# BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JAN. 19 and 26, 1934.\*

# I.-GENERAL; PLANT; MACHINERY.

Higher output of rotary furnaces, its calculation and recommendations for further extensions in the use of this type of furnace. J. LAMORT (Feuerfest, 1933, 9, 137–140).—Mathematical discussion of the heat economics of various types of furnace indicates the superiority of rotary over stationary furnaces since the former heat the charge directly by the flame and indirectly through the hot walls. Practical tests show that the heat efficiency of rotary enamel kilns is 36%, of rotary cement kilns 31%, of open-hearth steel furnaces about 21.5%, and of glass-melting tanks < 18%, generally 10–15%. A. R. P.

Measurement of tube-wall temperatures in heattransfer experiments. G. M. HEBBARD and W. L. BADGER (Ind. Eng. Chem. [Anal.], 1933, 5, 359—362).— A thermocouple is inserted in a hole drilled through a chord of the wall, and the leads are carried in an annular groove around the tube (T) and withdrawn at the opposite side. Solder is admitted through a plug hole to bring the junction into metallic contact with T. The groove and chord hole are then filled with bakelite cement of special composition. E. S. H.

Buhrstone mill dressing. BALTIMORE PAINT & VARNISH PRODUCTION CLUB (Amer. Paint & Varnish Manufrs.' Assoc., Nov., 1933, Circ. No. 445, 432—437).— A machine for dressing the stones is described, and the factors which control the output and fineness of the product are discussed. S. M.

Errors in determination of carbonate in boiler waters. W. C. SCHROEDER (Ind. Eng. Chem. [Anal.], 1933, 5, 389–393).—Winkler's method is inaccurate in presence of  $SO_4^{\prime\prime}$ . The phenolphthalein–Me-orange titration is inaccurate in solutions containing  $\leq 60$  p.p.m.  $CO_3^{\prime\prime}$ . E. S. H.

Foam fire-fighting methods. H. S. SIMONIS (Chem. & Ind., 1933, 957-958).—The use of foam prepared mechanically from air, saponin, and H<sub>2</sub>O is indicated.

D. K. M.

Quantitative organic microchemical analysis in industrial problems. W. R. KIRNER (Ind. Eng. Chem. [Anal.], 1933, 5, 363–369).—The methods and advantages are reviewed. R. S. C.

Colour measurement. G. S. FAWCETT (Chem. & Ind., 1933, 1005-1007).—A review.

[Boilers for] S dyes.—See IV. Wear of muller tyres.—See VIII. Clarifiers etc.—See XIII.

#### PATENTS.

Rotary kiln. F. W. CORKILL, Assr. to PACIFIC COAST BORAX CO. (U.S.P. 1,898,480, 21.2.33. Appl., 12.8.29).— A rotary kiln is provided with a no. of obstructions alternately of annular form attached to, and disc form spaced from, the shell, the annuli being perforated in order to permit normal progress of fine material while delaying the coarse. B. M. V.

Rotary and semi-rotary furnaces. W. F. WILT-SHIRE (B.P. 400,956, 29.4. and 26.7.32).—In a rotatable or rockable melting furnace the burner and the outlet flue are in or near the same end of the furnace and the opposite end is curved to deflect the gases downwards and backwards. B. M. V.

**Tunnel dryer.** T. W. GARVE (U.S.P. 1,897,387, 14.2.33. Appl., 29.4.30).—A tunnel kiln has longitudinal flues in the roof for both hot entering and moist leaving gases, the central inlet ports being provided with aprons to distribute gases through the goods. B. M. V.

Heat exchanger. J. PRICE and S. BROWN, ASSTS. to GRISCOM-RUSSELL CO. (U.S.P. 1,897,213, 14.2.33. Appl., 28.7.32).—A joint ring for a cover plate of a header is described; when the header is divided into a no. of compartments the divisions are formed by comparatively light internal plates. B. M. V.

Pasting mechanism for chemical dryers. A. O. HURXTHAL, Assr. to PROCTOR & SCHWARTZ, INC. (U.S.P. 1,897,904, 14.2.33. Appl., 25.3.30).—Material that is to be dried on a foraminous conveyor is pasted on by a hopper (H) and roller, and the excess that passes through the perforations is scraped off the back (and returned to H) so that the grip of the dry material will not be too great. B. M. V.

Dryer roll. T. L. BRANNEN, Assr. to BROWN Co. (U.S.P. 1,899,256, 28.2.33. Appl., 6.9.30).—A very large roll is constructed of a no. of cast-Fe sections having embedded steam-pipes. The blocks are held resiliently on a spider by centripetal spring tension.

B. M. V. Grinding mill. R. P. JUGEL, ASST. to AMER. BALL MILL CO. (U.S.P. 1,898,187, 21.2.33. Appl., 31.3.30).— The lining sections of a ball mill are provided with outwardly extending lugs which enter recesses in the shell and are secured by wooden wedges driven between the pairs of lugs on adjacent sections. B. M. V.

**Pulverising mills.** BABCOCK & WILCOX, LTD. (B.P. 401,115, 16.11.32. U.S., 16.11.31).—The apparatus comprises a lower ring of balls (A), an inner upper ring (B), and an outer upper ring (C). The lower race for Ais fixed, the upper race of A and the lower races of Band C are on a single-driven member which is free to rise, and separate upper races are provided for B and Cwhich are free to rise against springs but are prevented

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

from rotation. The material passes through B and C and the oversize through A, being classified by various fractions of the air current, the material from A passing into the total current, which is powerful enough to lift it completely into a classification zone above.

B. M. V.

Pulverising apparatus. M. FRISCH, ASST. to FOSTER WHEELER CORP. (U.S.P. 1,898,086, 21.2.33. Appl., 12.6.30).—In an air-borne coal-pulverising system, electromagnetic means are described for closing dampers in the conduits before and after the grinding mill and for stopping the fan motors in the event of a blow-back, or failure of, or manual interruption of, the electrical power. B. M. V.

Pulverising apparatus and the like. C. H. CORBISHLEY, ASST. to FOSTER WHEELER CORP. (U.S.P. 1,899,015, 28.2.33. Appl., 20.11.30).—A noise-deadening sheath for a ball mill is described. B. M. V.

Apparatus for pulverising and classifying ore and the like. F. C. PEREW, Assr. to E. T. WALLACE (U.S.P. 1,897,176, 14.2.33. Appl., 31.12.28).—An apparatus comprising a gyrating annular pestle and rotating mortar, suitable for amalgamation and other treatments, is described. B. M. V.

Pulverising of minerals and similar materials. INTERNAT. PULVERISING CORP. (B.P. 401,512, 21.12.32. U.S., 18.1.32).—A mass of loose pieces of material is pulverised by impact caused by a no. of horizontal jets of fluid, *e.g.*, superheated steam, which converge and, after uniting, form a substantially free upward passage through which all the material is conveyed away by the fluid to a separating zone; the oversize drops back to the impact chamber. B. M. V.

Pulveriser. H. L. WILKINS, ASST. to ARCHER DANIELS MIDLAND CO. (U.S.P. 1,897,744, 14.2.33. Appl., 14.7.30).—Swing hammers with renewable shoes are described. B. M. V.

Stirrer mechanism. A.-G. KÜHNLE, KOPP, & KAUSCH, and K. FOERSTER (B.P. 401,583, 10.4.33).— Agitator blades of the disc or propeller type are situated on axes which are oblique to the shaft. B. M. V.

Filters. C. G. VOKES (B.P. 401,287, 28.4.32).— A pleated, cylindrical (or other shaped) filter medium is substantially self-sustaining, but excessive pressure from the end caps is taken by thrust members that take no part in sustaining the pleats. B. M. V.

Filtration medium. H. E. BIRKHOLZ, ASST. to AMER. AIR FILTER Co., INC. (U.S.P. 1,897,976, 14.2.33. Appl., 25.11.27. Renewed 21.2.31).—A filtering medium for air or gas that may be scrapped when choked is formed of several superposed sheets of paper of very loose texture compressed together along widelyspaced lines, to obtain slight adherence for easier handling. B. M. V.

Liquid straining device. L. B. GRUMAN, ASST. to AMES PUMP Co., INC. (U.S.P. 1,899,030, 28.2.33. Appl., 27.8.30).—An easily cleaned strainer for insertion in a pipe-line is described. B. M. V.

Sedimentation tanks, especially for sewage treatment. R. A. DIBDIN (B.P. 401,604, 22.5.33).— A no. of horizontal or slightly inclined plates are arranged in columns or rows, the plates being alternately closely spaced and more widely spaced in the vertical direction. B. M. V.

Fractional distillation of volatile liquids and other processes of contacting immiscible fluids of different densities. W. J. PODBIELNIAK (B.P. 401,484, 13.10.32).—Fractionation or other form of countercurrent contact is effected in a conical or flat spiral tube, or set of tubes in parallel, rotated at centrifugal speed, the heavy fluid being fed in at the small end and the light being forced in at the large end of the spiral. B. M. V.

Bubble tower. R. R. BOTTOMS, ASST. to GIRDLER CORP. (U.S.P. 1,899,409, 28.2.33. 14.8.31).—A bubble cap in which the gas, after forming the usual bubbles in the pool, is trapped and mixed with the liquid flowing down from the plate above is described. B. M. V.

Apparatus for treating liquids. A. JENSEN (U.S.P. 1,897,613, 14.2.33. Appl., 12.3.30).—An apparatus for mixing, dispersing, heating, and cooling, *e.g.*, for the reconstitution of milk, comprises a drum rotating closely to a stationary drum embodying conduits for a heat-exchanging fluid. A form of hygienic gasket for the cover plate is claimed. B. M. V.

Apparatus for mixing solids with liquids. Bell BROS. (MANCHESTER, 1927), LTD., and C. G. BENSON (B.P. 400,917, 19.1.32).—A paste of, e.g., CaCO<sub>3</sub> and  $H_2O$  is displaced from a closed vessel provided with an agitator into a main stream of  $H_2O$  to be treated, or into a by-pass connected across the mouth and throat of a Venturi tube, the displacement being effected by  $H_2O$  pumped by a turbine in the main stream connected to a pump of positive but variable delivery. B. M. V.

Means for supplying air or gas in a more or less finely-divided state to liquids. ACTIVATED SLUDGE, LTD., E. R. JONES, A. B. DENTON, and J. A. COOMBS (B.P. 400,940, 22.2.32).—The apparatus described in B.P. 397,841 (B., 1933, 945) is modified. B. M. V.

Separation of liquid from vapours and gases. J. PRIMROSE, ASST. to FOSTER WHEELER CORP. (U.S.P. 1,897,766, 14.2.33. Appl., 31.1.25).—Forms of bubbling trays in a fractionating tower are described; exact adjustment of level is provided for. B. M. V.

[Air-]separating system. J. M. LE GRAND, ASST. to CARRIER ENG. CORP. (U.S.P. 1,898,733, 21.2.33. Appl., 27.7.28).—In a pulveriser (P) with air separator (S), part of the air from the fan is sent through P, and another part, adjustable to alter the fineness of the delivered material, is sent direct to S. B. M. V.

Separator. J. H. FEDELER (U.S.P. 1,899,498, 28.2.33. Appl., 4.12.28).—A dust separator of the stationary centrifugal type is built around a chimney stack (S), the entry and exit being placed below and above a damper in S. B. M. V.

Mechanically-operated gas analyser. A. B. CUN-NINGHAM, Assr. to REPUBLIC FLOW METERS CO. (U.S.P. 1,897,721, 14.2.33. Appl., 18.6.26).—A CO<sub>2</sub> recorder comprises : a rotary pump for delivering excess of gas sample, a reciprocating measuring pump in an oil-seal, a caustic absorbent solution in the base through which the measured gas is bubbled, and a bell for measuring the final vol. of the gas. B. M. V.

**Removal of materials from reaction vessels.** F. UHDE (B.P. 401,429, 11.7.32. Ger., 11.7.31).—Materials, whether gases, liquids, or fluent solids, are "expanded" from a high-pressure vessel to one of lower pressure by a reciprocating or other type of engine, the valves of which are adjusted so that on the outstroke of the piston the inlet valve closes when sufficient material has entered for the pressure to be at the lower val. at the end of the stroke, and on the instroke the exhaust valve closes when sufficient material remains to be compressed to the higher pressure at the end of that stroke. B. M. V.

Friction element. C. DE LUKACSEVICS, Assr. to L. BYRNE (U.S.P. 1,899,239, 28.2.33. Appl., 15.12.28). —A brake lining is composed of black graphitic clay 45, ZrO<sub>2</sub> 25, felspar 15, agalmatolite 5, magnesite 5, kaolin 5%. B. M. V.

Combustion in shaft furnaces.—See II. Washing powder [for boiler feed-H<sub>2</sub>O].—See VII.

## II.—FUEL; GAS; TAR; MINERAL OILS.

Microstructure of the coal of certain fossil tree barks. H. G. A. HICKLING and C. E. MARSHALL (North Eng. Inst. Min. Mech. Eng., Oct., 1933. Advance copy, 16 pp.; cf. B., 1933, 5).-A study has been made of the microstructure of a no. of samples of vitrain (I) formed from the bark of fossil trees belonging to the Bothrodendra, Sigillariæ, or Lepidodendra. The types of structure are illustrated by photomicrographs and described in full. It is concluded that plant tissues may be preserved in coal in three conditions: (1) as (I) in which the cells or fibres of the plant have suffered compression so that the cell cavities have been more or less completely eliminated, the (I) consisting essentially of the closely-packed cell walls; (2) as (I) in which the original cell cavities are uncompressed and are occupied by org. material which now forms the greater part of the coal; and (3) as fusain (II), which is formed of the walls only of the original cells or fibres, without any trace of org. content, the walls being either uncompressed or impacted by fracture. The investigation has established the existence of a condition of preservation intermediate between (I) and (II), in which the cell cavities of the tissues remain unfilled, as in (II), whilst the cell walls have not undergone the extreme alteration which leads to opacity, but remain transparent in thin section. Whilst in some cases the walls have become sufficiently brittle to yield to pressure by fracture, giving rise to "bogen-struktur," in other cases they show the folding characteristic of the more plastic walls of vitrainised tissue. The mode of origin of (I) and the precise connotation of the term are discussed. The microscopic components of coal that have been generally recognised are spores, cuticles, resin bodies, fine granular matrix ("residuum"), (I), and (II). (I) and (II) alone may occur in pieces of such size as to form macroscopic constituents. Clarain and durain, however, are compounded of all these microscopic constituents in varying proportions, including small fragments of (I) and (II) (cf. B., 1932, 757). A. B. M.

**Reactivity of coal.** C. COLLINA and I. GIORDANO (Acqua e Gas, 1933, 22, 46—48; Chem. Zentr., 1933, ii, 308).—" KV" vals., given by  $KV/u = -2 \log (1 - \alpha)$ , where  $\alpha$  is the degree of dissociation of the CO<sub>2</sub> passed over the coal, and u is its velocity in c.c. per sec., are: graphite 0.159—0.436, gas-coke 0.441—0.622, anthracite 0.360—1.043, lignite semi-coke 3.774— 1.844. A. A. E.

Relative efficiencies of domestic fuels in relation to their costs and their advantages. R. A. SHER-MAN (Amer. Inst. Min. Met. Eng., Contr. No. 63, 1933, 8 pp.).—Fixed charges (C) form an important fraction of the total cost of domestic heating using any mechanical equipment. The greater the % of the total cost that is represented by C the less important is the thermal efficiency (T) of the system. Over a large part of the U.S.A., coal, hand-fired or stoker-fired, is cheaper than gas or oil; where the latter have been adopted the reason has been their greater convenience and cleanliness and not their higher T. Attention is directed to the need for automatic combustion equipment for coal at a lower first cost. A. B. M.

"Fliesskohle" from brown coal. G. BENTHIN (Angew. Chem., 1933, 46, 742-744).—A lecture on the prep. of pulverised coal in oil. E. S. H.

Combustion. XVI. "A" and "C" layers of the Tandjoeng-Enim coal deposits. D. J. W. KREULEN (Chem. Weekblad, 1933, 30, 718-722; cf. B., 1933, 898).-The temp. at which the rate of loss of volatile matter (V) is a max. is the only property which varies regularly with V%. All other properties, such as the heat of combustion, are governed primarily by the degree of carbonisation of the polymerised (alkaliinsol.) humic acids of the coal, although the carbonisation of the free humic acids is also of importance in certain cases. The variation of the activity of the cokes produced from the different coals is discussed. The results of applying the methods of examination used for the Tandjoeng-Enim coals to 4 samples of German H. F. G. coal are given.

Coke formation. X. Plasticity of coal. R. G. DAVIES and R. A. MOTT (Fuel, 1933, 12, 371-382; cf. B., 1933, 993) .- The solidification temp. (T) with the coal free to expand, *i.e.*, the temp. at which on release of the plunger, previously clamped, no penetration of the coal occurred, varied from 408° to 443° for a series of coals, and agreed with the temp. of max. expansion for nearly all coals of which the swelling was < 100%. The measurement of plasticity is discussed; resistance to gas passage (cf. Foxwell, J.S.C.I., 1921, 40, 193 T) is not an accurate measure of the degree of plasticity of the heated coal. The swelling pressure (P), *i.e.*, the pressure required to maintain the charge at const. vol., and T under these conditions have been determined for some coals in an apparatus consisting of a steel tube within which sides a plunger which could be weighted and allowed a clearance of  $\frac{1}{64}$  in. for the escape of the volatile products of carbonisation. The max. P vals. (rate of heating 1°/min.) varied from 400 to 800 lb./sq. in., being, in general, higher the better was the coking coal. Whereas with coals free to expand there was a steady increase in the vol. of gas evolved

with rise of temp. above 350°, with the coals prevented from expanding there was little evolution of gas until about 500°, when there was a sudden release over a narrow temp. range. The temp. at which this occurred agreed with the so-called temp. of max. plasticity obtained by Foxwell's method. The calc. pressures due to the imprisoned gas are as high as 5000 lb./sq. in. It is concluded that gas pressure is the primary cause of plasticity. An attempt has been made to determine the vol. of open and closed pores in coke made at const. vol.; it has been shown, however, that the % of closed pores in a coke prepared at 450—500° and cooled is not the same as in the product as it exists before cooling. A. B. M.

[Determination of volatile matter in coal etc. Improved burette.] H. A. J. PIETERS (Chem. Weekblad, 1933, 30, 722—723).—A burner of which the height can be varied by means of a screw device is described; by its use the distance between the top of the burner and the crucible can be adjusted to a const. val. By means of a small glass gas regulator the gas pressure can be maintained const. at a predetermined val. A burette is described in which an upper bulb contains 10, 20, or 30 c.c., whilst the lower part, on which the reading is taken, consists of graduated smallbore tubing; it may be employed for determinations of d. H. F. G.

Sulphur-containing substances in liquid products of Ukrainian brown coals. N. N. ORLOV and P. T. BONDARTSCHUK (J. Appl. Chem. Russ., 1933, 6, 903—909).—The benzine (I) and kerosene (II) fractions contain  $3\cdot5-5\%$  S, represented by S, H<sub>2</sub>S, and mercaptans [13% in (I), 28% in (II)], thiophen and its homologues [77% in (I), 62 $\cdot5\%$  in (II)], and sulphides [5—12% in (I) and (II)]. Disulphides are absent from (I) and (II). R. T.

Calculation of the calorific value of fuels from the ultimate analysis. R. VONDRÁCEK (Brennstoff-Chem., 1933, 14, 404—405).—A better general agreement between calc. and observed calorific vals. for a wide range of fuels is obtained with Vondrácek's formula (B., 1927, 179) than with Dulong's or with that due to Grumell and Davies (B., 1933, 610). In particular, the latter gives low vals. for fuels of high O content, e.g., wood or peat. For fuels of high C content Dulong's formula is the most suitable. It is recommended that when the difference between the calc. and observed vals. is > 50kg.-cal. the determination should be repeated.

A. B. M.

Calculating the calorific value of solid fuels. A. J. V. UNDERWOOD (Gas World, 1933, 99, 528).—A nomogram, based on the Grumell-Davies formula (B., 1933, 610), is given. D. K. M.

