared in the Werner apparatus (B., 1929, 260) may be used to



distinguish certain drugs. Detailed descriptions of the ash structure of a number of common drugs are given.

A. A. LEVI.

I. "Etrates," a new highly-active type of drug.  
 II. Determination of the extractive content of drugs. C. GRIMME (Pharm. Zentr., 1930, 71, 657—659; 1931, 72, 193—197).—The "Etrate," obtained by evaporating to dryness a concentrated extract of a drug with a proportion of the drug itself, presents advantages for oral administration over other preparations. The compositions of Etrates from cinchona bark, bearberry leaves, valerian root, rhubarb root, senna leaves, and ergot of rye are compared with those of the corresponding fluid extracts and tinctures.

II. Previously employed procedures are reviewed and the amounts of extractive obtained by 12 methods from the first 4 drugs mentioned above and the 4 corresponding Etrates are tabulated. Exhaustive extraction with boiling H<sub>2</sub>O gives the highest results. The results with EtOH vary considerably with its H<sub>2</sub>O content and are, therefore, of little value, except in special cases.

H. E. F. NOTTON.

**Hyoscyamus extract, Ital. P. V.** A. SCOCCIANI (Boll. Chim. farm., 1931, 70, 355—358).—The alkaloid content of 0.5% cannot be attained by the method prescribed in the Italian pharmacopœia V. From survey of foreign pharmacopœias and from experimental data it appears that the max. limit for the alkaloid content of hyoscyamus extract should be 0.3%. This standard should be adopted by the International Commission for Dangerous Drugs.

O. F. LUBATTI.

**Mitchella repens, L., N.F.V.** W. P. BRIGGS (J. Amer. Pharm. Assoc., 1931, 20, 224—226).—The EtOH extract of the drug contains a resin, but no alkaloid, glucoside, or saponin. The drug contains moisture (10.2%), ash (6.2%), insol. ash (2.2%), reducing sugars, tannins, starches, waxes, fats, and protein (4.9%). Aq. or alcoholic extracts have no marked pharmacological activity. H. E. F. NOTTON.

**Hydrastis canadensis (Golden seal).** I. E. GILLIS and H. A. LANGENHAN (J. Amer. Pharm. Assoc., 1931, 20, 210—224).—The % of Et<sub>2</sub>O-sol. alkaloid in the root usually runs parallel with, and about one unit lower than, that in the rhizome (4.3—2.5%; mean, 3.5%). The alkaloid content of the rhizome remains fairly constant (4.2%) from November until June, rising slightly in May and declining to a minimum (3.3%) in September; that of the root shows similar but less regular variations. The alkaloid contents on a moisture- and soil-free basis are also tabulated. H. E. F. NOTTON.

**Tests of Oleum ricini, D.A.B. VI.** W. MEYER (Chem.-Ztg., 1931, 55, 279).—Results of tests of samples of pharmaceutical castor oil are described and compared with the Biechele-Brieger specifications for preparations described in D.A.B. VI. No cryst. material separated, as required by the specification, nor did the oil become pasty, even at -16°. A small quantity of free acid was present, and it is recommended that a test for acidity be applied, especially if the oil is to be used for the manufacture of explosives. H. F. GILLBE.

**Comparison of the chemical and biological assays of Oleum chenopodii.** W. F. REINDOLLAR and J. C.

MUNCH (J. Amer. Pharm. Assoc., 1931, 20, 443—445).—The U.S.P. assay confirms the greater ascaridole content (87—100%) and the higher sp. gr. (0.9883—0.9993) reported for "high-test" oil of chenopodium, as compared with those of "normal" oil (50.0—75.6% and 0.9549—0.9765). No biological assay which bore any relationship to the ascaridole content could be established. E. H. SHARPLES.

**Determination of oil and alkaloids in delphinium seed.** L. N. MARKWOOD (J. Amer. Pharm. Assoc., 1931, 20, 454—455; cf. A., 1927, 1227).—Alkaloids are separated from the light petroleum extract of the ground seed with 5% H<sub>2</sub>SO<sub>4</sub> and the acid extract is made alkaline with 15% NaOH and extracted with CHCl<sub>3</sub>. This last extract is treated again with 5% H<sub>2</sub>SO<sub>4</sub> and the acid extract treated as above. The procedure is repeated and the final CHCl<sub>3</sub> extract is washed and evaporated to give the total alkaloids. The oil content is obtained by evaporation of the light petroleum extract after separation of the alkaloids. *D. consolida* gave 28.3—28.7% of oil and 1.01—1.06% of alkaloids and *D. staphisagria* 34.7—35.4% and 1.22—1.35%, respectively. E. H. SHARPLES.