Behaviour of cokes in the open grate. E. I. LLOYD, F. BELL, and H. J. HODSMAN (Gas J., 1933, 204, Suppl., 12—16).—The behaviour of cokes when consumed in the open grate has been correlated with the properties of the cokes capable of measurement in the laboratory, e.g., reactivity to  $CO_2$  and to  $O_2$ , ignition temp., ash content, and bulk d. The ignitability of a fuel depends on its structure and is hindered by increase of d. The hourly output of heat by radiation is influ-

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enced also by the form of the pieces. Both structure and form influence the bulk d of a fuel, which is therefore important in determining the behaviour of a hightemp. coke in the open grate. A method of testing the behaviour of cokes in the open grates is described. An approx. relationship between the properties of a coke and its rate of output of radiation is advanced. The ignitability of cokes, especially from continuous vertical retorts, can be increased by the application of a skin of coal. H. J. H.

Determination of small amounts of nitric oxide in, and its removal from, coke-oven gas. A. GUYER and R. WEBER (Brennstoff-Chem., 1933, 14, 405-408; cf. B., 1933, 257).-The gas (20-30 litres/hr.) is passed through aq. KMnO4 acidified with H2SO4 to oxidise the NO to NO<sub>2</sub>, which is then determined colorimetrically by absorption in Griess' reagent  $[\alpha - C_{10}H_7 \cdot NH_2 +$  $m-C_6H_4(NH_2)$ ·SO<sub>3</sub>H in aq. AcOH]. The method is more rapid than that in which the NO is oxidised by means of air or O<sub>2</sub>. Fe sulphide or oxide (the latter being converted into sulphide by the H<sub>2</sub>S in the gas) is an efficient absorbent of NO from coke-oven gas. It is estimated that with an NO content < 2-5 p.p.m. no separation of NO, will occur with the pentadiene fraction when the gas is fractionated by the low-temp. process of Bronn and Linde, Claude, etc., and therefore no trouble will arise through the formation of gum or explosive compounds. This concn. car readily be attained by passing the gas over Fe sulphide. A. B. M.

Determination of air in technical acetylene. S. J. MILOSLAVSKI and D. L. GLIZMANENKO (J. Appl. Chem. Russ., 1933, 6, 963—969).—Known methods and apparatus are described. R. T.

Viscosity of pitch. A. B. MANNING (Dept. Sci. Ind. Res., Fuel Res. Tech. Paper No. 39, 1933, 20 pp.) .--The abs. viscosities  $(\eta)$  of low-temp. (A), vertical-retort (B), horizontal-retort (C), and coke-oven pitches (D)have been determined over the temp. range 30-110°. Three methods were used : (a) depending on measuring the flow under load of a flat disc of the material, and suitable when  $\eta = 10^8 - 10^9$  poises, (b) using a rotatingcylinder viscosimeter ( $\eta = 10^4 - 10^9$  poises), and (c) using a capillary-tube viscosimeter ( $\gamma = 10 - 10^4$  poises). The  $\eta$  of all the samples decreased rapidly with rise of temp., e.g., that of C fell from  $2 \cdot 3 \times 10^9$  poises at  $30^\circ$  to  $30 \cdot 2$ poises at 110°. The slope of the log.  $\eta$ -temp. curve for C or D was somewhat > that for A, the curve for B having an intermediate slope. The twist points, determined by Taylor's method (J.S.C.I., 1926, 45, 417 T), and the softening points, by the ring-and-ball method corresponded approx. with  $\eta$  10<sup>7</sup> and 10<sup>4</sup> poises, respectively; their vals., however, were functions of the thermal diffusivities of the material as well as of its  $\eta$ . All the pitches behaved as true viscous liquids in that, within the limits of experimental error,  $\eta$  was independent of the rate of shear to which the material was subjected. A sample of bitumen (petroleum pitch), however, was found to possess anomalous viscous properties, the observed  $\eta$  decreasing with increase in the rate of shear. The  $\eta$  of the bitumen, measured at an arbitrary rate of shear, had a much lower temp. coeff. than that of the coal-tar pitches. A. B. M.

Identification and composition of wood tars. A. P. SNESAREV (J. Appl. Chem. Russ., 1933, 6, 915– 921).—Tars from birch bark, red beech, and white beech contained respectively:  $H_2O \ 2 \cdot 0$ ,  $9 \cdot 8$ ,  $18 \cdot 8$ ;  $H_2O$ -sol. acids  $3 \cdot 1$ ,  $2 \cdot 1$ ,  $6 \cdot 1$ ; light oils (b.p. 91— $160^\circ$ )  $5 \cdot 0$ ,  $5 \cdot 0$ ,  $2 \cdot 0$ ; heavy oils (b.p. 160— $270^\circ$ )  $28 \cdot 0$ ,  $24 \cdot 0$ ,  $14 \cdot 4$ ; pitch 60, 55, 48; and phenols  $2 \cdot 7$ ,  $21 \cdot 2$ ,  $10 \cdot 4\%$ . R. T.

Behaviour of low-temperature tar on dialysis. G. FREE (Brennstoff-Chem., 1933, 14, 401-404).--A low-temp. tar, in glacial AcOH solution, has been fractionated by dialysis through parchment. The C and H contents of the fractions showed no regular variation, whereas the O + N content diminished and the calorific val. rose as dialysis continued.  $\eta$  rose to a max. and then fell again. A small proportion of each fraction was insol. in Et<sub>2</sub>O and was examined separately. The dialysates were further subjected to fractionation by means of solvents (cf. Keppeler and Hoffmann, B., 1933, 48). The products of greater mol. vol., *i.e.*, slower rate of diffusion, were present mainly in the benzine-sol. fractions (I), and those of lower mol. vol. in the C<sub>6</sub>H<sub>6</sub>-sol. fractions. The bases, phenols, and paraffin wax were determined in (I). The phenol content was highest in the first dialysate and decreased as dialysis continued. The paraffin wax was practically completely held back by the parchment membrane. The val. of dialysis as a method of studying the composition of tar is discussed. A. B. M.

Composition of petroleums. A. SACHANEN and R. WIRABIANZ (Erdöl u. Teer, 1933, 9, 170–172, 187–189, 202–203, 220–222; Chem. Zentr., 1933, ii, 310).—Results of determinations of aromatic (I), naphthene (II), and paraffin hydrocarbons for oils from Grozni, Baku, Emba, Kaluga, and Perm are tabulated. The (I) in the fractions of higher b.p. are chiefly di- and tri-cyclic; the (II) of high b.p. (>550°) contain also tetracyclic and even more complex substances. Lubricating properties are discussed in relation to complexity and b.p. A. A. E.

Hydrogenation of petroleum. II, III. C. E. N. DE ARAUJO, JUN. (Chimica, 1933, 1, 219–225, 243– 244; cf. B., 1933, 901).—A brief review.

Determination of water in hydrocarbons. H. BROCHE and W. SCHEER (Brennstoff-Chem., 1933, 14, 408—409; cf. B., 1932, 872).—The method described previously can conveniently be modified by enclosing the CaH<sub>2</sub> in a capsule made of material impermeable to H<sub>2</sub>O vapour but sol. in C<sub>6</sub>H<sub>6</sub>; for this purpose the artificial resin "trolitul," a polystyrene, is suitable. The method can be applied to solid materials by distilling them with dry xylene and determining the H<sub>2</sub>O in the distillate. Attention is directed to the necessity of correcting for fluctuations of atm. temp. and pressure, taking into account the v.p. of the hydrocarbon, if high accuracy is desired. A. B. M.

Problems of technical reactions. II. Separation of carbon compounds (cracking process; sugar degradation). O. SCHMIDT (Z. Elektrochem., 1933, 39, 969-981).—A review.

Application of Raman spectrography to the study of mineral oils. A. ANDANT (Chim. et Ind.,

1933, **30**, 1011—1019).—Curves and Raman spectral photographs are given, from which the constituent hydrocarbons in the distillation fractions of 5 natural petrols were determined. N. M. B.

tish Gaemical Abstracts-B.

Improved lamp for determination of sulphur in light petroleum products. E. R. GILLIS (Ind. Eng. Chem. [Anal.], 1933, 5, 421).—A more easily controlled modification of the standard (A.S.T.M.) lamp is described. R. S. C.

Modern methods of refining lubricating oils by solvent extraction. K. FÜRST (Petroleum, 1933, 29, No. 44; Motorenbetr., 6, 2—6).—The requisite properties of suitable solvents for the purpose are discussed and an account is given of results which have recently been obtained in the technical application of the following solvents: (a) liquid  $SO_2 + C_6H_6$ , (b) PhNO<sub>2</sub>, (c) PhOH, and (d)  $\beta\beta'$ -dichlorodiethyl ether ("chlorex"). Treatment of the oils with these solvents effects a marked rise in the viscosity index, lowers the C residue (Conradson), and also improves their other physical characteristics. A. B. M.

Degree of solvation [of lubricating oils in organic solvents]. (A) A. RABL, (B) E. L. LEDERER, (c) A. RABL (Petroleum, 1933, 29, No. 44; Motorenbetr., 6, 6-8).-(A) The assumptions and methods of calculation used by Lederer (I.P.K. Druckschrift, No. 18,664, 1932) are adversely criticised. (B, c) Polemical.

A. B. M. Effect of presence of graphite on lubricating properties of oils. A. BOUTARIC and R. AMIOT (Ann. Office nat. Combust. liq., 1933, 8, 129–131; Chem. Zentr., 1933, ii, 313).—The  $\eta$  and surface tension of oils are scarcely affected by addition of small quantities of graphite (4% Castordag or Oildag). Although measurements of interfacial tension between graphitised oil and Hg indicate greater adhesion of the unimol. oil film to the metal surface, centrifuging oiled plates shows that adhesion of a thick film is  $\gg$  normal. A. A. E.

Passau graphite for refractories.—See VIII. Mastic asphalt roofing.—See IX.

See also A., Dec., 1253, Incomplete oxidation of  $CH_4$  with O<sub>2</sub> and air. 1290, Components of petroleum. 1305, N compounds in petroleum distillates.

## PATENTS.

Removal of impurities from coal. W. R. CHAPMAN (B.P. 400,644, 23.4.32).—Coal which contains very finelydivided clay etc. is pre-washed with  $H_2O$ , whereby the fine impurities are removed in suspension in the wash- $H_2O$ . The coal may be first washed in a mixing box and then passed on to a fine screen where it is sprayed with  $H_2O$ . A. B. M.

Conversion of distillates from coal and the like into products of lower b.p. A. G. L. TRY and M. STUART (B.P. 400,604, 23.2.32. Addn. to B.P. 363,360; B., 1932, 217. Cf. B.P. 400,234; B., 1934, 8).— The process of the prior patent is applied, not to the entire distillate, but only to a fraction thereof.

A. B. M.

Influencing the processes of combustion in the hearths of shaft furnaces. M. KÜNKELE (B.P. 400,793, 28.12.32. Ger., 28.12.31).—The shape and

extent of the oxidation spaces in front of the tuyères (T) of a blast or other shaft furnace is varied according to the working conditions by means of deflecting bodies (D) disposed within T and capable of axial displacement therein. E.g., D may be conical and mounted on a guide rod with the base directed towards the mouth of T.

A. B. M.

Hydrogenation of carbonaceous materials. GAS LIGHT & COKE CO., R. H. GRIFFITH, R. N. B. D. BRUCE, and S. G. HILL (B.P. 400,628, 27.4.32).—The initial material, e.g., creosote oil, gas oil, or low-temp. tar oil, is treated with H<sub>2</sub> at, e.g.,  $460-480^{\circ}/200$  atm., in presence of a catalyst consisting of basic Ti phosphate, vanadate, chromate, molybdate, or tungstate. The catalyst may be made into pellets, either alone or mixed with clay or other binder. The process is carried out continuously. The initial material is converted into a product containing 35-60% of spirit, b.p. < 180°.

A. B. M.

Treatment of carbonaceous materials with hydrogenating gases. I. G. FARBENIND. A.-G. (B.P. 400,844, 29.3.33. Ger., 26.8.32).—Carbonaceous materials containing asphalt (I) are destructively hydrogenated in the presence of a suitable catalyst which has previously been dispersed in an oil which is free from (I) and on admixture with the material to be treated does not cause flocculation of (I) therein. A. B. M.

Heating of coke ovens and the like. J. BECKER, Assr. to KOPPERS CO. OF DELAWARE (U.S.P. 1,898,970, 21.2.33. Appl., 22.1.27).—The gases for the purpose, whether partly purified coal gas or any other gas, are heated to 30—60° before entering the oven structure, to prevent condensation and consequent corrosion.

By-product coke oven. C. H. HUGHES, ASST. to SEMET-SOLVAY ENG. CORP. (U.S.P. 1,899,275, 28.2.33. Appl., 13.8.26).—Each heating wall between the coking chambers has two series of horizontal zig-zag flues connected by a cross-over so that there are only two inlet-outlets to reversing regenerators below; the latter are so arranged that there is one to each retort, the positive and negative functions alternating along the row. B. M. V.

Gas generators. F. C. WHITE (B.P. 400,727, 4.7.32).—In the type of generator having a fuel-supply shaft and a converter shaft both superposed on a grate chamber (cf. B.P. 201,385; B., 1923, 918 A), a chamber wherein steam is either produced or superheated is provided in the generator structure in a position to intercept heat from the grate chamber which would otherwise pass to the exterior. A. B. M.

Gas generator. W. I. BATTIN and C. S. CHRISMAN, Assrs. to U.G.I. CONTRACTING CO. (U.S.P. 1,897,950, 14.2.33. Appl., 25.7.27).—The lower part of the inner wall of the generator is formed of metallic blocks cooled by vertical  $H_2O$ -tubes forming part of a boiler.

B. M. V.

Manufacture of water-gas. POWER-GAS CORP., LTD., N. E. RAMBUSH, and J. M. BALLINGALL (B.P. 400,857, 22.4.33).—A water-gas plant, employing upand down-stream runs, is provided with H<sub>2</sub>O-sealed gas valves the actuating device of which is connected with the steam valves in such a manner that the supply of steam and the consequent production of gas is stopped before the gas valve change-over is commenced, and started again when it is completed. A. B. M.

Absorption of constituents from gases and vaporous mixtures. C. D. GARD, Assr. to UNION OIL CO. OF CALIFORNIA (U.S.P. 1,898,579, 21.2.33. Appl., 30.5.30).—In, *e.g.*, the recovery of petrol from natural gas, the gases are scrubbed with clean absorption oil, a part of which is stripped by heating, cooled, and returned to the absorber; the other portion is, in addition, distilled to remove accumulated high-boiling matter. B. M. V.

Manufacture of stable fluid emulsions. INTER-NAT. BITUMEN EMULSIONS CORP. (B.P. 401,131, 28.12.32. U.S., 1.11.32).—Aq. emulsions of tar, pitch, oils, etc. stabilised with alkali caseinate are partly neutralised with acid ( $H_3PO_4$ ,  $H_3BO_3$ ), e.g., to  $p_H 8.5$ , to prevent deterioration of the casein. A preservative, e.g., CH<sub>2</sub>O, may also be added. C. H.

Treatment of hydrocarbon oils. J. C. MORRELL and G. EGLOFF, Assrs. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,897,582, 14.2.33. Appl., 29.1.31).—A purifying process especially suitable for petrols or similar distillates comprises treating the heated vapour with SO<sub>3</sub>, steam, and  $Fe_2(SO_4)_3$ . B. M. V.

Treatment of hydrocarbon distillates. R. C. OSTERSTROM, ASST. to PURE OIL CO. (U.S.P. 1,897,328, 14.2.33. Appl., 19.4.29).—Distillates, especially heavy fractions of cracked vapours, are mixed with a catalyst (e.g., fuller's earth) and the mixture is pumped at > 200 lb./sq. in. to a heater affording a temp.  $> 260^{\circ}$ ; the pressure is then released and the resulting vapours are freed from the catalyst, rectified in a tower, and passed through a bed of catalyst before condensation.

B. M. V.

Treatment of petroleum distillates. L. A. MEK-LER, ASST. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,897,617, 14.2.33. Appl., 18.11.29).—The decomp. of mercaptans therein by X-rays, assisted if desired by oxidation, is claimed. B. M. V.

Preparation of synthetic lubricant. J. J. GREBE and S. M. STOESSER, ASSTS. to DOW CHEM. Co. (U.S.P. 1,905,850, 25.4.33. Appl., 25.4.30).—Ph<sub>2</sub>O alone or with aromatic hydrocarbons or their oxygenated derivatives is heated at  $650-750^{\circ}$  or at  $400^{\circ}$  up to the decomp. point in presence of a metallic oxide catalyst (Al<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>-CuO) in a closed vessel. The fraction of the product b.p. 200—350°/5—10 mm. (about 15% at equilibrium) is claimed as a high-temp. lubricating oil. H. A. P.

Pulveriser [for coal].—See I. Hydrogenation (etc.) of hydrocarbons.—See III. NH<sub>3</sub> oxidation.— See VII.

# III.—ORGANIC INTERMEDIATES.

Recovery of carbon tetrachloride. W. L. MEN-DENHALL, C. W. MCCLURE, and M. HUNTSINGER (Science, 1933, 78, 482–483).—The CCl<sub>4</sub> is distilled into Hg covered by ice cubes. L. S. T.

Conversion of acetylene and acetaldehyde into acetone by the contact method. N. D. ZELINSKI,

B. M. V.

M. I. USCHAKOV, V. M. MICHAILOV, and J. A. ARBUZOV (J. Chem. Ind. Russ., 1933, 10, No. 7, 63-65).-96% yields of COMe<sub>2</sub> are obtained by passing MeCHO over FeO-MnO catalyst at 400°. Under similar conditions,  $1:10 C_2H_2$ -H<sub>2</sub>O mixtures give 88%, or, if the issuing gases, containing 4% of C<sub>2</sub>H<sub>2</sub>, are again passed over the catalyst, 98% yields of COMe<sub>2</sub>, according to the reaction  $2C_2H_2 + 3H_2O \rightarrow COMe_2 + CO_2 + 2H_2$ . R. T.

Hydrogenation of oxygen-containing compounds. I. Catalytic reduction of phenol and o-cresol. B. L. MOLDAVSKI and S. E. LIVSCHITZ (J. Gen. Chem. Russ., 1933, 3, 603-614).-90% yields of  $C_6H_6$  or PhMe are obtained by heating PhOH or o-cresol with MoS<sub>2</sub> catalyst at 350° under a partial H<sub>2</sub> pressure of 26 atm.; at higher H<sub>2</sub> pressures further hydrogenation to cyclohexene and -hexane, p-cyclohexyl-phenol and -benzene, or to the corresponding o-Me derivatives takes place. R. T.

Hydrogenation of some carboxylic acids. C. M. CAWLEY (Fuel, 1933, 12, 366-370; cf. B., 1933, 260).-The experiments were carried out in a rotating mild-steel converter of 2 litres capacity. At 450° and 100 atm. initial pressure of H<sub>2</sub>, after heating for 2 hr., BzOH [or  $Fe(OBz)_2$ ] was converted into  $C_6H_6$  (24%), PhMe (41%), and a small amount (2%) of a high-boiling neutral oil. The reaction commenced between 300° and 350°; at these temp.  $(CH_2Ph)_2$  was also formed (13-17%). Under the above experimental conditions NaOBz was incompletely converted into C<sub>6</sub>H<sub>6</sub> (15%), PhMe (5%), and  $CH_2Ph_2$  (4-8%).  $COPh_2$  was partly converted into  $CH_2Ph_2$  (about 40%), with small amounts of  $C_6H_6$ and PhMe. Salicylic acid yielded mainly PhOH (57%), and phthalic anhydride mainly C6H6 (13%) and PhMe (35%). The effect of an NH<sub>4</sub> molybdate-charcoal catalyst, either alone or with S, was small; only with salicylic acid was the catalyst effective in hydrogenating the aromatic nucleus. A. B. M.

S-containing substances from brown coal. Determining air in  $C_2H_2$ . Wood-tar constituents.— See II. Furfuraldehyde from flax refuse.—See V. Textile penetrants.—See VI. Prep. of pentryl.— See XXII.