**Essential oil from the wood of Cedrela Toona, Roxb.** P. P. PILLAI and B. S. RAO (J.S.C.I., 1931, 50, 220—221 T).—The essential oil from the wood of the tree *Cedrela Toona*, Roxb., yield 0.44%,  $d_{25}^{25}$  0.9287,  $n_D^{25}$  1.5007,  $[\alpha]_D^{25}$  +2.5°, acid val. 1.1, ester val. 2.1, acetyl val. 13.3, has been shown to have the approximate composition *l*-copaene 35%, *l*-cadinene and other dicyclic sesquiterpenes 45%, *l*-cadinol 13.0%. The semicarbazone of the ketonic acid, C<sub>15</sub>H<sub>24</sub>O<sub>3</sub>, from the oxidation of *l*-copaene has m.p. 231—232° and that of its Me ester 199—200° (cf. Semmler and Stenzel, A., 1915, i, 427).

**Camphor and camphor oil from Cinnamomum camphora grown in New Zealand.** I. R. SHERWOOD and W. F. SHORT (J.S.C.I., 1931, 50, 195—198 T).—The leaves of 8-year-old camphor trees yielded 1.9% of camphor, *d*- $\alpha$ -pinene, camphene, dipentene, caryophyllene (more than 20%), and cadinene (probably). Wood-oil from 40-year-old trees (lower and middle stem) contained camphor,  $\alpha$ -pinene, camphene, dipentene, cineole, terpineol, safrol (10%), eugenol, cadinene (probably), and a substance m.p. 162°. The sesquiterpene alcohol fraction contained neither primary nor secondary alcohols. The phenylhydrazone and semicarbazone methods of determining camphor are discussed.

**Hydrocarbon constituents of Japanese peppermint oil.** K. J. DUNCAN and W. F. SHORT (J.S.C.I., 1931, 50, 198 T).—Besides *l*-limonene (Schimmel Ber., Apr., 1912, 103), Japanese peppermint oil contains  $\alpha$ -pinene, caryophyllene, and a small quantity of another sesquiterpene (dextrorotatory).

**Hungarian essential oils.** M. JANICSEK (Mezőgazdasági Kutatások, 1929, 2, 153—157).—Oils from experimental plots gave high characteristic numbers: peppermint oils, menthol content 69.7—71.4%; coriander oil, alcohol content (coriandrol) 72.6%; *Salvia sclarea*, ester content 70%; *Lavandula vera*, ester content 62.5%. Oils of marjoram, chenopodium, and *Salvia officinalis* were also examined. H. NICOL.

Ethereal oils from *Backea frutescens*, L., and from *Dalbergia parviflora*, Roxb. D. B. SPOELSTRA (Rec. trav. chim., 1931, 50, 433—440).—Steam-distillation of the leaves of *B. frutescens*, L., at 120° gives 0.9—1.8% of an oil from which by fractional distillation are isolated: *l*- $\alpha$ - and - $\beta$ -pinene (58%); *l*-limonene and dipentene (3.1%); *p*-cymene (0.7%); cineole (7.4%); *l*-linalool, fenchyl alcohol, *l*-borneol, and *l*- $\alpha$ -terpineol (approx. 10%); a dicyclic sesquiterpene, b.p. 126—135°/12 mm. (1.4%), dehydrogenated to cadalene; a dicyclic sesquiterpene alcohol, b.p. 152—157°/13 mm.,  $[\alpha]_D^{20}$  -12° (3%), also giving cadalene on dehydrogenation; phenolic components (2.4%); and a phenolic ether, m.p. 104°, probably identical with that isolated by Penfold (B., 1924, 576). Fractionation of the ethereal oil (0.45—0.8% yield) obtained by steam-distillation of the wood of *D. parviflora*, Roxb., affords *l*-nerolidol, which is the main constituent, whilst traces of furfuraldehyde and farnesol are also probably present.

J. W. BAKER.

Essential oil of *Taiwania Cedar*. K. KAFUKU and R. KATO (Bull. Chem. Soc. Japan, 1931, 6, 65—74).—The wood from *Taiwania cryptomerioides*, Hayata, gave 0.23% of an oil having  $d_4^{15}$  0.9593,  $n_D^{15}$  1.5104,  $\alpha_D^{15}$  -2.00°, acid value 0.38, ester value 10.52, ester value after acetylation 83.00. Caryophyllene, humulene, and cadinene were identified, and there was also obtained a sesquiterpene alcohol "taiwanol," b.p. 141—143°/6 mm.,  $d_4^{20}$  0.9692,  $n_D^{20}$  1.5045,  $\alpha_D^{20}$  -42.56° (urethane, m.p. 134—135°). With HCl the substance gave cadinene dihydrochloride, whilst oxidation gave a substance, m.p. 170°, and dehydration with formic acid gave cadinene. The alcohol is a tertiary alcohol either identical or isomeric with cadinol (A., 1914, i, 63). Cadinene oxidised with  $\text{KMnO}_4$  in aq.  $\text{COMe}_2$  gives a substance,  $\text{C}_{12}\text{H}_{22}\text{O}_2$ , m.p. 142—143°.

A. A. LEVI.

Essential oil of leaves of *Chamaecyparis obtusa*, Sieb. et zucc., f. *Formosana*, Hayata, or Arisan "Hinoki." II. K. KAFUKU and T. NOZOE (Bull. Chem. Soc. Japan, 1931, 6, 111—118; cf. B., 1931, 565).—The high-boiling portions of this oil contain a new sesquiterpene, *sesquichamene*, b.p. 122.5—123.5°/12 mm.,  $n_D^{25}$  1.5021,  $d_4^{25}$  0.9277,  $\alpha_D^{25}$  -89.85°, [nitroschloride, m.p. 77.5—78.5° (decomp.); nitrobenzylamine, m.p. 165—166° (decomp.)], oxidation of which with  $\text{KMnO}_4$  in  $\text{COMe}_2$  gives a substance (*semicarbazone*, m.p. 233°), a substance,  $\text{C}_{15}\text{H}_{26}\text{O}_2$ , m.p. 89—91°, and a substance, m.p. 240—245° (decomp., sinters 220°). Treatment of *sesquichamene* with alcoholic  $\text{H}_2\text{SO}_4$  gives *isosesquichamene*, b.p. 129—131°/12 mm.,  $n_D^{21.5}$  1.5109,  $d_4^{21.5}$  0.9320,  $\alpha_D^{21.5}$  -8.52°. Other constituents are cadinene and cadinol.

A. A. LEVI.

Volatile oil of *Pycnanthemum miticans*. H. W. WERNER (J. Amer. Pharm. Assoc., 1931, 20, 445—448).—Steam-distillation of the dried plant gave 1.57% of oil having:  $d_{20}^{20}$  0.9250,  $\alpha_D^{20}$  +16.49°,  $n_D^{20}$  1.4807, ester val. 12.64 (equiv. to 4.47% of  $\text{C}_{10}\text{H}_{18}\text{OH}$  as acetate), ester val. after acetylation 61.29 (equiv. to 17.90% of  $\text{C}_{10}\text{H}_{18}\text{OH}$  in original oil), solubility in 70% EtOH 1 in 1.82, ketones ( $\text{Na}_2\text{SO}_3$  method) 71.75%. Menthone, pulegone (60—75.75%), menthol or *d*-neomenthol, and another ketone ( $\beta$ -thujone?) were present, together

with AcOH and formic acid, terpenes, and sesquiterpenes.

E. H. SHARPLES.

Determination of hexamethylenetetramine.—See III. Solution of Pb subacetate (U.S.P.). Alkali bitartrates. Sodium bismuth citrate.—See VII. Ethereal oils as germicides.—See XXIII.