See also A., Dec., 1249, Autoxidation of aldehydes. 1253, Incomplete oxidation of  $CH_4$  with  $O_2$  and air. 1254, Electrochemical reduction of  $C_5H_5N$ . 1266, Sol. anhydrite as desiccating agent. 1274, Prep. of esters. 1276, Prep. of cryst. lactic acid. 1280, Prep. of NH<sub>2</sub>Ac and NH<sub>2</sub>Bz. 1283, Syntheses in the Ph<sub>2</sub> and C<sub>6</sub>H<sub>4</sub>Ph<sub>2</sub> series. 1284, Prep. of 1 :5- and 1 :8-C<sub>10</sub>H<sub>6</sub>(NO<sub>2</sub>)<sub>2</sub> and 5-nitro- $\alpha$ -naphthylamine. 1294, Prep. of Ph<sub>2</sub> derivatives. 1299, Prep. of diaminoanthraquinones. 1305, N compounds in petroleum distillates. 1333, Bacterial formation of CH<sub>4</sub>.

#### PATENTS.

Hydrogenation or dehydrogenation of hydrocarbons. W. F. HUPPKE and F. E. FREY, Assrs. to PHILLIPS PETROLEUM CO. (U.S.P. 1,905,383, 25.4.33. Appl., 22.7.30).—Cr<sub>2</sub>O<sub>3</sub>,nH<sub>2</sub>O gel, prepared by pptn. of Cr salts with NaOH or KOH or by aq. NH<sub>3</sub> in presence of AcOH, H<sub>2</sub>SO<sub>4</sub>, Al salts, or sol. silicates, is used as catalyst at 325—550°. H. A. P. Stabilisation of chlorinated aliphatic hydrocarbons. Dow CHEM. Co. (B.P. 401,210, 19.5.33. U.S., 22.6.32).—CCl<sub>4</sub>, C<sub>2</sub>Cl<sub>4</sub>, CHCl<sub>3</sub>, C<sub>2</sub>HCl<sub>3</sub>, etc. are stabilised by addition of, *e.g.*, 0.001-0.1 vol.-% of a mercaptan, *e.g.*, BuSH. C. H.

Concentration of formaldehyde solutions. H. A. BOND, ASST. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,905,033, 25.4.33. Appl., 9.12.27).—Dil. aq.  $CH_2O$  is distilled at > 1 atm. (4 atm.). The column may contain Cu to depolymerise ( $CH_2O$ )<sub>x</sub>. Apparatus is claimed, by suitable operation of which solutions up to 62 wt.-% or gaseous  $CH_2O$  are obtained. H. A. P.

Production of concentrated aliphatic [acetic] acids. H. DREYFUS (B.P. 400,944, 1.4.32).—The acid is extracted with a mixture of a low-boiling solvent (< 50%), preferably one forming with H<sub>2</sub>O an azeotropic mixture, and a high-boiling O-containing org. compound, *e.g.*, EtOBz, Me<sub>2</sub> or Et<sub>2</sub> phthalate, CH<sub>2</sub>Ph·OBz, COPhMe, etc. Suitable solvents are EtOAc, HCO<sub>2</sub>Bu, COMEEt. C. H.

**Production of acetone.** H. WALTER and H. SCHULZ, Assrs. to DEUTS. GOLD- U. SILBER-SCHEIDEANSTALT **VORM.** ROESSLER (U.S.P. 1,892,742, 3.1.33. Appl., 19.2.31. Ger., 26.1.28).—AcOH,  $C_2H_2$ , MeCHO, or EtOAc and steam are passed over active C and a catalyst comprising the oxide or acetate of Fe, Ca, Zn, Ce, or Th (ZnO) at 400—500°. The catalyst chamber is of Cu or its alloy with Zn, Sn, Ag, Ni, Al, or steel containing Ni and approx. 18% of Cr. An apparatus is claimed in which fresh catalyst is continuously fed into, and spent catalyst extracted from, the reaction chamber.

H. A. P.

[Preparation of] salts of organic dithiophosphates. L. J. CHRISTMANN, ASST. to AMER. CYANAMID Co. (U.S.P. 1,893,018, 3.1.33. Appl., 22.10.28).—Alkali, alkaline-earth, or  $NH_4$  dithiophosphates (from  $C_2$ — $C_8$  alcohols and  $P_2S_5$ ) are claimed as froth-flotation agents. H. A. P.

Manufacture and use of washing, cleansing, emulsifying, dispersing, wetting-out, or like agents. DEUTS. HYDRIERWERKE A.-G. (B.P. 400,986, 4.5.32. Ger., 18.5.31).—Unsaturated aliphatic alcohols of  $C_8$ — $C_{20}$  are sulphonated and halogenated. C. H.

Manufacture of sulphuric acid derivatives of fatty esters and thioesters [wetting, cleansing, and foaming agents]. HENKEL & Co. G.M.B.H. (B.P. 401,116, 21.11.32. Ger., 5.12.31).—Ethers or thioethers of hydroxy- or thiol-aliphatic esters, containing OH or aryl groups or double linkings, are sulphonated. Examples are :  $C_{12}H_{25}$ ,  $C_{18}H_{37}$ , and oleyl phenoxyacetates ; oleyl ethoxyacetate and thiodiglycollate ;  $C_{12}H_{25}$  $\beta$ -hydroxyethoxyacetate, etc. C. H.

Manufacture of esters, including thioesters, substituted in the acid residue. HENKEL & Co. G.M.B.H. (B.P. 401,117 and Addn. B.P. 401,120 [A] 25.11.32, [B] 1.12.32. Ger., [A] 5.12.31, [B] 16.12.31).— (A) Ethers or thioethers of hydroxy- or thiol-aliphatic acids are esterified with aliphatic alcohols  $\leq C_8$  or with naphthenic or resin alcohols. Examples are phenoxyacetates of  $C_{12}H_{25}$  (m.p. 31°),  $C_{18}H_{37}$  (m.p. 55°),  $C_8H_{17}$ ,  $C_{16}H_{35}$  (m.p. 47°),  $C_{14}H_{29}$  (m.p. 39°), oleyl and coconut oil alcohols; thiodiglycollates of  $C_{12}H_{25}$ ,  $C_8H_{17}$ , and

oleyl alcohols; oleyl cyclohexyloxyacetate, b.p. 225– 235°/2 mm. (B) Aliphatic alcohols or mercaptans  $\langle C_6$  are esterified with fatty acids carrying OR or SR groups (R = alkyl, aryl, aralkyl, or hydroaryl), with or without other substituents. Examples are Bu  $\alpha$ -phenoxylaurate and Et  $\alpha$ -phenylthiolstearate. C. H.

Manufacture of high-molecular sulphur compounds. HENKEL & Co. G.M.B.H. (B.P. 401,118, 25.11.32. Ger., 15.12.31 and 25.1.32).—Sulphuric or phosphoric esters of aliphatic, hydroaromatic, naphthenic, or resin alcohols  $> C_5$  are heated with H<sub>2</sub>O-sol. hydrosulphides, sulphides, or polysulphides, or with mono- or poly-hydric thiol compounds, particularly thiophenols or heterocyclic thiols. The following products are described : C<sub>12</sub>H<sub>25</sub>·SH, b.p. 142—145°/15 mm.; p-C<sub>6</sub>H<sub>4</sub>Me·S·C<sub>12</sub>H<sub>25</sub>, m.p. 29·6°; C<sub>12</sub>H<sub>25</sub>·S·CH<sub>2</sub>·CO<sub>2</sub>Na; C<sub>12</sub>H<sub>25</sub>·S·CH<sub>2</sub>Ph, m.p.  $< 16^{\circ}$ . [Stat. ref.] C. H.

Preparation of higher ethers. HENKEL & Co. G.M.B.H. (B.P. 401,142, 3.2.33. Ger., 26.3.32).—Sulphuric or phosphoric esters of aliphatic or hydroaromatic alcohols > C<sub>7</sub> are heated with alcohols or phenols in alkaline medium or with alkoxides or phenoxides. Examples are : OH·C<sub>2</sub>H<sub>4</sub>·O·C<sub>12</sub>H<sub>25</sub>, b.p. 170—174°/15 mm.; mono-C<sub>12</sub>H<sub>25</sub> (b.p. 210—215°/15 mm.), -C<sub>10</sub>H<sub>21</sub> (b.p. 198°/3 mm.), -C<sub>14</sub>H<sub>29</sub> (b.p. 206—207°/3 mm.), -C<sub>16</sub>H<sub>33</sub> (b.p. 205—206°/1 mm.), -C<sub>18</sub>H<sub>37</sub> (b.p. 215—220°/2 mm.), -C<sub>8</sub>H<sub>17</sub> (b.p. 135—137°/2 mm.), -C<sub>10</sub>H<sub>21</sub> (b.p. 159—161°/2 mm.), -naphthenyl, and ω-methoxy-decyl ethers of glycerol; C<sub>12</sub>H<sub>25</sub>·OPh, m.p. 25°; bis-βγ-dihydroxypropyl ether of αω-decylene glycol. C. H.

Production of  $1[\alpha] - p$  - aminophenyl -  $2[\beta]$  methylaminopropanol - 1. M. OBERLIN (U.S.P. 1,892,532, 27.12.32. Appl., 19.7.30. Ger., 7.8.29).— NHMe·CHMe·CHPh·OH (I) is nitrated (1H<sub>2</sub>SO<sub>4</sub>,  $d \cdot 84 : 1$ HNO<sub>3</sub>,  $d \cdot 4$ ,  $\leq 30^{\circ}$ ), and the p-NO<sub>2</sub>-compound, m.p. 105—107° (nitrate, m.p. 171—173°; hydrochloride, m.p. 266—268°), reduced (Pd-H<sub>2</sub>, SnCl<sub>2</sub>, Na-Hg and HCl) to the p-NH<sub>2</sub>-compound, m.p. 114—116° (hydrochloride, decomp. 176—178°), which is claimed to have the physiological effects of (I) but to be less toxic. H. A. P.

Manufacture of condensation products [of phenols with unsaturated oils]. I. G. FARBENIND. A.-G. (B.P. 401,237, 6.7.33. Ger., 7.7.32).—Phenols or their derivatives (e.g., alkyl ethers) are allowed to interact with unsaturated fatty acids, their esters, or glycerides (e.g., olive oil, linseed oil, stand oil) in presence of BF<sub>3</sub>. The reaction occurs with decrease of I val. and increase of Ac val. (of the oil). The products are viscous liquids to adhesive or rubber-like masses. H. A. P.

Production of esters. T. S. CARSWELL, E. T. STEHLBY, and N. J. G. ALOZERIJ, ASSTS. to MONSANTO CHEM. WORKS (U.S.P. 1,905,144, 25.4.33. Appl., 6.8.28).—Approx. equimol. amounts of a benzenemono-carboxylic acid non-volatile at  $< 120^{\circ}$  (o-OH·C<sub>6</sub>H<sub>4</sub>·CO<sub>2</sub>H) and a H<sub>2</sub>O-miscible alcohol, b.p.  $< 100^{\circ}$ , are distilled, the distillate is continuously fractionated to remove H<sub>2</sub>O, and the alcohol returned to the reaction mixture. Apparatus is claimed. H. A. P.

Preparation of substitution products of aminobenzoyl-o-benzoic acid. E. T. Howell and I. GUBELMANN, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,892,890, 3.1.33. Appl., 2.7.28; cf. B., 1931, 290).—4'-Amino-2-benzoylbenzoic acid is converted into hydrolysable N-acyl or N-alkyl- or -aryl-sulphonyl derivatives, convertible by hot conc.  $H_2SO_4$  into 2-amino-anthraquinone. The Ac, m.p. 277°, CHPh., m.p. 215—219°, and phthaloyl, m.p. 294°, derivatives are described. H. A. P.

Purification of non-volatile organic acids. A. O-JAEGER, Assr. to SELDEN Co. (U.S.P. 1,892,396, 27.12.32-Appl., 7.9.28).—Non-volatile org. acids which form volatile anhydrides are separated from volatile impurities (e.g., phthalic acid from BzOH; naphthalic acid from oxidation products of acenaphthene) by distillation with steam at a temp. < that at which the anhydride is formed. H. A. P.

Preparation of alkyl-substituted aromatic hydroxy-compounds. F. LINNER, Assr. to BECK, KOLLER & Co., INC. (U.S.P. 1,892,990, 3.1.33. Appl., 16.10.29. Austr., 27.6.29).—Phenols, naphthols, etc. are C-alkylated by action of an olefine and a little alkyl halide (EtCl) in presence of AlCl<sub>3</sub>, FeCl<sub>3</sub>, or ZnCl<sub>2</sub>. The method is applied to ethyl-, *iso*propyl-, m.p. 60°, *p-tert.*-butyl-, m.p. 96—98° (from CMe<sub>2</sub>:CH<sub>2</sub>), and *p-tert.*-amyl-phenol, m.p. 93—94°, *tert.*-butyl-β-naphthol, m.p. 155—160°, (?) 5-*iso*propyl-, b.p. 230—235°, and *tert.*-butyl-o-cresol, b.p. 245—250°, (?) 6-*iso*propyl-, b.p. 228—235°, and *tert.*-butyl-*m*-cresol, b.p. 222—226°, and *tert.*-amyl-*p*-cresol. H. A. P.

Production of *p*-cymene. I. W. HUMPHREY, Assr. to HERCULES POWDER Co. (U.S.P. 1,893,802, 10.1.33. Appl., 4.5.28).—A terpene, *e.g.*, dipentene, or pine oil is heated with a dehydrating catalyst (fuller's earth) at  $170-250^{\circ}$  for 10 hr., and the product distilled, the *p*-cymene being separated from the distillate, b.p.  $< 195^{\circ}$ , if desired, by sulphonation or nitration.

H. A. P. Preparation of nitro-derivative of [tert.-]butylcymene. H. BARBIER (U.S.P. 1,892,128, 27.12.32. Appl., 29.5.30. Ger., 12.4.30).—The  $3:5-(NO_2)_2$ -derivative, m.p. 132°, is produced by nitration of 1:2:4-  $C_8H_3MePr^{\beta}$ -CMe<sub>3</sub> (fuming HNO<sub>3</sub>  $< 0^\circ$ ). It has an odour of musk. H. A. P.

**Preparation of styrene.** G. L. DOROUGH, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,892,386, 27.12.32. Appl., 21.12.29).—PhEt is chlorinated at 1—10° in sunlight, the CHCIPhMe thus produced heated with  $C_5H_5N$  at 130—140°, and the resulting quaternary salt further heated at 275—300°. The method is applied also to a mixture of *o*- and *p*-C<sub>6</sub>H<sub>4</sub>Cl·CH:CH<sub>2</sub>, and to  $C_6H_4$ (CH:CH<sub>2</sub>)<sub>2</sub> (chlorination at 70—90°). H. A. P.

Purification of abietenesulphonic acid compounds. C. O. HENKE, ASST. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,892,889, 3.1.33. Appl., 25.5.31).—The (dil. neutral) aq. solution is freed from unsulphonated impurities by extraction with an inert solvent, e.g.,  $C_6H_6$ . H. A. P.

Isolation of ergosterol. A. ZIMMERLI, ASST. to ACETOL PRODUCTS, INC. (U.S.P. 1,893,317, 3.1.33. Appl., 28.4.30).—The mycelium of *Aspergillus*, *Penicillium*, and *Citromyces* (*Aspergillus niger* from citric acid manufacture) is extracted with EtOH and a little NaOH, the

extracts are evaporated, and the product is crystallised from  $\text{Et}_2\text{O}$ . H. A. P.

Production of chlorinated diaryls. R. L. JENKINS, Assr. to SWANN RESEARCH, INC. (U.S.P. 1,892,397, 27.12.32. Appl., 28.9.29).—An apparatus is claimed in which  $\text{Cl}_2$  bubbling through  $\text{Ph}_2$  or homologue in contact with an Fe catalyst induces circulation from top to bottom of the column through an external annular space. Uniform increase in d and  $\eta$  up to approx. 66% Cl is claimed. H. A. P.

[Manufacture of] halogenated dihydroxydiphenyls [bactericides]. F. W. NITARDY, W. G. CHRISTIANSEN, and S. E. HARRIS, ASSTS. to E. R. SQUIBB & Sons (U.S.T. 1,892,000, 27.12.32. Appl., 7.10.31).— Mono- and di-halogenodihydroxydiphenyls in which all the substituents are in the same nucleus are prepared by treating homonuclear (3:4- and 2:5-)dihydroxydiphenyls with halogenating agents (SO<sub>2</sub>Cl<sub>2</sub>, Br).

H. A. P.

[Vapour-phase] oxidation of fluorene [to fluorenone]. A. O. JAEGER, Assr. to SELDEN Co. (U.S.P. 1,892,768, 3.1.33. Appl., 31.10.28).—Fluorene or a derivative, an oxidising gas (air), and excess of steam are passed over  $V_2O_5$ , Fe pyrovanadate, or similar derivative of an element of group V or VI preferably present in a base-exchange material in non-exchangeable form, at 300—500°. Maleic and phthalic anhydrides, if formed, are removed by cooling the oxidised gases to just  $< 190^\circ$ . H. A. P.

Preparation of organic compounds of anthraquinone series. P. H. GROGGINS (U.S.P. 1,892,302, 27.12.32. Appl., 30.6.31).—Chloro- and bromo-anthraquinones or their (NO<sub>2</sub>-)derivatives are heated with aq. NH<sub>3</sub> under pressure (at 150—225°) in presence of an inorg. nitrate, and a chlorate, perchlorate, or other oxidising agent, and in presence or absence of a metal following H in the electrochemical series (Cu, Cu<sub>2</sub>O, Ag salts), to produce  $\alpha$ - or  $\beta$ -aminoanthraquinone.

H. A. P.

Manufacture of benzanthrone. G. BACHARACH and E. MCCAULIFF, JUN., Assrs. to RADIO PATENTS CORP. (U.S.P. 1,893,575, 10.1.33. Appl., 2.5.30).—A mixture of glycerol, anthraquinone,  $H_2SO_4$ , and  $Ac_2O$  is heated at 150°. The  $Ac_2O$  is claimed to moderate the reaction, prevent charring, and give a higher yield (80.5%) of a purer product. H. A. P.

Synthetic lubricant.—See II. PCl<sub>5</sub> [for prep. of acyl chlorides].—See VII.

#### IV.—DYESTUFFS.

Chemical resistance of materials in the manufacture of sulphur dyes. G. E. BONVETCH (Anilinokras. Prom., 1933, 3, 263—267).—The resistance to corrosion of chemical plant by chemicals during various processes of S-dye manufacture was investigated. Vessels made of boiler plate resisted fairly well the action of  $C_6H_3Cl(NO_2)_2$ -NaOH, but approx. 50% aq. NaOH rapidly corroded them. H<sub>2</sub>S and polysulphides, especially at high temp., were extremely destructive. The degree of attack on the vessels was not uniform, but depended on the local conditions. Attempts to mend cracks etc. by welding resulted in enhanced corrosion. It is recommended that the vessels should be so designed as to enable corroded parts to be rapidly and cheaply replaced. M. Z.

See also A., Dec., 1268, Anthraquinone dyes in graebeite. 1285, Analysis of hair dyes. 1286, Glucoside azo dyes. Azo dyes. 1297, Prep. of *Bz*-1-chlorobenzanthrone. 1302, Natural colouring matters and their analogues. 1305, [Dyes from] aliphatic keto-anils. 1308, Sandmeyer indigo synthesis. 1310, Pyrrole blacks.

#### PATENTS.

Manufacture of easily soluble ester-salts of leuco-vat dyes. DURAND & HUGUENIN A.-G. (B.P. 401,137, 24.1.33. Ger., 25.1.32).—The Li salts of disulphuric esters of certain leuco-vat dyes, *e.g.*, indanthrone, dichloroindanthrone, dibenzanthrones, pyranthrones, dichloro- and dibromo-anthanthrones, are more sol. than the Na salts. C. H.

Manufacture of [acid] dyes of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 401,132, 5.1.33. Ger., 6.1.32).—A 4-halogeno-1-aminoanthraquinone-2-sulphonic acid is condensed with a *m*- or *p*-aminoarylalkylsulphone,  $\rm NH_2$ ·Ar·SO<sub>2</sub>R, where Ar and/or R carry  $\lt$  1 substituent; R may, e.g., carry OH, CO<sub>2</sub>H, or CO<sub>2</sub>R'. Blue acid dyes are thus obtained from 4-bromo-1-aminoanthraquinone-2-sulphonic acid and : *m*-NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·SO<sub>2</sub>Me; the *p*-compound, m.p. 134—135°; *m*-NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·SO<sub>2</sub>·CH<sub>2</sub>·CO<sub>2</sub>H; 5-amino-2-acetamidophenylmethylsulphone, m.p. 197—198°; its 2-Cl-derivative; *p*-NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·SO<sub>2</sub>·C<sub>2</sub>H<sub>4</sub>·OH, m.p. 107—108°; 2-amino-5:6:7:8-tetrahydro-4-naphthylmethylsul-phone, m.p. 85—86°. C. H.

[Preparation of] water-soluble [salts of] di-iodohydroxymercurifluorescein. H. M. CHILES, ASST. to PYRO-PACK PRODUCTS CO. (U.S.P. 1,893,226, 3.1.33. Appl., 18.8.30).—The Na<sub>2</sub>, K<sub>2</sub>, and (NH<sub>4</sub>)<sub>2</sub> salts are claimed as germicidal dyes for cosmetics. H. A. P.

[Preparation of a] colour base and compositions containing same. J. H. CROWELL, ASST. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,892,871, 3.1.33. Appl., 21.6.28).—Oil-sol. coloured compounds are obtained by heating hydroxyanthraquinones (crude chloroquinizarin containing quinizarin) with an aromatic primary amine (NH<sub>2</sub>Ph in excess) and H<sub>3</sub>BO<sub>3</sub> in a stream of inert gas at 145—180°. Lacquers, stains, and plastics coloured by these compounds are claimed; it is also claimed that more sol. colouring matters are obtained by using mixtures of polyhydroxyanthraquinones with mixtures of amines. H. A. P.

Azo dyes [ice colours]. G. BONHÔTE, ASST. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,892,480, 27.12.32. Appl., 14.6.32. Switz., 1.7.31).—Black dyeings, fast to light, are produced from 2:3-hydroxynaphthoic 4'-amino-3':6'-dialkoxy- or -3'-alkoxy-6'-alkyl-anilides by successive treatment with  $HNO_2$  and alkalis. The anilides [-3':6'-(OMe)<sub>2</sub> and -(OEt)<sub>2</sub>] are prepared by condensing the appropriate 4'-NHAc- or 4'-NO<sub>2</sub>-compound with 2:3-hydroxynaphthoic acid and hydrolysing or reducing the product. H. A. P.

Manufacture of water-soluble [acid] azo dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. British Chemical Abstracts-B.

56

400,920-2, 16.3.32).-An aminoarylamide of an aliphatic acid  $> C_9$  is diazotised and coupled with (A) a naphtholdisulphonic acid or derivative, or (B) a sulphoarylpyrazolone or sulphoarylamide of acetoacetic acid etc.; in (c) a coupling component carrying an aliphatic chain  $> C_9$  is coupled with a diazotised sulphonated arylamine. The products give dyeings fast to washing. Examples are: (A) stearic p-aminoanilide (I)  $\rightarrow$  G-acid (red-orange); ricinoleic p-aminoanilide (blue-red); lauric 2-amino-p-toluidide  $\rightarrow$  benzoyl-H-acid (blue-red); (B) lauric p-aminoanilide  $\rightarrow$  1-m-sulphophenyl-3-methyl-5pyrazolone (yellow); (I)  $\rightarrow$  acetoacetic sulpho-oanisidide (yellow); (c) 1:5:7-naphthylaminedisulphonic acid  $\rightarrow$  acetoacetic *p*-stearamidoanilide (yelloworange); sulphanilic acid  $\rightarrow$  dodecylaniline; dianisidine  $\rightarrow$  2 mols. of oleyl-H-acid (blue), etc. [Stat. refs.] C. H.

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

Differentiation of flax from hemp fibre's. F. JANÁK (Chem. Listy, 1933, 27, 388–392).—Analytical data are given for the ash from flax (I) and hemp (II) fibres; that from (I) differs from the latter chiefly in its lower SiO<sub>2</sub> content [8\*92—11.5%, as compared with  $23\cdot33$ — $26\cdot5\%$  for (II)], and in that it contains no Cl [(II) contains  $0\cdot11$ — $0\cdot66\%$ ]. R. T.

Preparation of furfuraldehyde from flax refuse. N. N. ORLOV and S. G. RIKLIS (J. Appl. Chem. Russ., 1933, 6, 910-914).—The material rejected in the preliminary treatment of flax yields 9% of furfuraldehyde on heating during 7 hr. at 160° with  $0.1N-H_2SO_4$  or -HCl; addition of NaCl to 5% reduces the time necessary to 3-4 hr. The yields obtained, using a no. of other solutions, under similar conditions are:  $H_2O$ 2.8, 15% NaCl 4, 1%  $H_3PO_4$  7.5,  $0.1N-H_2C_2O_4$  4.35, and 0.2N-NaHSO<sub>4</sub> 7-7.9%. R. T.

Standard classification of purified cotton-wool according to viscosity. Technical method for preparation of collodion wools of various viscosity. D. GALPERIN and D. TUMARKIN (J. Appl. Chem. Russ., 1933, 6, 850–868).—The  $\eta$  of solutions of cellulose nitrate (I) of a given N content depends on that of the original cotton-wool (II), on the temp. of nitration, and on the conditions of stabilisation. Standard conditions for purification and dissolution in Schweitzer's reagent of (II) are given. Preps. of (I) of low  $\eta$  are obtainable from any (II) by heating the (I) during 1 hr. at 150° with H<sub>2</sub>O. R. T.

Influence of chemical and thermal treatment of viscose films (artificial leather) on their imbibition of water. A. A. MOROZOV and V. I. ALEXEENKO (J. Appl. Chem. Russ., 1933, 6, 869–879).—Imbibition of  $H_2O$  by viscose films (I) is smaller after treatment with tanning extracts, aq.  $Al_2(SO_4)_3$ , or aq. Cr alum, followed by drying at 150°. (I) largely lose their elasticity after treatment with mineral mordants. R. T.

I. Colloidal and mechanical properties of cellulose nitrates and their fractions. II. Effect of addition of certain substances on the mechanical properties of cellulose nitrate and its fractions. A. I. MEDVEDEV (J. Appl. Chem. Russ., 1933, 6, 880-888, 889-896).-I. Cellulose nitrate (I) was fractionally pptd. by adding 1:1 H<sub>2</sub>O-COMe<sub>2</sub> mixture (II) to its COMe<sub>2</sub> solutions; the bulk of (I) is pptd. (fraction A) when the H<sub>2</sub>O content attains a certain sp. val. characterised by an abrupt fall in  $\eta$ . Further addition of (II) yielded a second fraction, B, the mother-liquor from which gave a third fraction on further dilution. Fraction A was purified further by repeating the above procedure a no. of times. The content of mineral substances and coloured impurities increases with the no. of pptns. in the fractions A, and decreases in the fractions B. The films obtained from B are colourless, transparent, and of high tensile strength, and remain transparent after denitrification. The N content of the various fractions is not significantly affected by the above procedure.

II. The rigidity of films of (I) and of the above fractions varies inversely with their content of polymerised vinyl acetate (III), whilst the resistance to elongation increases with the (III) content up to 20%and then diminishes. The mechanical properties of films of denitrated (I) are greatly improved by the presence of 10% of (III). R. T.

Fractionation of commercial cellulose acetate and ethylcellulose. L. UBBELOHDE (Cellulosechem., 1933, 14, 169).—The viscosity measurements of Herzog and Deripasko (B., 1932, 223) and of Okamura (A., 1933, 1280) are criticised. H. B.

Change in chemical pulps during beating. K. O. BERG (Papier-Fabr., 1933, 31, 605-611, 617-622).-The effect of beating in a multiple ball mill on fibre length and unit fibre tensile strength is investigated. A method (I) is described of determining fibre length by rate of sedimentation of the pulp suspended in 96% EtOH or EtOH-glycerin mixture. Results obtained thus are not comparable with those obtained by microscopical methods (II) since they include all fibres, together with the finest particles of mucilage which cannot be measured by (II). The ratio of fibre length to diam. determined by (II) decreases with prolonged beating of soft pulps (from German wood), but rises in the case of harder pulps (from slower-growing Scandinavian wood). Beating produces a sharp rise in unit fibre strength with a decrease in diam., the relation between the two being determined by the hardness of the pulp. Slime particles become finer on beating.

D. A. C.

Effect of different-sized fibres on the physical properties of groundwood pulp. E. R. SCHAFER and M. SANTAHOLMA (Paper Trade J., 1933, 97; T.A.P.P.I. Sect., 224—229).—By screening experiments it is found that both strength and freeness are directly related to fibre length. H. A. H.

Chemical properties of screen fractions of black gum and slash-pine groundwood pulps. E. R. SCHAFER and M. SANTAHOLMA (Paper Trade J., 1933, 97; T.A.P.P.I. Sect., 230-231).—Both lignin content and solubility in 1% NaOH are higher in the finer than in the coarser fractions. The chemical properties appear to have no bearing on the papermaking qualities of a pulp. H. A. H.

Chemistry of alkaline wood-pulp processes. IV. J. S. MARTIN, M. W. BRAY, and C. E. CURRAN (Paper

Quality of pulp from sulphite cooks. E. RICHTER (Zellstoff u. Papier, 1933, 13, 560–563).—The effects of the personal element, variation in liquor concn., moisture content of the wood, size of the chips, and cooking time on the variation in pulp quality are investigated. A relatively small increase in the free SO<sub>2</sub> of the liquor decreases the quality, the yield being unaltered; an increase in the combined SO<sub>2</sub> produces only a slight increase in strength, but raises the consumption of S. Air-dried wood containing 38% H<sub>2</sub>O produces a pulp of higher quality and bleachability than soaked wood of 45% H<sub>2</sub>O. Quality is expressed as: (burst × freeness)/Cu no. D. A. C.

Development and control of micro-organisms in a pulp- and paper-mill system. J. R. SANDBORN (J. Bact., 1933, 26, 373–378).—It is impossible to clear slime-encrusted pipes by  $Cl_2$  treatment; mechanical means must be adopted. Fresh accumulations may be materially reduced by heavy  $Cl_2$  dosage of the system or, where this is impracticable, by treatment with Cu salts instead of or in addition to mild chlorination.

A. G. P.

**Fungoid growth on sulphite pulp.** E. OPFERMANN (Papier-Fabr., 1933, **31**, 642—643).—Various fungi developed in a consignment of sulphite pulp during 2 days' transit by rail. This was found to be due to the presence of artificial manure in the truck, containing P, which acted as a nutrient for the bacteria already present in the pulp. D. A. C.

Analysis of alkaline black liquors of varying sulphidity [from soda- and sulphate-pulp processing] by ammonia-distillation method. M. A. HEATH, M. W. BRAY, and C. E. CURRAN (Paper Trade J., 1933, 97; T.A.P.P.I. Sect., 237—241).—The method described previously (B., 1933, 343) has been further developed and modified, and now gives reproducible results with black liquors of widely varying composition. Black liquor changes on keeping, the NaOH content usually increasing at the expense of the Na<sub>2</sub>S.

Treatment and preparation of size in the paper industry. E. PALLAS (Papier-Fabr., 1933, 31, 634– 637).—The properties of various types of size are described. Methods of sampling and testing consignments are given, together with an account of its digestion. D. A. C.

Sizing [of paper], sizing problems, and the preparation of resin size. ECKE (Papier-Fabr., 1933, 31, 667-673).—The prep. of the size milk which will give the best results is described. Sizing depends on the pptn. of Ca and Al resinates with the formation of  $Al_2O_3$  and basic Al sulphates, all of which are deposited on the fibre by mutual neutralisation of charges. The ability of a pulp to be sized is a function of its adsorptive capacity and is a property inherent in the pulp depending on its treatment during cooking. Providing all other conditions are const., the strongest pulps give the best

sizing effects. The addition of  $H_2SO_4$  (I) and NaOH (II) has no effect on the degree of sizing, though (I) increases the whiteness of the sheet, and (II) gives a brownish shade. Drying completes sizing by fusion of the particles, to aid which small quantities of montan wax may be added. A high  $p_{\rm H}$  during beating will retard fusion. The best results are obtained with very highly dispersed size milks which may be obtained by mixing the resin soap under pressure with cold  $H_2O$  at a resultant temp. of 50—60° with exclusion of air, and addition to softened (boiler)  $H_2O$  at 80°. D. A. C.

Colloidal control in starch sizing [of paper]. B. W. ROWLAND (Paper Trade J., 1933, 97; T.A.P.P.I. Sect., 249—250).—A procedure for pptg. starch on to pulp fibres with  $Al_2(SO_4)_3$  after beating is described. It is essential to subject the furnish to the least possible mechanical agitation after pptn. Increased bursting and tensile strength is claimed, in presence or in absence of filler, but tear and opacity are somewhat reduced.

H. A. H.

D. A. C.

Dyeing of paper fibres.—See VI. Uses of "liquid resin."—See XIII.

See also A., Dec., 1235 and 1243, Structure of cellulose gels. 1236, Structure of thin celluloid films. 1243, Viscosity of silk solution. 1244, Action of alkaline Cu solutions on silk fibroin. 1266, Paper hygrometers. 1280, Esterification of cellulose. Fractionation of ethylcellulose.

#### PATENTS.

Preparation of high- $\alpha$ -cellulose fibre for conversion into cellulose derivatives. G. A. RICHTER, Assr. to BROWN CO. (U.S.P. 1,906,885, 2.5.33. Appl., 24,12.25).—Unbleached sulphite pulp is treated at room temp. with NaOH of 18% concn.; the product after thorough washing is a fibre containing 96—98%  $\alpha$ -cellulose. It is then treated with a 2—3% bleach. In the case of well-cooked kraft and soda pulps it is preferable to pretreat them with liquors containing 1—3% available Cl<sub>2</sub>. D. A. C.

Hydrolysis of cellulose. L. A. STEGEMEYER and J. O. PEIRCE, ASSTS. to TWITCHELL PROCESS CO. (U.S.P. 1,906,429, 2.5.33. Appl., 1.4.30).—Sawdust mixed with  $H_2O$  and a small quantity of sludge-layer mineral oil sulphonic acid is boiled under pressure (130 lb. per sq. in.). The resulting liquid of fermentable sugars is removed and the process repeated. Farm waste may be so treated. D. A. C.

(A) Cellulosic, (B) cellulose ester, and (c) cellulose organic derivative, composition containing (A) a dialkyl hydrophthalate, (B) the tetraethyl ester of a saturated aliphatic tetracarboxylic acid, and (c) a  $\beta\beta'$ -dialkoxyethyl carbonate. H. B. SMITH, ASST. to EASTMAN KODAK CO. (U.S.P. 1,905,516—8, 25.4.33. Appl., [A] 17.6.31, [B, c] 10.8.31).—Cellulose

H. A. H.

esters [(A) acetate, (B) acetate or nitrate] plasticised by (A) (Et<sub>2</sub> or Bu<sub>2</sub>) hexahydrophthalates (10-20%), and (B) aliphatic Et<sub>4</sub> tetracarboxylates (20-30% of Et<sub>4</sub> ethane- $\alpha\alpha\beta\beta$ - or propane- $\alpha\alpha\beta\gamma$ -tetracarboxylates) are claimed as film- or fibre-forming materials. (c) Cellulose ethers or esters (acetate) plasticised by (20-50 wt.-%) of a  $\beta\beta'$ -dialkoxyethyl carbonate (OMe, OEt) are claimed as film-forming and moulding (thermoplastic) compositions. H. A. P.

**Production of pulp.** L. BRADLEY and E. P. MCKEEFE, Assrs. to BRADLEY-MCKEEFE CORP. (U.S.P. 1,906,836, 2.5.33. Appl., 2.7.26. Renewed 25.1.30).—Liquors from  $Na_2SO_3$  (I) and  $Na_2SO_4$  (II) cooking processes are combined in a cycle so that the residual liquor from (I) provides all the Na and S for (II), and the spent liquor from (II) makes most of its Na content available for (I). The spent liquor from (I) is smelted to  $Na_2CO_3$  and  $Na_2S$  and causticised with Ca(OH)<sub>2</sub> to contain sufficient NaOH and  $Na_2S$  to be used in (II), the spent liquor from which when burned in a mildly reducing atm. will contain chiefly  $Na_2CO_3$ . This by treatment with  $SO_2$ or NaHSO<sub>3</sub> is converted for use in (I). D. A. C.

Recovering and utilising the valuable compounds in spent cooking liquors. G. A. RICHTER, Assr. to BROWN CO. (U.S.P. 1,906,886, 2.5.33. Appl., 23.11.27).—The spent sulphate liquor is evaporated to 50% concn. and smelted in a reducing atm., thus producing a mixture of Na<sub>2</sub>CO<sub>3</sub>, Na<sub>2</sub>S, and small quantities of Na<sub>2</sub>SO<sub>4</sub> of 25—35% sulphidity. The smelt is run into H<sub>2</sub>O, and, on cooling to 30°, the Na<sub>2</sub>CO<sub>3</sub> crystallises out, giving a mother-liquor of 50% sulphidity, or to 10° with 80—90% sulphidity. D. A. C.

Safety paper. B. W. SMITH, ASST. to TODD Co., INC. (U.S.P. 1,906,824, 2.5.33. Appl., 16.1.31).—Paper is impregnated by addition to the pulp or the web of a colourless solution of pp'-dihydroxydiphenyl dissolved in EtOH, which develops a bright orange colour on addition of NaOCl. Alternatively, a colourless ink may be used, made by mixing with varnishes or the usual vehicles.

Centrifugal pots for spinning of artificial silk. Soc. de Constructions Mécaniques de Stains (B.P. 402,362, 7.7.33. Fr., 28.7.32).

Means [press rolls] for extracting water from paper pulp and like substances in paper-making and like machines. F. REDDAWAY and J. MUSKETT (B.P. 401,800, 4.7.32).

Coating of paper [webs] and the like. CARRIER-Ross ENG. Co., LTD. (B.P. 401,980, 10.5.33. U.S., 10.5.32).

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

Vat dyeing on [cotton and rayon] piece goods. C. W. NELSON (Amer. Dyestuff Rep., 1933, 22, 685–688, 713–716).—Practical methods are described. Oxidation of the dyed fabric is preferably effected with NaBO<sub>3</sub> instead of Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> + AcOH. A. J. H.

Direct dyes and cellulose from the viewpoint of substantivity. S. M. NEALE and L. H. GRIFFITHS (Amer. Dyestuff Rep., 1933, 22, 651-654).--Modern views on the absorption of direct dyes by cellulose, and their physical properties, are described. In the early stages of dyeing most of the absorbed dye is on the surface of the cellulose material; subsequently the dye diffuses inwardly until there is no concn. gradient. A. J. H.

Determinations of specific gravity. II. Influence of dyeing and of superficial hydrolysis on the specific gravity of cellulose acetate silk. P. M. HEERTJES, W. COLTOF, and H. I. WATERMAN (Rec. trav. chim., 1933, 52, 1001—1006; cf. B., 1933, 541).— Measurements of d show that when acetate silk is dyed (with Cellit Fast Red B) the pores beome partly clogged (cf. B., 1931, 1007). Partial hydrolysis of the silk, whether combined or not with dyeing (Benzopurpurin), prevents such clogging. H. F. G.

Factors governing absorption of dyestuffs by paper fibres. F. A. SODERBERG (Paper Trade J., 1933, 97; T.A.P.P.I. Sect., 251-257).—Numerous factors affecting the dyeing of cellulose fibres, both in the beater and at the calender, are discussed. In particular, the wetness of the stock and the order of adding the various ingredients are important. The use of BaCl<sub>2</sub> is advocated when excessive quantities of acid dyes are required. Traces of Cl<sub>2</sub> in bleached pulp are detrimental in many cases; efficient washing is the best remedy. When  $ZnS_2O_4$  is used for bleaching mechanical pulp (B., 1933, 56) it should be allowed to exhaust before dye is added. H. A. H.