See also A., June, 684, Dibismuthyl monosodium citrate solutions. 698, Capillary analysis. 725, *N*-Phenylalkyl-*N*-methylamino-alkyl benzoates and *p*-aminobenzoates. 731, Anaesthetics of the diphenyl ether series. 742, Antimalarials. 750, Colour reactions of brucine. Fluorescence of alkaloids. 752, Determination of narcotine, santonin, etc. Titration of alkaloid salts. 753, Determination of iodobismuthate. 763, Toxic action etc. of ethylene glycol. Styryl- and anil-benzthiazole derivatives as antiseptics etc. 764, Toxicity of *Corydalis caseana*. 769, Bacterial lipins. 771, Extraction of insulin. 775, Rhusinic acid. 777, Constituents of "Salpamisri." 778, Microchemical detection of colchicine, piperine, etc., and of choline in plants.

PATENTS.

Arylamino-alcohol [therapeutic] compound. R. ADAMS, Assr. to ABBOTT LABORATORIES (U.S.P. 1,767,423, 24.6.30. Appl., 27.1.28).—Compounds of the type,  $\text{Ph}\cdot\text{CH}(\text{OH})\cdot\text{CHR}\cdot\text{NR}'\text{R}''$ , where R and R' are alkyl and R'' is H or alkyl above  $\text{C}_1$ , have a pressor and sympathomimetic effect. They are produced by reduction of the corresponding ketones. In the examples, R = Me, R'' = H, and R' = Et (ketone, m.p. 183—184°; alcohol, m.p. 190—191°), Pr<sup>a</sup> (m.p. 167—168°, and 205—206°), Pr<sup>b</sup> (m.p. 213—213.5°, and 193°), Bu (m.p. 158—159°, and 220—221°), or *n*-amyl (m.p. 150°, and 219°); or R = Me, R' = R'' = Et (m.p. 167—168°, and 205—206°); or R' = Me, R'' = H, and R = Et (m.p. 190—192°, and 197—199°) or Pr<sup>a</sup> (m.p. 182—184°, and 224—225°).

C. HOLLINS.

Preparation of stable solutions of acetylcholine salts. G. B. ELLIS. From Soc. Des Usines Chim. Rhône-Poulenc (B.P. 345,713, 13.1.30).—Solutions of acetylcholine salts (hydrohalides) in incompletely acylated polyhydric alcohols, e.g., in glycerol mono- or di-acetate or  $\beta$ -hydroxyethyl acetate, are stable cold or hot and are suitable for injections.

C. HOLLINS.

Manufacture of double compounds of the acridone series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 345,631, 24.12.29. Admn. to B.P. 328,212; B., 1930, 741).—Technical mixtures produced in the alkylation of 3:7-diaminoacridine are used, after analysis, for the preparation of the double compounds of the prior patent.

C. HOLLINS.

Mercury compound of *m*-cresol. D. MACD. COPLEY and J. P. SNYDER, Assrs. to NORWICH PHARMACAL Co. (U.S.P. 1,782,090, 18.11.30. Appl., 14.9.27).—*m*-Cresol, dissolved in alcoholic KOH, is mixed with alcoholic  $\text{HgCl}_2$  at 15° and heated at 100° until pasty. The product is filtered off, washed, and dried at 100°.

C. HOLLINS.

Preparation of cineole-containing oil mixtures. RHEINISCHE KAMPFER-FABR. G.M.B.H. (B.P. 345,517, 5.4.30. Ger., 15.4.29).—In the production of cineoles

from terpineol, terpinenol, terpin, terpin hydrate, etc. by isomerisation and/or dehydration, 1:8-cineole is the main product below 80°, *e.g.*, with 30–40% H<sub>2</sub>SO<sub>4</sub> at 35° or 60% acid at –10°. The product must be removed as formed, *e.g.*, by distillation in vac., or by extraction with PhMe or CHCl<sub>3</sub>. Hydration products from pinene or sabinene are suitable starting materials.

C. HOLLINS.

**Basic products. Albumose-like products.**—See III. **Sol. cellulose esters.**—See V.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

See A., June, 722, **Light-sensitive diazo compounds.** 752, **Determination of diazo-N in light-sensitive compounds.**

### PATENTS.

**Light-sensitive layer and manufacture of photographic images.** J. EGGERT, R. SCHMIDT, and B. WENDT, Assrs. to AGFA ANSCO CORP. (U.S.P. 1,782,259, 18.11.30. Appl., 12.7.29. Ger., 17.7.28).—The conversion of *cis*- into *trans*-isomerides by light is utilised, the print being fixed by means of a selective solvent etc. *trans*-Dibenzoyl ethylene on black paper gives a positive from a positive on washing with EtOH. Maleic acid on white paper with a trace of Br as catalyst gives a positive from a negative on conversion of the fumaric acid into insol. Ag or Hg salt, followed by washing and development with chromate. Phenylcitraconic acid on collodion with phenacyl bromide as catalyst is washed with CHCl<sub>3</sub>, and developed with a solution of a Hg, Ag, or Tl salt to a white image of the phenylmesaconate, which becomes black in a Na<sub>2</sub>S bath. C. HOLLINS.