Standard methods for determining the fastness of dyes on textile fabrics towards washing, perspiration, and water. W. PLUZANSKI (Ann. Chim. Analyt., 1933, [ii], 15, 433—441).—An apparatus for moving strips of dyed fabric up and down at a regular rate in soap and other solutions is described, and details of standard procedures are given for determining the bleaching effect of hot and cold soap solutions, of ammoniacal and AcOH NaCl solutions, and of  $H_2O$  on dyed fabrics. A. R. P.

Dyeing and printing industries. G. MARTIN (Bull. Soc. chim., 1933, [iv], 53, 1001-1015).—A lecture on recent advances and current practice.

Evaluation of textile penetrants. Centrifugal method. S. LENHER and J. E. SMITH (Ind. Eng. Chem. [Anal.], 1933, 5, 376—381).—The weighed textile is immersed in the solution of penetrant for a given time and centrifuged under standard conditions; the increase in wt. is taken as a measure of the penetration. Results are given for 5 commercial products applied to cotton, wool, and silk. The surface tension of a solution towards air is no criterion of its ability to penetrate textile fibres. E. S. H.

Machine for continuous de-sizing of artificial silk fabrics. Anon. (Textilber., 1933, 14, 606).

See also A., Dec., 1241, Absorption of dyes by cellulose.

#### PATENTS.

Coloration [dyeing in black shades] of textile materials of or containing cellulose esters or ethers. BRIT. CELANESE, LTD., G. H. ELLIS, and H. C. OLPIN (B.P. 401,338, 4.5.32).—A dischargeable full black is produced by dyeing with a mixture of an aminazo compound (*p*-aminobenzeneazodi-alkyl- or -ary

D. A. C.

aniline), which when diazotised and suitably coupled gives a navy-blue or thin black, with a dischargeable yellow, orange, or red dye stable to diazotisation (4-nitro-2-methoxybenzeneazodi-methyl- or -ethyl-aniline or benzeneazobenzeneazophenol), diazotising, and developing (with 2:3-hydroxynaphthoic acid). Coloured discharges may be produced by introduction of undischargeable dyes. H. A. P.

Discharge pastes and discharge printing processes [on cellulose esters or ethers]. E. I. DU PONT DE NEMOURS & Co. (B.P. 401,350, 12.5.32. U.S., 15.5.31).—A mixture of alkali formaldehydesulphoxylate, Zn(SCN)<sub>2</sub>, and diethylene glycol is claimed. H. A. P.

Weighting of animal fibres. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 401,477, 21.5.32).—The goods are treated with > 10 wt.-% of alkyl H sulphates  $\ll C_8$  or their salts in acidified aq. solution (at 80—100°). Halogenated products of the type described in B.P. 394,043 (B., 1933, 823) are specifically claimed. The process may be combined with dyeing or other textile processes. H. A. P.

Wetting etc. agents .- See III.

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

Apparatus for production of sulphuric acid by the lead-chamber process. P. GUARESCHI (l'Ind. Chimica, 1933, 8, 1227—1231, 1384—1394).—The design and operation of the Pb chamber are discussed, with particular reference to the ratio of surface area (S) to vol. (V) and the temp. relationships in the various parts of the plant. For various types of European and American plant the daily output in kg. per cu.m. is given by  $\alpha(S/V)^2$ , where  $\alpha$  is 20—25. H. F. G.

Reactions in the lead chamber ; modern sulphuric acid plant. P. GUARESCHI (L'Ind. Chimica, 1933, 8, 964-973, 1119-1127).-Theories of the reactions involved are discussed, and modern improvements in plant are noted. The velocity of the reaction in the Pb chamber (C) is proportional to the  $SO_2$  concn. (up to 13%) and to the NO concn., and falls with increase of the rate of flow and on ionisation of the gas. The ratio NO2. NO/SO2 should be a max., and the optimum temp. is 90-100°; the optimum H<sub>2</sub>O v.p. depends on the composition of the mixture. The rate of oxidation of NO is approx. const. throughout C, lowering of temp. being compensated by reduction of the O2 concn. Reduction of NO2 occurs when the relative SO2 concn. exceeds a crit. val. The walls promote the reaction, either by their cooling action or by a sp. surface effect. The pressure should be higher in the Glover tower than in C. The application of these and other conclusions to the design of plant is described. H. F. G.

Formation of calcium chloride in the liquors of salt deposits. I. Action of chloride solutions on gypsum at normal pressure. F. KRÜLL (Kali, 1933, 27, 67–69, 84–88; Chem. Zentr., 1933, ii, 425).— Aq. KCl reacts at normal pressure with gypsum:  $2(CaSO_4, 2H_2O) + 2KCl = CaSO_4, K_2SO_4, H_2O + CaCl_2, 3H_2O$ . NaCl, MgCl<sub>2</sub>, and NH<sub>4</sub>Cl do not react. The quantity of CaCl<sub>2</sub> increases with time, fall in temp., and

increasing KCl concn.; addition of NaCl or MgCl<sub>2</sub> reduces it. A. A. E.

Calcium carbide and calcium cyanamide. J. GELHAAR (Tekn. Tidskr., 1933, 63, No. 14, Kemi, 25–31; No. 19, Kemi, 37–40; Chem. Zentr., 1933, ii, 197).— The effect of particle size, composition of tube, temp., and addenda on the velocity of reaction between  $CaC_2$ and N<sub>2</sub> has been studied.  $CaF_2$ ,  $CaBr_2$ ,  $CaCl_2$ , and LiCl accelerate the reaction in that order owing to increase in the ionisation of N<sub>2</sub>. Hydrocarbons retard it. Arno's observed relation between velocity and particle surface is confirmed. A. A. E.

Poisoning by arsenic of a tin-barium-vanadium catalyst previously inactivated by contact poisons. I. E. ADADUROV, P. P. PERSCHIN, and G. V. FEDOROVSKI (J. Appl. Chem. Russ., 1933, 6, 797-802).—A V catalyst (I) poisoned by lighting gas does not undergo further inactivation in the reaction  $2SO_2 + O_2 \rightarrow 2SO_3$  by poisoning with  $As_2O_3$ , and combines with considerably less  $As_2O_3$  than does the unpoisoned (I). At 550° both active and poisoned (I) give 91.5% conversion of  $SO_2$ . It is concluded that oxidation of  $SO_2$  takes place at quite different centres from those combining with  $As_2O_3$  or other contact poisons. R. T.

Oxidation of sulphur dioxide to sulphur trioxide by the contact method under pressure. I. P. M. LUKJANOV, I. N. BUSCHMAKIN, M. V. RISAKOV, and I. R. MOLKENTIN (J. Appl. Chem. Russ., 1933, 6, 772-784).-The highest yields of SO<sub>2</sub> from SO<sub>2</sub>, using Ural pyrites slag (I) as catalyst, are obtained at 650°; the yields are unaffected by varying concn. of SO2 from 6 to 8%, and of O2 from 10 to 20%, but increase with increasing pressure and diminishing velocity of flow. The activity of (I) is diminished by a H<sub>2</sub>O content of the reaction gases > that remaining after they are passed through conc.  $H_2SO_4$ , by  $SO_3$  concns. > 10%, and by even transient fall in temp. below 650° during the reaction; even under optimum conditions (I) is largely inactivated after 4 hr., but may be reactivated by passing dry air at 650° during 3-5 hr. R. T.

Determination of bromine in presence of chlorine in mineral waters. I. ORLOV, O. KIRIANOVA, and T. KSENOFONTOVA (Khim. Farm. Prom., 1933, 2, 72-75). -200 c.c. of conc.  $H_2O$  (40-80 mg. Br) are freed from I with  $KNO_2$ ; 3 g. of fresh  $MnO_2$  and 25 c.c. of 20%  $H_2SO_4$  are added, and the mixture is distilled into 3% NaOH with some 5% Na<sub>2</sub>SO<sub>3</sub>. The distillate is treated with 20 c.c. of 20%  $H_2SO_4$ , 3 g. of  $MnO_2$ , and 2 g. of  $MnSO_4,5H_2O$ , neutralised, and titrated potentiometrically or by Volhard's method. CH. ABS.

Determining NO in gas.—See II. Determining MgO in dolomite etc.—See IX.  $CaC_2$ . Anodes for  $Cl_2$  production.—See XI. Prussian-blue and Marsyellow.—See XIII.

See also A., Dec., 1252, Simultaneous prep. of  $H_2SO_4$  and HCl. Transformation of alkaline-earth cyanides into cyanamides. 1253, Pt catalyst on SiO<sub>2</sub> gel. Catalytic decomp. of NaOCl solutions. 1254, Electrochemical reduction of acid perrhenate solutions, and oxidation of Cr<sup>III</sup> to CrO<sub>4</sub>". 1257, Prep. of Cu selenate tetrammine dihydrate

crystals. Prep. of  $B_5H_{11}$ . 1258, Pb-chamber process. Prep. of permolybdates. 1259, Recovering I from residues. 1260, Determinations of Cl', I', F', ClO<sub>4</sub>', H<sub>2</sub>S, S'', S<sub>2</sub>O<sub>3</sub>''. 1261, Determining SeCN'. 1266, Sol. anhydrite as desiccating agent.

#### PATENTS.

Sulphuric acid concentration. S. A. MONTGOMERY and J. A. HUBBARD, Assr. to STANDARD OIL CO. (INDIANA) (U.S.P. 1,906,399, 2.5.33. Appl., 24.10.29).—Dil. acid  $(d \ 1\cdot 26)$  at 10—38° is heated in a preheater (P) to 65—93°/1 atm. before being introduced into a vac. concentrator (C) at a pressure of 26—27 in. Hg, where rapid evaporation takes place. Part of the acid in C may be continuously returned to P for re-heating, thus avoiding the necessity for steam-coils in C. W. J. W.

**Preparation of strong hydrogen halide gas.** E. O. BARSTOW and S. B. HEATH, ASSTS. to DOW CHEM. Co. (U.S.P. 1,897,996, 21.2.33. Appl., 4.10.29).—In a process as described in U.S.P. 1,853,330 (B., 1933, 105), the tower is divided into two sections, viz., a lower distilling zone (D) and an upper fractionating zone (F). The heating gases are circulated only through the heater (H) and D and gradually attain const.-boiling composition. Corrosion of ordinary metals in the blower etc. is prevented by having a by-pass across D, the mixture of used cool gases and by-passed hot gases (being the feed to H) not being allowed to fall below 150°, and the hot gases being > 300°. B. M. V.

Manufacture of hydrocyanic acid. ROESSLER & HASSLACHER CHEM. Co. (B.P. 401,351, 12.5.32. U.S., 14.5.31).—An alkali cyanide is caused to react with an acid in presence of  $Na_2SO_3$ , so that the product retains 0.05-0.5% SO<sub>2</sub>, which acts as a stabilising agent.

W. J. W.

Acid vat. H. E. FRITZ, ASST. to B. F. GOODRICH CO. (U.S.P. 1,899,413, 28.2.33. Appl., 17.3.31).—A tank provided with adherent rubber lining is also lined with bricks. B. M. V.

Ammonia-oxidising apparatus. I. HECHENBLEIK-NER and N. TITLESTAD, Assrs. to CHEM. CONSTRUCTION CORP. (U.S.P. 1,898,775, 21.2.33. Appl., 3.4.29).—  $\rm NH_3$  is evolved from  $\rm NH_3$  liquor by heat derived from the exothermic oxidising reaction and transferred by a circulation of  $\rm H_2O$  (cf. U.S.P. 1,748,646; B., 1930, 1026). B. M. V.

Dehydrating caustic alkali solutions. C. E. MILLER, ASST. to CHAMPION FIBRE CO. (U.S.P. 1,899,627, 28.2.33. Appl., 19.8.31).—The solution is exposed directly to burning  $H_2$ , which may be the by-product of the electrolytic production. B. M. V.

Extraction or purification of sodium chloride. R. HARA (B.P. 401,612, 14.6.33).—The raw material is treated at  $< 0^{\circ}$  and under vac. with liq. NH<sub>3</sub> to dissolve NaCl. The solution is filtered and evaporated, or heated to 30° to recrystallise part of the NaCl, which is filtered off from the remaining solution. Traces of NH<sub>3</sub> are removed by final heating at 150—200°.

W. J. W. Producing a double salt from a single salt. G. B. BURNHAM, Assr. to BURNHAM CHEM. Co. (U.S.P. 1,899,310, 28.2.33. Appl., 26.5.30). — Crystals of  $Na_2CO_3$ ,  $2Na_2SO_4$  mixed with NaCl are obtained from, e.g., Searles Lake brine by rapid evaporation with agitation. The crystals are separated by hydraulic classification, the NaCl being the heavier. B. M. V.

Composition for use as a washing powder etc. F. C. BOWMAN, Assr. to A. R. MAAS CHEM. Co. (U.S.P. 1,907,316, 2.5.33. Appl., 19.2.30).—A mixture of  $Na_4PBO_{6,}18H_2O$  and  $Na_3PO_4,12H_2O$  is free-flowing and does not cake. It is suitable also as a softener for boiler feed-H<sub>2</sub>O. W. J. W.

Manufacture of alkali cyanides. N. V. STIKSTOF-BINDINGSIND. "NEDERLAND" (B.P. 401,627, 3.8.33. Ger., 4.8.32).—Alkali thiocyanates are heated at 400— 800° with an alkaline-earth oxide or carbonate or with certain heavy-metal oxides in a current of CO or gases containing CO. The NaCN or KCN is extracted from the reaction product with a mixture of liquid NH<sub>3</sub> and EtOH. A. R. P.

Manufacture of ammonium chloride and sodium carbamate. MATHIESON ALKALI WORKS, Assees. of R. B. MACMULLIN (B.P. 401,554, 2.3.33. U.S., 24.3.32). —CO<sub>2</sub> is passed into a solution of NaCl in liquid NH<sub>3</sub> until the solution is saturated with NH<sub>4</sub>Cl (134 g. per 100 g. NH<sub>3</sub>) and, after separation of the NH<sub>2</sub>·CO<sub>2</sub>Na, more CO<sub>2</sub> is passed into the liquid, whereby 25% of the NH<sub>4</sub>Cl is pptd. The mother-liquor is returned to the first stage. A. R. P.

Manufacture of ammonium magnesium phosphate. C. D'ASSEEV (B.P. 401,407, 21.6.32. Belg., 22.6.31).—By pptg.  $NH_4H_2PO_4$  solution with  $Mg(HCO_3)_2$  solution, the use of an alkali, *e.g.*,  $Na_2CO_3$ , for neutralising the two H atoms is rendered unnecessary.

W. J. W.

Apparatus for calcining gypsum. L. C. KARRICK and W. L. ELLERBECK (U.S.P. 1,897,259, 14.2.33. Appl., 30.1.28).—A closed rotary cylinder heated by internal elements, *e.g.*, electric resisters, which on rotation pass through the gypsum is described. Additional agitation is effected by releasing the generated steam pressure suddenly at regular intervals. B. M. V.

Gypsum calciner. W. M. McNEIL, Assr. to UNITED STATES GYPSUM CO. (U.S.P. 1,897,394, 14.2.33. Appl., 17.11.30).—A closed vessel in which calcination is effected by high-pressure steam is provided with a reciprocating axial shaft carrying suitable arms for agitation. B. M. V.

Manufacture of nickel carbonyl. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 401,438, 18.7.32).— Scrap Ni in a coarse form, e.g., wire, sheet, or Ni-plated metal, is treated with CO at  $> 100^{\circ}/> 10$  atm.

A. R. P.

Purification of aluminium sulphate solutions. F. TELLER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,897,740, 14.2.33. Appl., 11.1.32. Ger., 13.1.31).— HCl, NaCl, or other chloride equiv. to the Fe<sup>TT</sup> present is added and the solution extracted with an O-containing org. solvent, b.p.  $> 100^{\circ}$ , slightly sol. in H<sub>2</sub>O, *e.g.*, *cyclo*hexanone. B. M. V.

Manufacture of stannic chloride. W. J. BUTT-FIELD, ASST. to VULCAN RETINNING Co. (U.S.P. 1,897,360,

14.2.33. Appl., 9.7.29).—Anhyd.  $\operatorname{SnCl}_4$  is manufactured by the action of dry  $\operatorname{Cl}_2$  on stacked ingots of Sn in presence of a liquid that is circulated through a cooler and back again, to remove exothermic heat and prevent loss of surface of the Sn by melting. The liquid may be  $\operatorname{SnCl}_4$  itself,  $\operatorname{CCl}_4$ , or any other that is inert and has a b.p. < the m.p. of Sn. B. M. V.

(A) Dissolution of titanium tetrachloride in aqueous solvents. (B, C) Preparation of, (C) concentrated, clear solutions of titanium chloride. P. KUBELKA, Assr. to KREBS PIGMENT & COLOR CORP. (U.S.P. 1,899,572-4, 28.2.33. Appl., 12.2.32. Ger., [A] 27.2.31, [B] 23.2.31, [C] 25.2.31).—TiCl<sub>4</sub> is passed into an agitated aq. solvent, below the surface, with inert gas for agitation if desired; continuous additions of H<sub>2</sub>O and TiCl<sub>4</sub> to a bulk of already formed solution may be made. (B) The free and combined HCl of the bulk solution is held at > 150 g./litre. (c) The initial turbidity produced by addition of TiCl<sub>4</sub> is removed by addition of further TiCl<sub>4</sub> until the (total) HCl is > 250 g./litre and the TiO<sub>2</sub> > 130 g./litre. B. M. V.

Preparation of a lead carbonate product. S. S. S. SVENDSEN, ASST. to G. BIRKENSTEIN (U.S.P. 1,898,405, 21.2.33. Appl., 12.9.28).—Scrap battery plates are tumbled in a solution of  $Na_2CO_3$  with a little NaOAc until bright metal and PbCO<sub>3</sub> are obtained. B. M. V.

Treatment of (A) vermiculite and the like, (B) liquor resulting from recovery of lamisilite from vermiculite ore. R. G. GUTHRIE and O. J. WILBOR. Assrs. to W. J. MOHR (U.S.P. 1,898,774 and 1,898,830. 21.2.33. Appl., [A] 26.1.31, [B] 14.12.31).-(A) Highly adsorptive and absorbent plates of pure SiO, are extracted from vermiculite ore by treatment with acid followed by decantation of the liquor and gravity or flotation separation of the remaining solids. (B) A sulphate liquor prepared as in (A) is filtered from any remaining SiO<sub>2</sub> and treated with an alkali that forms an insol. sulphate sufficient in amount to ppt. Fe and Al; MgSO4 is then recovered by evaporation. Al(OH)3 may be leached out of the residue by caustic alkali and pptd. by CO<sub>2</sub>. B. M. V.

Manufacture of phosphorous pentachloride. W. V. WIRTH, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,906,440, 2.5.33. Appl., 27.11.31).—Cl<sub>2</sub> is passed into a suspension of P or PCl<sub>3</sub> in POCl<sub>3</sub> at 45—60°, so as to give PCl<sub>5</sub> in a finely-divided form particularly adapted for the manufacture of org. acyl chlorides.

W. J. W.

Production of sulphur from sulphides. R. F. BACON (U.S.P. 1,897,921, 14.2.33. Appl., 21.10.27).— As a preliminary step to known processes of recovery of S,  $H_2S$  is evolved by the action of HCl on, *e.g.*, FeS; afterwards the FeCl<sub>2</sub> is decomposed by steam with regeneration of HCl. B. M. V.

Recovering iodine from natural waters and the like. L. C. CHAMBERLAIN, Assr. to Dow CHEM. Co. (U.S.P. 1,897,031, 14.2.33. Appl., 2.3.31).—I having been released in the brine by known methods, it is removed by a current of air, adsorbed on charcoal, and recovered by superheated steam ( $\gg 300^{\circ}/1$  atm.) from which it is separated in liquid form after condensation.

The regeneration may be effected in two stages with saturated and superheated steam, respectively.

B. M. V. Mixing solids  $[CaCO_3]$  with liquids  $[H_2O]$ . Gas analyser.—See I. Recovering compounds from cooking liquors.—See V. B<sub>4</sub>C.—See VIII. Compositions for treating molten metals.—See X. Electrodes [for graphitic oxide].—See XI. Arsenical prep.—See XVI.

#### VIII.—GLASS; CERAMICS.

Mechanical analysis of raw material for ceramics. M. LARCHEVEQUE (Céramique, 1933, 36, 101-117; Chem. Zentr., 1933, ii, 269).—Sieve, elutriation (Schulze-Harkort), and pipette (Berg) analytical methods are applied to ceramic raw material. Shrinkage and porosity are determined after firing. A. A. E.

Plasticity, shrinkage, and other fundamental properties of clay in relation to the form of the clay particles. J. ARON (Notizbl. des Deuts. Vereins f. Fabrication von Ziegeln, Tohnwaaren, Kalk, u. Cement, 1873, 9, 167; Trans. Ceram. Soc., 1933, 32, 513-532).--Attempts to isolate by elutriation, from several German clays, the "clay substance" responsible for plasticity and shrinkage, and measurements of the drying shrinkage of the finest elutriated fractions are described. The relation between pore vol. and total vol. of the dried clay was approx. the same for several widely different clays, and roughly = that between vol. of unoccupied space and total vol. of spheres packed as closely as possible, suggesting that the clay particles were spherical, which was confirmed microscopically. The drying of clay ware and the plasticity and greasiness to the A. L. R. touch of clay are discussed.

Wear of metal in muller tyres in the clay industry. J. MAYER (Bull. Amer. Ceram. Soc., 1933, 12, 312—313).—A study of abrasion-resisting metals used in the ceramic industry showed that for the tyres of muller grinding pans the use of Mn steel offers no service advantage commensurate with its cost; in the generally used chilled-Fe tyres the amount of hard surface (white Fe) which can be applied is limited by the backing of softer grey Fe required. Chilled-Fe tyres wear concave owing to the centre of the face being softer than the corners, which often break off. Alloy-Fe tyres wear concave and often develop pockets by abrasion of soft spots. A new Cr-Mo steel, in which, when suitably heat-treated, toughness, strength, and hardness are combined, is described, which in 3-year service tests was equal or superior to other alloys.

A. L. R.

Passau graphite for refractories. E. BUCHHOLTZ (Feuerfest, 1933, 9, 140—142).—For the manufacture of melting crucibles (I) the refractory used must generally possess different properties from those of a refractory for furnace linings (II). Thus only graphite (III) in flakes can be used for (I), since amorphous (III) does not impart the necessary elasticity to the clay-(III) mixture used, whereas amorphous (III) gives the best results with (II). The development and growth of the (III) crucible industry in lower Bavaria, where Passau (III) is used, is outlined. A. R. P.

Rotary furnaces.—See I. Enamelled cast Fe.— See X

See also A., Dec., 1235,  $Al_2O_3$ -SiO<sub>2</sub> refractories. X-Ray examination of ceramic raw materials. 1245, M.-p. diagram of refractory oxide mixtures with CaO. 1247, SiO<sub>2</sub>-Na<sub>2</sub>O-BeO glasses. Melting region of SiO<sub>2</sub>-glass oxide systems. 1257, Syntheses of mullite. Burning and colouring of gem stones.

#### PATENTS.

Manufacture of glass mix for opaque containers. C. W. CROWELL (U.S.P. 1,899,230, 28.2.33. Appl., 3.10.30).—Opacifiers for Na<sub>2</sub>O-CaO or other glasses are formed from C, S, Pb (or PbO), and/or another metal or suboxide which forms a black sulphide, *e.g.*, Cu, Hg, Ni, Fe, Ag, Mo. The quantity of opacifier is  $\geq 2\%$  of the glass. B. M. V.

Melting of glass. H. A. WADMAN, ASST. to HART-FORD-EMPIRE Co. (U.S.P. 1,897,973, 14.2.33. Appl. (5.5.30).—In a glass tank heated by the electrical resistance of the glass between graphite electrodes, wells (W) are formed in the bed below each electrode and, if desired, connecting troughs, so that initial melting may be effected on a small quantity of glass, which may, if discoloured, be withdrawn through temporary tap holes leading from W. B. M. V.

Production of homogeneous glass in tanks. F. ECKERT, ASST. to W. A. MORTON and P. L. GEER (U.S.P. 1,898,039, 21.2.33. Appl., 3.1.28. Ger., 4.3.27). —The glass is passed from a melting chamber to a refining chamber through a submerged passage and then through another submerged port to the bottom of a comparatively small homogenising chamber provided with a mechanical stirrer and maintained at a temp. above that for casting; the glass leaves by an upper port and flows in a thin layer over an unheated forehearth to the casting machine. B. M. V.

Laminated glass. H. C. ROHLFS, ASST. to GEN. ELECTRIC Co. (U.S.P. 1,899,588, 28.2.33. Appl., 18.1.30). —The safety layer is composed of an alkyd resin formed from phthalic anhydride, glycerin, glycol, and succinic acid or other compounds of similar class. It is applied in a partly cured state, then cured at 110°/50 lb. per sq. in. for 5 min. followed by treatment at 70—75°/ 200 lb. per sq. in. B. M. V.

Glass treatment. F. G. SCHWALBE (U.S.P. 1,899,640, 28.2.33. Appl., 15.2.29).—Glass articles are annealed by the passage of electric current through them from depending curtains to the conveyor. The return run of the conveyor is also heated, by other means.

B. M. V.

Purifying art. [Removing contaminating iron from silica sand.] I. D. HUBBELL, Assr. to SILICA Co. OF CALIFORNIA, LTD. (U.S.P. 1,897,638, 14.2.33. Appl., 4.3.30).—The sand is treated in piles with a solution of  $H_2SO_4 \ge 10$  and  $SO_2 \ 2-4\%$ . A no. of percolations may be effected on the countercurrent principle for 24-hr. periods. B. M. V.

Production of a cellular clay body. T. HUSAIN and G. A. BOLE, Assrs. to CELLULAR CLAY CORP. (U.S.P. 1,897,667, 14.2.33. Appl., 17.5.30).—The clay is bloated while in the fluid state by means of gas generated from a carbonate and acid in the slip, the time of setting being shortened by addition of a hydrophile, *e.g.*, plaster of Paris or  $SiO_2$  gel. B. M. V.

Separation of grain particles from a bonded mass. O. HUTCHINS, ASST. to CARBORUNDUM CO. (U.S.P. 1,898,371, 21.2.33. Appl. 7.10.30).—SiC is recovered from ceramically bonded scrap by grinding and screening dry, followed by grinding and screening wet and by magnetic separation. B. M. V.

Manufacture of boron carbide. R. R. RIDGWAY, Assr. to NORTON Co. (U.S.P. 1,897,214, 14.2.33. Appl., 14.5.30).—The hard, strong, and very stable compound  $B_4C$ , free from parting planes of graphite and containing  $\lt 90\%$   $B_4C$ , is claimed, also its manufacture from  $2B_2O_3 + 7C$  heated in an electric-resistance furnace at a temp. in a central zone of 2400°, which is above the b.p. of B and the m.p. of  $B_4C$ . B. M. V.

Tunnel dryer.—See I.

# IX.—BUILDING MATERIALS.

Rotary [cement] kiln exit gases—some relationships. W. T. Howe (Cement, 1933, 6, 343—359). —The efficiency of the burning operation can be assessed from the analysis of the kiln exit gases. Mathematical equations are developed to show the relationship. It is shown that in the complete combustion of a given class of coal, the ratio  $(CO_2 + SO_2): N_2$  is const. In works' practice, the CaCO<sub>3</sub> content of the slurry is also maintained const. A simpler form of equation can therefore be derived and suitable nomograms constructed. T. W. P.

Change in viscosity of cement slurry on addition of alkali. H. SALMANG and H. E. SCHWIETE [with E. ZILG] (Zement, 1933, 22, 301—302; Chem. Zentr., 1933, ii, 430).—Addition of Na silicate produces increased viscosity (V) with lapse of time. Differences in behaviour are due chiefly to differences in CaO content. Increased V in absence of addenda is due to swelling of clay.

A. A. E.

Influence of the alumina modulus on the unsoundness of Portland cement caused by an excess of calcium sulphate. F. THLO (Zement, 1933, 22, 261-264).—Unsoundness through CaSO<sub>4</sub> is dependent on the constitution of the cement. Free aluminates react with CaSO<sub>4</sub> to give Ca sulphoaluminate and may cause unsoundness, but 4CaO,Al<sub>2</sub>O<sub>3</sub>,Fe<sub>2</sub>O<sub>3</sub> in cement does not react in this way. Cement will not deteriorate through exposure to sulphate waters if the Al<sub>2</sub>O<sub>3</sub> modulus is  $\leq 1$ . T. W. P.

Lime standard for Portland cement. H. KÜHL (Tonind.-Ztg., 1933, 57, 460–464; Chem. Zentr., 1933, ii, 268).—The practical limit is:  $CaO_{max} =$  $2 \cdot 8SiO_2 + 1 \cdot 1Al_2O_3 + 0 \cdot 7Fe_2O_3$ . A. A. E.

Influence of gypsum on soundness of highlime clinker. F. F. TIPPMANN (Tonind.-Ztg., 1933, 57, 125—126).—High-Ca clinkers may be unsound through the presence of free CaO. The latter slowly hydrates to amorphous Ca(OH)<sub>2</sub>, but gypsum catalyses the hydration to give cryst. Ca(OH)<sub>2</sub>, with a resulting increase in compressive strength. T. W. P.

an entrance month

63

Manganese in Portland cement clinker. O. GOFFIN and G. MUSSGNUG (Zement, 1933, 22, 218–221, 231–234; Chem. Zentr., 1933, ii, 268).—Mn may replace Fe; it is present as  $Mn_2O_3$ ,  $Mn_2O_3 + MnO_2$ , or  $Mn_3O_4 + MnO$ , depending on the grain size. The oxidation of the Mn takes place chiefly after sintering. High Mn contents reduce the setting time. A. A. E.

Binding qualities of mixtures of clay-cement and (A) calcified clay, (B) Portland cement. A. V. FILOSOFOV (J. Appl. Chem. Russ., 1933, 6, 825—828, 829—831).—(A) The mechanical strength (I) of blocks made from sand and clay-cement (II), prepared from powdered brick and Ca(OH)<sub>2</sub>, is unaffected by adding  $\geq 20\%$  of a mixture of unfired clay and Ca(OH)<sub>2</sub>.

(B) Addition of 20-25% of Portland cement (III) to (II) yields a product the (I) of which equals that of low-grade (III).

Volumetric determination of MgO in presence of Ca in cement, dolomite, and dolomitic limestones. S. S. KOROL (J. Appl. Chem. Russ., 1933, 6, 976—979).—Diener's method (Concrete, 1931, No. 6, 47) is to be preferred. R. T.

Chemistry of Australian timbers. III. Chemical composition of four pale-coloured woods of the genus Eucalyptus : E. gigantea, E. obliqua, E. regnans, E. sieberiana. W. E. COHEN, A. G. CHARLES, and A. B. JAMHESON (Counc. Sci. Ind. Res., Australia, Pamph., 1933, No. 44, 22 pp.; cf. B., 1932, 1081).—Analyses indicate certain characteristic differences which may serve to identify the species. Data bearing on the pulping val. of woods are discussed.

A. G. P.

Swelling of wood in water and aqueous solutions. C. G. SCHWALBE and W. BEISER (Papier-Fabr., 1933, 31, 655-667) .- Various methods of measuring the swelling of pine- and beech-wood both in the form of discs and by the sedimentation vol. of a fixed quantity of sawdust are described. Swelling is governed to the extent of 50% by the proportion of spring and summer wood present in the sample, the breadth of the respective rings, as well as the d, and the length and method of drying the wood prior to the determination. Acids and bases both increase the swelling as compared with distilled H<sub>2</sub>O, which is especially marked in the case of sawdust, where 0.1% H<sub>2</sub>SO<sub>3</sub> solution has the greatest effect, 1% HCl has no effect, 1% H2SO4 has little, whilst bases have an effect intermediate between HCl and  $H_2SO_3$ . These differences cannot be detected when the wood has been rapidly dried. The rate of swelling in H<sub>2</sub>O is much more rapid than in H<sub>2</sub>O vapour. D. A. C.

Mastic asphalt roofing. D. M. WILSON (Chem. & Ind., 1933, 959—960).—Mastic asphalt roofing that had eracked (I) after short service was found to contain a smaller quantity of mineral aggregate < 200-mesh than good roofing. The aggregate of (I) consisted of cryst. limestone which had a lower bitumen-carrying capacity than the asphalt-rock aggregate. D. K. M.

Rotary furnaces.—See I. Paint failure on plaster. Priming coats for new wood. Paints for acoustical treatment.—See XIII. Fodder from wood.—See XIX. See also A., Dec., 1246, Hydrolytic decomp. of 3CaO,SiO<sub>2</sub>.

# PATENTS.

Plants for use in burning [Portland] cement or like materials. N. S. BORCH (B.P. 401,726, 18.5.32).— The raw constituents are passed through a preheater down an inclined path formed by a no. of gas-permeable rotary rollers (R) with interspaced stationary guides. The chamber may be divided by a transverse partition which directs the gases to the underside of the R in the upper section, in which the R are placed on a higher plane. C. A. K.

Burning of cement. R. W. P. HORN and J. N. NISSLEY (U.S.P. 1,898,776, 21.2.33. Appl., 22.9.27).— A rotary kiln is formed with a main section of full diam. and a cooling section of reduced diam. connected by a tapering section in which most of the combustion takes place, but supplies of air and fuel are also admitted through the shell at a no. of distributed points.

B. M. V.

Heat-insulating cement. L. R. NICHOLS (U.S.P. 1,899,473, 28.2.33. Appl., 21.3.31).—A composition adherent to metal comprises a mixture of soot and a hydraulic cement, e.g., >50% of flue soot and <50% of asbestos–MgO cement. B. M. V.

(A) Protecting concrete surfaces during curing. (B) Curing concrete. (A) P. C. LEMMERMAN and R. F. REMLER, (B) W. K. SCHWEITZER, Assrs. to (A, B) GRASS-ELLI CHEM. Co. (U.S.P. 1,899,576 and 1,899,590, 28.2.33. Appl., [A] 13.11.30, [B] 17.11.30).—Green concrete is treated with Na silicate containing an amount of weak non-caustic electrolyte insufficient to cause actual pptn. of SiO<sub>2</sub>, the addition being (A) AcOH or NaOAc, and (B) NH<sub>4</sub>Cl. B. M. V.

Hardening of lime-mortar. R. W. GELDER (U.S.P. 1,898,358, 21.2.33. Appl., 10.1.29).—CaSO<sub>4</sub>,  $\frac{1}{2}$ H<sub>2</sub>O and minute quantities of MgCl<sub>2</sub> and MgSO<sub>3</sub> are added. B. M. V.

Plaster composition. J. S. OFFUTT, Assr. to UNITED STATES GYPSUM CO. (U.S.P. 1,897,956, 14.2.33. Appl., 13.3.30).—A finishing coat is composed of finely-ground CaSO<sub>4</sub>, ${}_{2}^{1}$ H<sub>2</sub>O 20—40 (30), Ca(OH)<sub>2</sub> 10—30 (20), finely-ground limestone 35—55 (47), ground asbestos 2—5 (3)%, and a retarder. B. M. V.

Manufacture of [plaster] models of jaws. L. Köhler, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,898,731, 21.2.33. Appl., 19.2.31. Ger., 3.3.30).— As a separator between a plaster of Paris mould and a modelling composition (which may also be plaster of Paris) the use of paint composed of aq. emulsion of vulcanised rubber is claimed. B. M. V.

Anhydrite plastic material. H. K. LINZELL, ASST. to UNITED STATES GYPSUM Co. (U.S.P. 1,898,636, 21.2.33. Appl., 16.2.31).—Natural CaSO<sub>4</sub> is hydrated by grinding at 40° in an aq. solution of an accelerator having  $p_{\rm H} \gg 7$ ; after neutralising the acid with CaO and decanting off excess liquor, the sulphate is dehydrated to CaSO<sub>4</sub>, $\frac{1}{2}$ H<sub>2</sub>O at  $> 78^{\circ}$  under pressure and the slurry immediately cast as desired. B. M. V.

Coating material [for walls]. F. C. ATWOOD (U.S.P. 1,898,518, 21.2.33. Appl., 25.10.28).—A size

for walls comprises gum arabic with a minor proportion of Na<sub>2</sub>SiO<sub>3</sub> and/or a pyrophyllite clay. B. M. V.

Impregnation of wood. C. SCHMITTUTZ (B.P. 402,023, 21.7.33. Ger., 25.7.32).—A growing tree may be impregnated for preservation or colouring of the wood by removing a portion of the bark, coating it with a  $H_2O$ -sol. substance (e.g., mycocide salt) in paste form, and covering it with a waterproof bandage. C. A. K.

Manufacture of sawdust wood-fibre board. J. V. NEVIN, Assr. to PACIFIC LUMBER Co. (U.S.P. 1,899,768, 28.2.33. Appl., 22.12.30).—Wood waste or bark fibres are dried to zero  $H_2O$ , mixed with 10—20% of resin-forming compounds (and a neutral catalyst if desired), compressed to form the board, and further compressed at, e.g., 175—200°/500—1000 lb. per sq. in. B. M. V.

Calcining gypsum.—See VII. Treating woody material.—See XI.

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

Metallurgical reactions. W. OELSEN (Z. tech. Physik, 1933, 14, 222—226; Chem. Zentr., 1933, ii, 274).—Fundamental considerations are discussed. A. A. E.

Effect of superheating on cast iron of different compositions. O. VON KEIL and A. LEGAT (Giesserei, 1923, 20, 214—217; Chem. Zentr., 1933, ii, 275).—The effect of addition of Si, Ni, and Al on the form of the separated graphite has been studied. The presence of highly dispersed slag nuclei is specially important. Separation is favoured by superheating. A. A. E.

X-Ray study of the diffusion of chromium into iron. L. C. HICKS (Amer. Inst. Min. Met. Eng., Contrib. No. 58, 1933, 10 pp.).—Results obtained by X-ray examination of specimens of electrolytic Fe heated in close contact with Cr powder in vac. confirm, in general, the work of earlier investigators. The Cr content of the cemented layer (1200°) decreases with increasing depth in a smooth curve until 15% Cr is reached, when there is an abrupt drop to < 1% Cr. The boundary line occurs at 8% Cr with a cementation temp. of 1350°, due to the narrowing of the  $\gamma$ -field at high temp. A. R. P.

Significance of analysis with regard to the enamelability of cast iron. A. KRÄUTLE (Emailwaren-Ind., 1933, 10, 109—113; Chem. Zentr., 1933, ii, 266). —The effect of impurities on strength, expansion, and surface quality, and therefore on enamelability, is discussed. A. A. E.

Examination of steel exposed to sea-water for 31 years. J. S. UNGER (Eng. News-Rec., 1933, 111, 593—594).—Steel work which has been subjected during this period to the action of sand and sea-H<sub>2</sub>O without maintenance showed that piles originally 6 in. in diam. lost  $\frac{1}{18}-\frac{1}{2}$  in. in thickness, the greatest loss being above the H<sub>2</sub>O level, and angle irons (containing C 0.25, Mn 0.43, P 0.02, S 0.029%, Cu trace) were corroded to a greater extent, due to the greater amount of surface exposed compared with its mass. D. K. M. Smelting operations at Roan Antelope Copper Mines, Ltd. C. R. WRAITH (Amer. Inst. Min. Met. Eng., Tech. Publ. No. 511, 1933, 25 pp.).—In the reverberatory smelting the charge contains 8-10% H<sub>2</sub>O, the fuel consumption averages  $15 \cdot 5\%$ , the matte fall 70% and the slag fall 26%, the CaO used to give a bisilicate slag with up to 15% Al<sub>2</sub>O<sub>3</sub> is 6%, and the Cu produced is  $53 \cdot 8\%$  of the charge, using concentrates containing 58% Cu. The slag contains  $1 \cdot 2\%$  Cu and the matte  $78 \cdot 7\%$  Cu; the total Cu recovery is nearly 98%. Details of the operation of the converters are also given. A. R. P.