**Production of colour-record kinematograph films.** TECHNICOLOR MOTION PICTURE CORP., Asses. of L. P. TROLAND (B.P. 349,318, 31.5.30. U.S., 15.6.29).

**Reproduction of colour-record images on lenticular films.** SOC. FRANÇ. DE CINÉMAT. ET DE PHOT. FILMS EN COULEUR KELLER-DORAIN, and J. L. VIDAL (B.P. 349,267, 2.5.30. Fr., 2.5.29).

**Nitrocellulose compositions. Films.**—See V.

## XXII.—EXPLOSIVES; MATCHES.

**Nitrocellulose.**—See V. **Oleum ricini.**—See XX.

See also A., June, 689, **Transmission of detonation.**

### PATENTS.

**Manufacture of nitrostarch.** O. A. PICKETT, Assr. to HERCULES POWDER Co. (U.S.P. 1,779,825, 28.10.30. Appl., 4.5.26).—The nitration acids contain 12–15% H<sub>2</sub>O, so as to promote swelling of the starch during nitration. The dried starch is sieved into the acid and forced beneath the surface by stirrers, the temp. of the acid being maintained at 20–30° by means of a cooling jacket. After nitration, the mixture of acid and nitrostarch is run into H<sub>2</sub>O, the acid wash-water is drawn off, and the nitrostarch stabilised, its viscosity being reduced, if desired, by boiling with steam. The product is readily sol. in the usual solvents employed in lacquer manufacture. W. J. WRIGHT.

**Priming mixtures.** J. D. McNUTT, Assr. to WINCHESTER REPEATING ARMS Co. (U.S.P. 1,774,992,

1,779,820—1, and 1,779,851, [A] 2.9.30, [B–D] 28.10.30. Appl., [A] 21.11.27, [B] 24.12.27, [C] 21.6.28, [D] 31.3.27).—(A) A mixture of 20–40% of PbO<sub>2</sub>, 5–10% of BaCrO<sub>4</sub>, 25–40% of Hg fulminate, and 3–15% of tetranitromethylaniline, together with 15–20% of ground glass, is claimed. (B) The aniline derivative in (A) may be replaced by pentaerythritol tetranitrate, and this latter, wholly or partly, by trinitroresorcinol; also Ba(NO<sub>3</sub>)<sub>2</sub> is substituted for BaCrO<sub>4</sub>. (C) A mixture of 45% of Ba(NO<sub>3</sub>)<sub>2</sub>, 35% of Hg fulminate, 16% of Pb(CNS)<sub>2</sub>, 2% of BaCO<sub>3</sub>, and 2% of charcoal is claimed. (D) The composition may consist of 475 pts. of Hg fulminate (16% H<sub>2</sub>O), 240 pts. of Ba(NO<sub>3</sub>)<sub>2</sub>, 255 pts. of Sb sulphide, 30 pts. of trinitrotoluene, and  $\frac{1}{2}$ –2 pts. of a stabiliser, *e.g.*, diphenylamine. W. J. WRIGHT.

**Explosive priming mixtures.** L. MELLERSH-JACKSON. From WINCHESTER REPEATING ARMS Co. (B.P. 346,092, 11.2.30).—The use of witherite, instead of artificial BaCO<sub>3</sub>, as a constituent of priming mixtures containing Sb sulphide, eliminates the use of ground glass. A suitable mixture consists of Hg fulminate 35–50, basic Pb nitrate 23–33, Pb(CNS)<sub>2</sub> 5–15, Sb sulphide 5–20, and witherite 5–10%. W. J. WRIGHT.

**Gelignite and gelatin dynamite.** R. L. HILL, Assr. to ATLAS POWDER Co. (U.S.P. 1,778,718, 21.10.30. Appl., 14.12.28).—Part or all of the NaNO<sub>3</sub> or KNO<sub>3</sub> of the explosive is added in the form of pellets, to give the explosive improved plasticity and higher sensitiveness to detonation. W. J. WRIGHT.