Experimental combination of shaft roasting and reverberatory smelting [of copper sulphide ore]. F. LAIST and J. P. COOPER (Amer. Inst. Min. Met. Eng., Tech. Publ. No. 510, 1933, 8 pp.).-The experimental plant comprised an oil-fired reverberatory (I) with a hearth 21 by 3 ft. and a roasting shaft (II) 3 ft. square and 20 ft. high built above the firing end of (I). Suitable fluxed flotation concentrates were fed with cold air over a spreading device at the top of (II) and the roasted calcines fell directly in front of the burners in (I); in this way considerable heat economy was effected since the charge entered (I) at  $> 850^{\circ}$ . The output of (I) with (II) in operation was  $2\frac{1}{2}$  times that of (I) without (II) with the same oil consumption, *i.e.*, the fuel consumption per ton of charge was decreased by 60%. A. R. P.

Purification of the electrolytes in copper refining. E. S. BARDWELL and R. J. LAPEE (Amer. Inst. Min. Met. Eng., Tech. Publ. No. 512, 1933, 12 pp.).-A certain vol. of the electrolyte is removed daily, boiled down in a series of 28% Cr-steel pans heated with natural gas until the d reaches 1.45, and set aside for the CuSO<sub>4</sub>,5H<sub>2</sub>O to crystallise. The remainder of the Cu is removed from the mother-liquor, together with the As and Sb, by electrolysis with insol. anodes, and the solution returned direct to the tanks or boiled down to  $d \ 1.55$  and cooled to separate NiSO4 and FeSO4. Steam-heated coils in tanks for evaporation were found to be unsatisfactory since they rapidly became encrusted with basic salts of Fe and Ni containing Sb and As. Details of the operation of this plant at Great Falls, Montana, over 3-4 years are tabulated and discussed. A. R. P.

Volumetric determination of copper and lead in babbitt metal. E. S. GAVRILENKO and V. S. KOBZARENKO (J. Appl. Chem. Russ., 1933, 6, 945—947).—The alloy is digested with HNO<sub>3</sub>, the solution, containing Cu and Pb, is treated with excess of  $(NH_4)_2C_2O_4$ , the ppt. of PbC<sub>2</sub>O<sub>4</sub> (I) is dissolved in H<sub>2</sub>SO<sub>4</sub>, and Pb is determined by titrating the H<sub>2</sub>C<sub>2</sub>O<sub>4</sub> formed with KMnO<sub>4</sub>. The filtrate from (I) is made acid with HCl, and Cu is determined iodometrically. R. T.

Mechanical properties of metals at low temperatures. II. Non-ferrous metals. E. W. COLBECK and W. E. MACGILLIVRAY (Inst. Chem. Eng., Nov., 1933. Advance proof, 13 pp.; cf. B., 1933, 590).—The ultimate strength, proof stress, elongation, and reduction in area of annealed Cu, Ni, Al, Cu-Ni (80/20, 55/45, and 30/70), "Y" alloy, brass (70/30), Ni-silver, Mn- and Al-bronze, and solder at temp. between 20° and —180° have been measured. Vals. are tabulated. D. K. M. Influence of impurities on the technical properties of zinc. W. HERRMANN (Chem.-Ztg., 1933, 57, 958—959).—The effects of the common impurities found in Zn on its rolling, drawing, etc. properties are described. D. A. C.

Working of nickel. G. HAMPRECHT and L. SCHLECHT (Metallwirt., 1933, 12, 281–284; Chem. Zentr., 1933, ii, 118).—Ni obtained by thermal decomp. of Ni(CO)<sub>4</sub> is heated at  $1000-1200^{\circ}$ , when the fine particles (0.0005-0.005 mm.) readily sinter and form larger crystallites, which are then compressed. A. A. E.

Removal of gases and other impurities from aluminium and its alloys by means of aluminium chloride with other salts. STEINHÄUSER (Metallwirt., 1933, 12, 284–285; Chem. Zentr., 1933, ii, 119).—A suitable flux is composed of AlCl<sub>3</sub> and NaCl in approx. equimol. proportions. A. A. E.

Mechanical and chemical properties of alloys of aluminium with chromium, iron, magnesium, manganese, titanium, and vanadium. H. BOHNER (Metallwirt., 1933, 12, 251–255, 265–267; Chem. Zentr., 1933, ii, 120).—Optimal mechanical properties are conferred by  $1 \cdot 0$ — $1 \cdot 5\%$  Cr or Mn, and  $0 \cdot 5\%$  Ti and V. Addition of  $1 \cdot 0\%$  Mg or  $0 \cdot 10 - 0 \cdot 15\%$  Ti to the binary alloys confers desirable properties. An Al-Mn-Ti alloy has greater chemical resistance than Al or Al-Mn alloys; the addition of 1% Mg to the ternary alloy greatly increases this resistance. Greatest resistance to corrosion was exhibited by Al-Mg-Cr-Ti alloys.

A. A. E.

Analysis of antifriction alloys. P. FOERSTER (Ann. Chim. Analyt., 1933, [ii], 15, 441-446).—The alloy is attacked with fuming  $HNO_3$  and the insol. oxides of Sn and Sb are digested with conc. Na<sub>2</sub>S, aliquot portions of the solution being used for the iodometric determination of Sn and the titration of Sb with  $KMnO_4$ . The insol. sulphides from the Na<sub>2</sub>S treatment are dissolved in  $HNO_3$  and the solution is added to the main filtrate, from which Cu and Pb are removed by electrolysis. The spent electrolyte is treated with aq. NH<sub>3</sub> to remove Fe and the Zn determined as  $ZnNH_4PO_4$  if > 1%, or by pptn. with  $(NH_4)_2S$ , dissolution of the ZnS in HCl, evaporation of the solution with HgO, and ignition of the residue to ZnO. A. R. P.

Preparation of iron electrolytically from magnetic ore. J. J. PITARD (Arch. Sci. phys. nat., 1933, [v], 15, 418–435).—The mineral is decomposed when employed as anode in an electrolyte of conc. HCl, Fe<sup>•</sup> NH<sub>4</sub> sulphate solution being used as the cathode solution, but  $Cl_2$  is evolved and the process is uneconomic. With 50%  $H_2SO_4$  as the anode liquid a deposit of  $Fe_2(SO_4)_3$  is formed and Fe is deposited at the cathode with a current yield of 60—70%, but production of 1 kg. of Fe requires about 30 kw.-hr. The production of Fe industrially by a direct electrolytic method thus appears to be entirely impracticable. H. F. G.

Platinum-plating of noble and base metals. E. R. THEWS and R. W. HARBISON (Chem.-Ztg., 1933, 57, 980—981).—The economics of Pt-plating are discussed and the prep. of ammino-phosphate and -nitrite baths is described. A. R. P. Analysis of chromium[-plating] baths. A. Wog-RINZ (Oesterr. Chem.-Ztg., 1933, 36, 187; cf. B., 1933, 710).—(a) To the diluted solution 2 drops of  $HNO_3$  and excess of  $Hg(NO_3)_2$  are added, the mixture is heated and filtered, the ppt. ignited in a Ni crucible, melted with  $Na_2O_2$ , dissolved in dil.  $H_2SO_4$ , KI added, and I titrated with  $0\cdot 1N$ -Na $_2S_2O_3$ . (b) The solution is evaporated to dryness with 30% NaOH, fused with  $Na_2O_2$ , filtered, and the filtrate titrated as under (a). E. S. H.

Rotary furnaces.—See I. Metal for muller tyres. See VIII. Rust-inhibitive primers and paints.— See XIII.

See also A., Dec., 1234, Lattice consts. of pure Fe and Fe-Cu alloys, and of Al-Cu alloys. 1236, Variation of Curie point in metallic solid solutions. Change of magnetic susceptibility of Pt, Cu, and Ag caused by cold-working. 1238, Properties of Cu alloys, Ni-Cr, Au-Cu, Sb-Pb, Sn-Pb, Mg alloys, Ag-Cu, and Pd-H<sub>o</sub>. Structure of eutectics and rate of cooling. Influence of magnetic fields on hardening of metals and alloys. System Fe-Ni-Al. 1239, System Fe-Co-Al. α-γ-Transformation in ternary Fe systems. Resistance of H<sub>2</sub>-charged Pd-Ag and -Au alloys. Superconductivity of alloys. Use of Th-B in the solution of metallographic problems. 1251, Corrosion of metals. 1254, Anodic films of Al. Deposition of Be on Cu etc. 1266, Precision wts. of Mo-Cr-Ni alloy.

#### PATENTS.

Metallurgical apparatus. C. R. KUZELL, Assr. to UNITED VERDE COPPER Co. (U.S.P. 1,899,161, 28.2.33. Appl., 2.11.29).—A furnace similar to a converter is heated by, and the metal treated with, gases generated from air and fuel in combustion chambers attached to the tuyères. B. M. V.

Blast-furnace operation. H. A. BRASSERT, Assr. to H. A. BRASSERT & Co. (U.S.P. 1,897,750, 14.2.33. Appl., 18.9.31).—The temp. of the top of the blast furnace and upper part of the charge is kept const. by H<sub>2</sub>O-sprays. B. M. V.

Smelting of fine [iron] ores, blast-furnace dust, and the like. P. JUNG (B.P. 400,933, 30.4.32. Ger., 2.5.31).—The material is mixed with the necessary fluxes and with finely-divided bituminous coal sufficient for smelting, the mixture is briquetted with or without addition of a binder, and the briquettes are coked after being embedded in fine coke to prevent cracking.

Mixing a furnace charge. C. HART, Assr. to W. S. JACKSON (U.S.P. 1,897,647, 14.2.33. Appl., 6.12.30).— A rotary furnace with internal ribs, for mixing a wrought-Fe puddling charge, is described, the material being caused to have zig-zag end-wise as well as circumferential motion. B. M. V.

Apparatus for separating metals or alloys. A. HANAK, ASST. to GIRARD SMELTING & REFINING Co. (U.S.P. 1,899,031, 28.2.33. Appl., 11.4.30).—A liquation furnace has an inclined, perforated bed which is violently agitated in a vertical direction, bumping against a fixed grid at the bottom of the stroke, so as to cause the

A. R. P.

unmelted metal to progress over and the melted metal to pass through the bed. B. M. V.

Annealing art. L. L. LEWIS, ASST. to BLAIR STRIP STEEL Co. (U.S.P. 1,898,674, 21.2.33. Appl., 3.9.30).— A pot for bright annealing is provided with a powder seal which is permeated (from a perforated pipe) by a neutral or reducing gas; a supply of the gas may also be led to the interior of the pot and means provided for exhaustion of fumes. B. M. V.

**Treatment of** (A) **sulphide**, (B) **iron**, **ores.** E. W. WESCOTT, ASST. to SULPHIDE CORP. (U.S.P. 1,898,701–2, 21.2.33. Appl., [A] 6.4.27, [B] 16.2.28).—(A) Fe sulphide ore is treated, in a first stage (I), with dil. Cl<sub>2</sub> sufficient to form FeCl<sub>2</sub> but not FeCl<sub>3</sub> or SCl<sub>4</sub> and at a temp. to volatilise S but not FeCl<sub>2</sub>. The FeCl<sub>2</sub> and gangue are transferred to a second stage and rechlorinated to produce FeCl<sub>3</sub> which is then (II) oxidised to Fe<sub>2</sub>O<sub>3</sub> and Cl<sub>2</sub>(+ N<sub>2</sub>) for re-use. (B) SCl<sub>4</sub> may be the primary source of Cl<sub>2</sub> for stage (I) only, and 350° is claimed to be a suitable temp.; in (II) the gases may be heated by combustion of fuel among them, the excess gas (which must be wasted) being passed through S in a SCl<sub>4</sub> generator and then through stage (I). B. M. V.

Conditioning of incoherent finely-divided materials. (B) Composition for and method of treating molten metals. W. R. JEAVONS and M. J. RENTSCHLER (U.S.P. 1,899,152—3, 28.2.33. Appl., [A] 14.11.30, [B] 19.5.32).—(A)  $BaO_2$  is rendered suitable for use in steel refining by the addition of a small proportion of BaO and equiv. cold  $H_2O$  and pressing. (B) BaO with  $Ba(OH)_2$  (but no free  $H_2O$ ), in proportion varied according to the m.p. desired, is fused (without altering the composition) and cast into masses suitable for metal refining. B. M. V.

**Refining of iron and steel.** W. W. TRIGGS. From E. V. DAVIES (B.P. 400,593, 15.3.32).—The molten metal is treated with a mixture of  $KClO_3$  12—20 (12),  $Na_2B_4O_7$  2—16 (8), NaF and/or  $Na_2CO_3$  2—8 (8), and  $MnO_2$  1—4 (4) pts. The mixture is preferably packed in a grey-Fe cylinder which is plunged below the surface of the bath. A. R. P.

Removal of oxides [scale] from ferrous metal. F. STOCKTON, ASST. to AMER. SHEET & TIN PLATE CO. (U.S.P. 1,899,734, 28.2.33. Appl., 18.1.30).—Previous to pickling, stainless steel (*e.g.*) is placed in a hot solution of NaOH 20 and  $\text{KMnO}_4$  1% (approx.). B. M. V.

Covering of metal [iron] surfaces. ARMSTRONG CORK Co., Assees. of R. M. ALLARD (B.P. 400,744, 9.8.32. U.S., 9.10.31).—Metal (e.g., Fe) plates for the decks of ships are coated with corrosion-inhibiting asphalt paint, which is allowed to dry, and linoleum or cork piles are then affixed to the treated surface by means of a manila gum cement. A. R. P.

Production of substantially carbon-free [chromium – nickel – iron] alloys. HERAEUS VACUUM-SCHMELZE A.-G. (B.P. 401,094, 5.10.32. Ger., 9.10.31).— High-C alloys of Fe, Cr, and Ni produced by any of the usual methods are decarburised by melting with  $Cr_2O_3$ in an atm. of  $H_2$  containing, if necessary to reduce oxides present, carbonaceous gases in quantity insufficient to carburise the metal. The process may be used for making C-free alloys of Cu, Co, Ni, or Fe with Cr, Mo, W, U, V, Ta, Ti, and Zr. A. R. P.

Treatment of mineral [by flotation]. R. B. MARTIN (U.S.P. 1,897,265, 14.2.33. Appl., 13.7.28).— A collecting agent comprises an NH<sub>4</sub> Et xanthate formed in the conditioned (frothed) pulp from the Na ester and NH<sub>4</sub>Cl, added in that order. B. M. V.

Flotation of ores. R. FROMM (B.P. 401,619, 30.6.33. Ger., 30.6.32).—The phosphatide prepared from soya beans, or the mud obtained as a by-product in the manufacture of soya oil, is used as a froth accumulator. A. R. P.

Condensation product of cinchona bark alkaloids with thiuram sulphides as inhibitors [in metalpickling baths]. R. E. LAWRENCE, ASST. to GRASSELLI CHEM. Co. (U.S.P. 1,905,915, 25.4.33. Appl., 19.12.31). —For preventing dissolution of Fe or steel when removing scale in an acid-pickling bath, a small quantity of the product obtained by heating the alkaloids with tetramethylthiuram mono- or di-sulphide is added to the acid. A. R. P.

**Refining copper.** ROESSLER & HASSLACHER CHEM. Co. (B.P. 401,008, 9.5.32. U.S., 9.5.31).—The excess Ca remaining in Cu after deoxidation is removed by blowing N<sub>2</sub>, H<sub>2</sub>, or particularly NH<sub>3</sub> through the molten metal. NH<sub>3</sub> will reduce the Ca content from 0.2% to < 0.005% in 5 min., H<sub>2</sub> in about 10 min., and N<sub>2</sub> in > 30 min. A. R. P.

Manufacture and usage of non-corrosive alloy. W. J. HAWKINS, ASST. to AMER. MACHINE & FOUNDRY CO. (U.S.P. 1,899,148, 28.2.33. Appl., 2.5.28).—A method of protecting Cu and Fe comprises applying a first coat, at 468°, of Pb alloyed with Zn 4.5 and Al 0.5%, and a second coat, at 399°, of Pb alloyed with 2% of phosphor-Sn. B. M. V.

Heat-treatment of metals [brass]. H. W. BROWNS-DON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 400,586, 13.4.32).—The metal is passed successively through a preheating zone (I), a heat-treatment zone (II), and a cooling zone (III), all of which are filled with a granular or finely-divided, heat-conducting substance, *e.g.*, graphite, metal powder, or fine metal shot, which does not react with the metal; the direction of movement is frequently reversed to exchange the functions of (I) and (III). For brass (II) is kept at 600—650°. A. R. P.

Cadmium-nickel alloys. C. E. SWARTZ and A. J. PHILLIPS (B.P. 401,229, 15.6.33. U.S., 30.6.32).— Alloys of Cd with 0.25—7% Ni and  $\geq 3\%$  of  $\ll 1$  of the metals Al, Sb, Cu, and Mg are claimed, particularly for use as bearing metals. A. R. P.

[Silver] alloys. R. H. LEACH, ASST. to HANDY & HARMAN (U.S.P. 1,899,701 and 1,899,873, 28.2.33. Appl., [A] 12.7.32, [B] 13.8.32).—A brazing spelter is composed approx. of (A, B) Ag 48—52, Cu 12—16, Zn 14—18, Cd 16—20%. (B) When no flux is to be used 0.5—2.0 of P may be included. B. M. V.

 Stainless silver alloy.
 T. TANABE (B.P. 401,527, 21.1.33).

 The alloy consists of Ag 60—92, Cd 20—4, and Sn 20—4%.
 A. R. P.

Foundry moulding sands. BINDERS INTERNAT. Co., LTD. From Soc. L'AvéBENE (B.P. 401,217, 30.5.33). —A binder for moulding sand, especially regenerated sand, comprises a mixture (3:1) of coal dust and the dried and pulverised product from sulphite-cellulose liquor. A. R. P.

Attaching portions of moulds or cores [for high-m.-p. metals]. C. M. SAEGER, JUN. (U.S.P. 1,897,149, 14.2.33. Appl., 17.2.30).—An adhesive core paste comprises a rubber-containing compound which becomes vulcanised at the drying temp. and disintegrated at the casting temp. B. M. V.

Uniting of metal parts. [Brazing spelter.] R. T. HURLEY (U.S.P. 1,898,487, 21.2.33. Appl., 23.3.32).— A brazing spelter is composed of Cu and <5% of Be. In use the cooling should be delayed at 300—314° for max. strength, which may be 154,000 lb./sq. in.

B. M. V.

Welding rod. H. W. R. BIERS, ASST. to OXWELD ACETYLENE Co. (U.S.P. 1,898,933, 21.2.33. Appl., 14.3.31).—A metal rod is provided with an electrically conducting coating composed of C  $2 \cdot 0 - 10 \cdot 0$  (5 $\cdot 6$ ), Zr 25-65 (30 $\cdot 0$ ), Si 20-60 (5 $1 \cdot 7$ ), alkaline-earth metal or oxide  $2 \cdot 0 - 15 \cdot 0$  (8 $\cdot 0$ )%. B. M. V.

(A) Welding rod. (B) Welding electrode. G. T. SOUTHGATE, ASST. to UNION CARBIDE & CARBON RESEARCH LABS., INC. (U.S.P. 1,898,908—9, 21.2.33. Appl., 10.2.31).—Rods for arc-welding are composed of an Fe core with a conducting coating of (A) graphite 1 pt., SiO<sub>2</sub> 3 pts., and 4—9 pts. of a Ca oxy-salt of Cr, Mo, W, Ti, Ce, or Zr, or (B) CaO with the above N<sub>2</sub>-combining metals in metallic form. B. M. V.

Electric arc-welding process for welding aluminium or aluminium alloys. M. ZACK (B.P. 401,487, 24.10.32. Ger., 3.11.31).—Three-phase current is used, the work being connected to one terminal and two graphite electrodes to the others; during welding the work is covered with a flux composed of KF 30,  $Na_3AlF_6$  30, and  $Al_2O_3.2SiO_2.2H_2O$  40%. A. R. P.

Melting and casting [small quantities] of metals. H. T. REEVE, ASST. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,897,589, 14.2.33. Appl., 29.10.30).—A metal to be cast in the absence of air is melted in a crucible in one limb and poured (by tilting) into a mould in the other limb of an L-shaped quartz tube which is maintained under vac. and heated by high-frequency induction. B. M. V.