**Manufacture of propellent explosives.** IMPERIAL CHEM. INDUSTRIES, LTD., R. BURNS, and O. W. STICKLAND (B.P. 346,295, 6.1.30).—In order to prevent metallic fouling in firearms, an organo-metallic compound, such as a substituted stannane, having approx. the same sp. gr. as have the other constituents of the explosive, is incorporated with the explosive. Suitable compounds for powders of the (a) solventless, (b) nitrocellulose, and (c) nitroglycerin type are the tetraphenyl, tetra-*n*-butyl, and propyl tri-*n*-butyl compounds of Sn, respectively, the last two compounds being dissolved in the volatile solvent employed. W. J. WRIGHT.

**Smokeless powder.** F. I. DU PONT, Assr. to U.S.F. POWDER Co. (U.S.P. 1,780,911, 11.11.30. Appl., 29.10.26).—In addition to the usual solvent employed in the production of propellent powders there is added about 1–5% of a substance, such as glycerin, which contains C and H, is liquid at room temp. and non-volatile at the drying temp. of the powder, forms a stable mixture with H<sub>2</sub>O, and is a non-solvent for nitrocellulose. Undue loss of residual solvent during drying and the consequent development of dangerous pressures in practice are thus avoided. W. J. WRIGHT.

**Absorbent material for explosives.** H. C. BUGBIRD (U.S.P. 1,779,530, 28.10.30. Appl., 1.11.24. Renewed 17.3.27).—Carbonised lignin residue, after leaching to remove alkaline and other water-sol. material and heating to redness in absence of air, is used as an absorbent for liquid O<sub>2</sub>. W. J. WRIGHT.

**Preparation of absorbents for liquid-oxygen explosives.** H. C. BUGBIRD (U.S.P. 1,781,214, 11.11.30. Appl., 28.9.25).—Lignin residues from the manufacture of wood pulp are carbonised, leached, and ground to a

relatively coarse granular product, the voids being then filled with a finely-divided absorbent material, *e.g.*, carbon black, having an absorptive capacity for liquid  $O_2$  equal to at least 2.6 times its wt. W. J. WRIGHT.

Nitrocellulose.—See V.

### XXIII.—SANITATION; WATER PURIFICATION.

**Organic constituents of fresh and ripe sewage sludge.** H. HEUKELEKIAN (New Jersey Agric. Exp. Sta. Bull., 1930, No. 502, 31—36).—Fresh solids contain mainly fats and crude protein with smaller proportions of  $H_2O$ -soluble and  $Et_2O$ -sol. matter, cellulose, hemicellulose, and lignin. During ripening, the principal loss of solids falls on fatty matter and cellulose.

A. G. POLLARD.

**Distribution of iodine in sewage and use of sewage sludge as iodine manure.** R. KÖHLER (Mitt. Lab. Preuss. Geol. Landesanst., 1930, No. 11, 1—14; Chem. Zentr., 1931, i, 1332).—Town sewage is relatively rich in I. Removal of the org. matter in the course of sewage purification reduces the I content of the effluent. The I content of crops may be increased by fertilisation with the sludge.

A. A. ELDRIDGE.

**Effect of certain chemicals on the vacuum filtration and gravity drying of ripe [sewage] sludge.** W. H. BAUMGARTNER (New Jersey Agric. Exp. Sta. Bull., 1930, No. 502, 17—25).—Addition of  $FeCl_3$  to ripe sewage sludge increases the rate of filtration and of the drainage from drying beds. The resulting sludge has a more open texture. Optimum effects corresponded with definite  $p_H$  values, peak values being obtained by the addition of 2 lb. and of 7 lb. of  $FeCl_3$  per cu. yd. of sludge.  $FeCl_3$  adjusts the reaction of the sludge to produce more complete flocculation of colloids and also liberates  $CO_2$ , which renders solids more porous and assists their flotation.

A. G. POLLARD.

**[Sewage-]sludge drying; relation between drainage and evaporation.** W. RUDOLFS and I. O. LACY (New Jersey Agric. Exp. Sta. Bull., 1930, No. 502, 26—29).—At temp. up to  $55^\circ$  the rate of drainage of  $H_2O$  from sewage sludge is a more important factor in drying than is the rate of evaporation.

A. G. POLLARD.

**Drying of fresh sewage solids.** A. J. FISCHER (New Jersey Agric. Exp. Sta. Bull., 1930, No. 502, 29—31).—The drying of fresh solids in the open after treatment with  $CaO$ , alum, or a mixture of these is unsuccessful on account of poor drainage and objectionable odours.

A. G. POLLARD.

**Effectiveness of sodium fluosilicate as compared with borax in controlling the house fly.** S. MARCOVITCH and M. V. ANTHONY (J. Econ. Entom., 1931, 24, 490—497).— $Na_2SiF_6$  proved superior to borax for the treatment of manure heaps for house-fly control, and the risk of subsequent soil injury was much reduced.

A. G. POLLARD.

**Disinfectant action of trichloroethylene.** W. RIMPAU (Z. Hyg., 1931, 112, 202—221).—Stable emulsions of  $C_2HCl_3$ ,  $CHCl_3$ , and  $C_2H_2Cl_4$  have strong disinfectant properties. Solutions of  $PhOH$  derivatives, salicylic acid, etc. in  $C_2HCl_3$  are active disinfectants only when emulsified.

A. G. POLLARD.

**Germicidal action of ethereal oils.** S. L. MALOWAN (Z. Hyg., 1931, 112, 93—94).—The bactericidal action of ethereal oils is closely related to their solubility in  $H_2O$ . Oils consisting principally of esters are less bactericidal than those mainly composed of alcohols.

A. G. POLLARD.

**Ferric iron coagulation [for river water].** A. POTTER and W. I. KLEIN (J. Amer. Water Works' Assoc., 1931, 23, 719—727).— $Fe^{+++}$  has proved a more satisfactory coagulant than  $Al_2(SO_4)_3$  in the 5 million gal./day plant at Mamaroneck, N.Y., which treats river water from a small watershed without impounding reservoirs and so is subject to rapid variations in colour, taste, etc. The advantage is largely due to the better settling qualities of the  $Fe(OH)_3$  floc and the consequent effect thereof on the condition of the sand filters.

C. JEPSON.

**Unusual colour-removal plant [for well-water supplies].** C. WILSON (J. Amer. Water Works' Assoc., 1931, 23, 689—695).—An experimental plant to treat 69,000 gal. per day has demonstrated that at Los Angeles a peaty well-water may be freed from colour at low cost by means of  $FeCl_3$ . The plant permits a continuous return of the  $Fe(OH)_3$  sludge to the reaction chamber, with mechanical agitation to remove gases and so facilitate settlement. The sand filters are of coarse material, 10 in. deep, and have 10 in. freeboard for backwashing. The influent is applied by means of a grid laid on the sand surface with holes so placed as to deliver the  $H_2O$  horizontally.

C. JEPSON.

**Photoelectric control of chlorine feed [in water-works' practice].** J. H. HARRINGTON (J. Amer. Water Works' Assoc., 1931, 23, 736—739).—The dosage of Cl is automatically varied by means of apparatus controlled by differences in the intensity of the light falling on to two light-sensitive cells after passing through two colour cells. Each colour cell contains a similar sample of the water and is placed opposite and equidistant from a common source of light, but while in one cell a colour is developed by a suitable reagent proportional to the amount of residual Cl, the other has coloured glasses interposed to represent the colour which would be produced by the residual desired. Any variation from this standard is communicated by the photoelectric cells to the apparatus controlling the rate of feed and is thus corrected.

C. JEPSON.

**Fluorescence of water in filtered ultra-violet light as an indicator of its purity.** H. IVEKOVIC (Z. Hyg., 1931, 112, 54—61).—The bluish-white fluorescence frequently shown in filtered ultra-violet light by  $H_2O$  samples is due to dissolved organic matter. The intensity of the fluorescence increases with the O consumption value of the sample. Comparative tests of purity may be obtained by means of the intensity of fluorescence of gelatin solutions made with the samples.

A. G. POLLARD.

#### PATENTS.

**Water-softening plants.** T. W. ELEY (B.P. 348,749, 1.3.30).

**Removing solid particles from liquids.**—See I. **Distillation of material.**—See II. **Sol. cellulose esters.**—See V.