Manufacture of an additional material for hardsoldering and welding purposes. H. KANZ (B.P. 400,817, 2.2.33. Switz., 4.2.32).—Brass rods for brazing are made by coating Cu rods or filling Cu tubes with Zn so that the composite material contains 42—58% Cu, and then swaging, rolling, or drawing to the desired size. The Cu may be replaced by a high-Cu brass and may contain Si, Al, or Mg as a protection against oxidation during use. A. R. P.

Protecting articles of oxidisable metals or metal alloys from oxidation, deterioration, or corrosion. METALLISATION, LTD., and R. A. PARKES (B.P. 400,752, 31.8.32).—The article is first sprayed with Cd, Sb, Bi, Pb, Mg, Sn, or Zn, and then with Al, or it may be sprayed with alternating thin coats of one of the first group of metals and Al, or with an Al alloy. The coated material is then heated to  $650-1200^{\circ}$  to produce diffusion of the coatings. Before heating, the article may be sprayed with bitumastic paint to produce a C coating after heating. A. R. P.

Light aluminium alloy. M. BOSSHARD, ASST. to ALUMINIUM IND. A.-G. (U.S.P. 1,899,133, 28.2.33. Appl., 25.11.31. Ger., 3.12.30).—Al is alloyed with Mg 0.2— 1.0, Mn 0.2—1.0,  $\lt$  1 of the elements Cd, Sb, As, Bi 0.05—2.00, and Si  $\Rightarrow 5.0\%$  but enough to combine with all the Mg and Mn. B. M. V.

Airplane propeller. R. J. NORTON, ASST. to BENDIX AVIATION CORP. (U.S.P. 1,899,631, 28.2.33. Appl., 28.5.29).—An Al-Be-Si or Al-Be-Mg alloy, heattreated and aged, is used. B. M. V.

Magnesium-base alloys. Dow CHEM. Co. (B.P. 401,244, 20.7.33. U.S., 21.7.32).—Claim is made for Mg alloys containing Al 8—15, Si 0.35—1, and Mn 0.1—0.5%. The alloys can be readily die-cast and have a good resistance to corrosion and good mechanical strength. A. R. P.

Casting of readily oxidisable metals such as magnesium and alloys thereof. J. A. GANN, J. B. REID, and H. A. REIMERS, ASSTS. to DOW CHEM. Co. (U.S.P. 1,898,437, 21.2.33. Appl., 4.5.32).—Moulding sand is mixed with 1—10 (3—7)% of at least one of the fluorides of Al, Sb, Bi, Ca, Cr, Fe, H, Zn. B. M. V.

Casting of readily oxidisable metals. A. W. WINSTON, ASST. to DOW CHEM. Co. (U.S.P. 1,899,906, 28.2.33. Appl., 20.12.29).—Bituminous coal dust is mixed with H<sub>2</sub>O-bound greensand or is applied as a facing. Other non-aq. materials having free C may be substituted. B. M. V.

Barrel [electro-]plating apparatus. C. G. FINK and R. D. EASH, ASSTS. to CHEM. TREATMENT Co., INC. (U.S.P. 1,899,679, 28.2.33. Appl., 3.6.26).—An apparatus suitable for plating with high c.d. comprises a rotating, perforated, polygonal or cylindrical cathode barrel which only dips into the bath, and a stationary anode resting inside the barrel. The no. of tumbles should be  $\gg$  15 per min. and, for Cr, the bath should contain 200—300 g. CrO<sub>3</sub> per litre. B. M. V.

Electro-cleaning process. T. E. DUNN, Assr. to BULLARD CO. (U.S.P. 1,898,765, 21.2.33. Appl., 20.6.32).— The work (Fe or steel) is made the cathode in a bath which will emit  $H_2$  and simultaneously plate a protective film of metal (Sn), both sol. and insol. anodes being provided, the latter becoming surrounded by a colloidal film of metastannic acid which prevents any Fe<sup>\*\*</sup> reaching the anode. B. M. V.

Pulverising (etc.) ores.—See I. Combustion in shaft furnaces.—See II. Dithiophosphates [as flotation agents].—See III. Electrolysis of fused baths [Mg]. Electrode alloy. Welding electrode: Coating radiant bodies. Conducting Cu coatings. —See XI. Pigmentary-base product.—See XIII.

## XI.—ELECTROTECHNICS.

Testing of graphite anodes for chlorine production. I. J. SIRAK (J. Appl. Chem. Russ., 1933, 6, 808-822, 823-824).—Graphite (I) electrodes are tested

for resistance to corrosion by determining loss in wt. after electrolysis of 1.5-6% NaCl under specified conditions. Corrosion of such anodes augments with their porosity and sp. conductivity, and with the degree of polarisation on electrolysis of dil. aq. NaCl. (I) has no depolarising action on O at the anode. Disintegration of (I) is due chiefly to formation of graphitic acid (II) in the pores, but also to oxidation to  $CO_2$  by OH' and OCl'. The nature and degree of protection of (I) by impregnation with various substances is approx. proportional to the yield of (II) obtained on electrolysis of 10% HNO<sub>3</sub>, with a c.d. of 100 amp. per sq. m. For practical purposes, conditions of testing should be strictly analogous to those obtaining in factory practice. R. T.

Development of the Söderberg electrode in the manufacture of calcium carbide. W. BLIEMEISTER (Chem.-Ztg., 1933, 57, 841—843).—The construction and use of modern types of this electrode for CaC<sub>2</sub> production are described. A. R. P.

Cu refining. Analysis of antifriction alloys. Pt-plating. Prep. of electrolytic Fe. Cr-plating baths.—See X. Treating polysaccharide solutions. —See XVII.

See also A., Dec., 1236, Relation between temp. and heat loss from heated wires. 1247, New electrolytic element. 1254, Reduction of acid perrhenate solutions, and oxidation of  $Cr^{III}$  to  $CrO_4''$ . Application of anodic films of Al. Deposition of Be on Cu etc. Reduction of  $C_5H_5N$ . 1261, Determining SeCN'. 1262, Determining Mg. 1264, Const.-temp. control circuit. H<sub>2</sub> ultra-violet lamp. X-Ray tubes. Electrode for spark spectroscopy. 1265, Thermocouple potentiometer. Simple high resistance. High-vac. transmitting valves. Measuring electrolytic conductivities.

#### PATENTS.

[Explosion-preventing] device for electric induction furnaces. E. F. NORTHRUP, ASST. to AJAX ELECTROTHERMIC CORP. (U.S.P. 1,898,207, 21.2.33. Appl., 22.8.31).—The  $H_2O$  for cooling, in the currentcarrying coil of the furnace, passes to the coil through the inlet, with reversed function, of an injector (I), the proper outlet (O) of which is normally closed. On any untoward occurrence, e.g., an arc between turns of the coil, causing the current to rise, O is opened by a magnetic trip and I reverts to its proper function, draining the coil instantly. B. M. V.

Apparatus for electrolysis of a fused bath. R. M. HUNTER, Assr. to Dow CHEM. Co. (U.S.P. 1,897,308, 14.2.33. Appl., 31.5.29).—In the electrolytic production of a metal, e.g., Mg, which floats on the bath, guiding and insulating means are described to keep the metal rising from the cathode pot away from the depending anodes. B. M. V.

Making [filaments for] electron-discharge devices. F. L. HUNTER, JUN., ASST. to DE FOREST RADIO TELEPHONE & TELEGRAPH Co. (U.S.P. 1,897,480, 14.2.33. Appl., 5.1.24).—An oxidised coating is coated with Mg condensed from vapour while in a preliminary evacuated vessel; after transfer of the filament to its final bulb and evacuation it is flashed to reduce the electronemissive metal in the undercoating. B. M. V.

Radio device. [Electron-emitting coating.] N. R. COLLINS, ASST. to FEDERAL TELEGRAPH CO. (U.S.P. 1,899,136, 28.2.33. Appl., 5.7.29).—The heated bare wire is rubbed with a block (formed by fusion) comprising  $BaI_2$ ,  $SrI_2$ , and a small proportion of  $Ba(OH)_2$  and subsequently raising the temp. of the cathode. B. M. V.

Coating of thermionic cathodes. L. McCulloch, Assr. to WESTINGHOUSE ELECTRIC & MANUFG. Co. (U.S.P. 1,899,765, 28.2.33. Appl., 22.8.28).—Base metal (e.g.) is coated with an aq. solution of alkaline-earth trinitride, heated in CO<sub>2</sub> to decomp., and then further heated to convert carbides into oxides. B. M. V.

Coating of radiant bodies. E. HARSÁNYI (U.S.P. 1,897,902, 14.2.33. Appl., 14.3.28. Ger., 14.3.27).— A metallic wire is provided with a coating of oxide of another metal by electrophoresis at such a low voltage (or in presence of a depolariser) that no gas is evolved, the desired oxide being in colloidal suspension in the electrolyte. Previous to electrophoresis the base wire may be etched electrolytically, and afterwards the base and coating may be re-drawn. B. M. V.

Forming an electrode for electron-discharge tubes. R. G. GUTHRIE and O. J. WOZASEK (O. J. WILBOR), ASSTS. to PEOPLES GAS BY-PRODUCTS CORP. (U.S.P. 1,897,933, 14.2.33. Appl., 2.8.28).—A cold electrode is formed of a metallic base (e.g., Ni) coated with graphite by heating in town's gas at  $>1010^{\circ}$  in presence, in the early stages, of a trace of H<sub>2</sub>O to produce a slight oxidation. B. M. V.

Production of electrically conducting coatings [of copper on non-conducting surfaces]. N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 401,022, 23.5.32. Holl., 27.10.31).—Finely-divided Cu or other metal is freed from oxide by washing with very dil.  $H_2SO_4$ , EtOH, and  $Et_2O$  and mixed with an org. binding agent; the mixture is applied to the surface to be metallised by spraying or painting. The Cu may be coated with Ag or other noble metal by simple replacement before mixing with the binder. A. R. P.

Electrode alloy. E. F. LOWRY, ASST. to WESTING HOUSE ELECTRIC & MANUFG. Co. (U.S.P. 1,899,623, 28.2.33. Appl., 30.7.30).—A non-sputtering electrode for glow-discharge tubes is composed (at least on the surface) of Ni–Co or Ni–Co–Fe, and a metal, *e.g.*, Ti, that forms an amphoteric hydroxide. B. M. V.

Welding electrode. L. B. THOMPSON and V. J. CHAPMAN, ASSTS. to GEN. ELECTRIC CO. (U.S.P. 1,898,068, 21.2.33. Appl., 28.1.32).—The electrode comprises Fe with C 0 · 17, Mn 0 · 54, S 0 · 013, P 0 · 025%, Si trace, and is coated with cellulosic material impregnated with low-C Fe-Mn 10, talc 10, felspar 30, and liquid Na<sub>2</sub>SiO<sub>3</sub> 24 pts. B. M. V.

Manufacture of electrodes. O. W. STOREY and C. A. SILVER, ASSTS. to C. F. BURGESS LABS., INC. (U.S.P. 1,899,064, 28.2.33. Appl., 3.7.28. Ger., 1.7.26).— An anode suitable for the prep. of graphitic oxide (and other processes in which the anode is disintegrated) is composed of graphite (< 10-mesh) and fused hardened rosin, the whole being compressed to remove excess rosin and obtain the desired conductivity. (Cf. B.P. 255,068; B., 1927, 17.) B. M. V.

Apparatus for purification [by electrodialysis]. H. J. SCHNEIDERWIRTH (U.S.P. 1,897,217, 14.2.33. Appl., 22.10.29).—The membrane (M) is in the form of a deep trough divided into a no. of compartments, and the liquid to be purified is caused to flow longitudinally under the influence of a corresponding no. of pairs of electrodes (E) outside the walls of M. Slots are cut in Mbetween the E so that purification takes place by electrolysis also, the impurities being retained in the spaces surrounding the E by means of porous filling such as animal refuse, hair, or other positively-charged material on the one side and vegetable matter, cotton, or similar negative material on the other. B. M. V.

Flameproof article [electrical conductor]. H. BOVING, ASST. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,897,629, 14.2.33. Appl., 9.10.24).—A composition of Se, Na<sub>2</sub>SiO<sub>3</sub>, and, if desired, S is used for flameproofing flexible cords which are preferably afterwards provided with a closely woven covering of cotton. B. M. V.

Electrothermic treatment of high-resistance [woody] material. I. H. DERBY, Assr. to P. C. REILLY (U.S.P. 1,899,233, 28.2.33. Appl., 8.5.29).—Wood or similar material is dried and/or softened for bending by the heat produced by electric currents between electrodes on opposite faces of the material, either with or across the grain. A p.d. of 15,000—20,000 volts is necessary.

B. M. V.

Impregnation of electrical condensers. M. KEVELSON (U.S.P. 1,899,155, 28.2.33. Appl., 24.5.29). —A paper dielectric is impregnated with mineral oil 40—30 and castor oil 60—70%. Methods of overcoming the difficulty of blending are described. B. M. V.

Galvanic cell. G. W. HEISE, Assr. to NAT. CARBON Co., INC. (U.S.P. 1,899,616, 28.2.33. Appl. 28.10.29).— In a cell composed of Zn,  $MnO_2$ , and C the electrolyte immobilised by, *e.g.*, flour and starch paste in the space between the C bobbin and the Zn vessel contains more  $NH_4Cl$  than the  $H_2O$  can dissolve. B. M. V.

[Open-topped] electric accumulators. CHLORIDE ELECTRICAL STORAGE Co., LTD., and H. DEAN (B.P. 402,210, 3.9.32).

Treating petroleum distillates.—See II. Calcining gypsum. PbCO<sub>3</sub> product.—See VII. Melting glass.  $B_4C$ .—See VIII. Electroplating apparatus. Electro-cleaning steel etc. Welding Al.—See X.

## XII.—FATS; OILS; WAXES.

Accelerating effect of metals on development of peroxides in oils and fats. A. E. KING, H. L. ROSCHEN, and W. H. IRWIN (Oil & Soap, 1933, 10, 204— 207).—The development of peroxides, as an index of rancidity, was followed by the authors' method (B., 1933, 876) in presence of metal stearates and strips of free metals. In all cases the process was accelerated. Stearates gave the following sequence : Cu (greatest acceleration), Mn<sup>II</sup>, Fe<sup>III</sup>, V, Cr, Ni, Zn, Al, Pb, Sn<sup>IV</sup> (least); and free metals : Cu (greatest), Pb, Zn, Sn, Fe, Al (least). The free metals, excepting Pb, did not dissolve appreciably under the conditions of the test. G. H. C.

Spoilage [rancidification] of fats and oils. R. NEU (Allgem. Oel- u. Fett-Ztg., 1933, 30, 533-537).—A review, with especial reference to the catalytic action of Fe, traces of which have been identified in butter fats. E. L.

Preparation of liquid shampoos. R. KRINGS (Allgem. Oel- u. Fett-Ztg., 1933, 30, 538-543).—The manufacture and properties of suitable soaps are described. E. L.

Uses of "liquid resin."—See XIII. Soya-bean lecithin products.—See XIX. Fatty oils from coriander seed.—See XX.

See also A., Dec., 1276, Determining the OH no. 1338, Fish oils. 1338–40, Vitamins (various). 1339, Halibut-liver oil.

## PATENTS.

Stabilisation of oils and fats. SWIFT & Co., W. D. RICHARDSON, D. P. GRETTIE, and R. C. NEWTON (B.P. 401,018, 11.5.32. U.S., 12.5.31).—Edible oils and fats, particularly those used in conjunction with  $H_2O$  (e.g., in baking), are stabilised against rancidity by an odourless, tasteless, non-toxic, oil-sol. and  $H_2O$ -insol. polyhydric phenol or derivative, aminophenol, or ketoneor aldehyde-phenol condensation product. "Stearolated pyrogallol" [C<sub>6</sub>H<sub>2</sub>(OH)<sub>3</sub>·CO·C<sub>17</sub>H<sub>35</sub>], 1:3:5-OH·C<sub>6</sub>H<sub>3</sub>(OMe)<sub>2</sub>, 1:4- and 1:5-C<sub>10</sub>H<sub>6</sub>(OH)<sub>2</sub> are specifically claimed. H. A. P.

Stabilisation of fats and/or fatty oils. Swift & Co., Assees. of R. C. NEWTON and D. P. GRETTIE (B.P. 399,639, 16.8.32. U.S., 11.4.32).—See U.S.P. 1,903,126; B., 1933, 1066.

Products from unsaturated oils. Substituted esters and thioesters.—See III.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Effect of china-wood and fish oils in outside paints. TORONTO PAINT & VARNISH PRODUCTION CLUB (Amer. Paint & Varnish Manufrs.' Assoc., Nov., 1933, Circ. No. 445, 477-480; cf. B., 1933, 976).-Five white pigment combinations were ground into linseed oil (I) which had been blended with varying proportions of tung oil (II), pilchard oil (III), or both. None of these paints showed increased  $\gamma$  after 14 months' storage, and skinning in the can was observed only with the (II) blends. The gloss, flow, and brushing qualities are tabulated, as also is the appearance of the films after 2 months' exposure on wood panels. Films containing (III) are elastic, possess better colour retention than (I) or its blends with (II), but display pronounced blooming with loss of gloss. (III), particularly when highly polymerised, also produced less after-yellowing than (II); in presence of both (II) and (III) the discoloration and yellowing were considerable. S. M.

Clarifying and straining of surface coatings. W. DECAMP and J. E. MCE. SANDERSON (Amer. Paint & Varnish Manufrs.' Assoc., Nov., 1933, Circ. No. 445, 438-442).—The use of filter-presses (aided with shredded asbestos fibre), centrifuges, and strainers is discussed. S. M.

Priming-coat reductions for painting new wood surfaces. F. L. BROWNE (Amer. Paint & Varnish Manufrs.' Assoc., Nov., 1933, Circ. No. 445, 454-458).-Exposure of TiO2-ZnO (A), white-Pb (B), and white-Pb-ZnO(C) paints in linseed oil for 26 months on redwood (I), red cedar (II), white pine (III), and Norway pine (IV) shows that there is no justification for varying the proportion of turpentine with the kind of wood. If the thinning has not been excessive, 2 coats of paint may be as durable as or even better than 3 coats. Flaking, in consequence of lack of adhesion to the summer wood (cf. B., 1930, 1038), increased in the order (I)-(IV). A was finally cleaner and more uniform than B or C, which varied in discoloration with the direction of exposure. Priming with an Al paint increased the durability in all cases; B, ZnO, and titanox were inferior to Al as primers. S. M.

[Paint] drier standardisation—the "aridyne." ST. LOUIS PAINT & VARNISH PRODUCTION CLUB (Amer. Paint & Varnish Manufrs.' Assoc., Nov., 1933, Circ. No. 445, 498—504).—The "aridyne" (B., 1931, 33) cannot be used as a standard for the following reasons. (1) Whereas the drying power of Pb, when introduced as the naphthenate into raw and alkali-refined linseed oil, is not affected by storing the varnish, the drying powers of Mn and Co undergo progressive degradation. This effect cannot be attributed to pptn. of metallic soap, since a cloudiness was observed only in the case of Pb. (2) The ratio of the drying times of 2 metals varies with their concn. and the weather conditions during drying (graphs given). S. M.

Effect of driers on durability. CINCINNATI-DAY-TON-INDIANOPOLIS PAINT & VARNISH PRODUCTION CLUB (Amer. Paint & Varnish Manufrs.' Assoc., Nov., 1933, Circ. No. 445, 508-520; cf. B., 1933, 975).-Tables are given to show the condition after 6 months' exposure of metal panels coated with linseed (A) and tung (B)oil-rosin-ester gum varnishes which contained varying amounts of Pb, Mn, and Co driers. Films of A had less gloss but better appearance than B, with less tendency to pit, flake, or scale. 1.5% of Pb or 0.4% of Mn did not accelerate failure of the film, but increasing proportions of Co, especially in A, induced progressive disruption. Comparison was also made of the tensile strength (I), elongation (II) and durability (III) of the varnish when new and after ageing in the dark for 6 months. Although the results (tabulated) are somewhat irregular, low proportions of driers generally increase (I) and diminish (II) and (III); with gradually increasing amounts of driers the curves pass through a max. or min. Pb and high concns. of Co and Mn tend to soften the film, probably by promoting formation of a liquid phase. Linoleate and naphthenate driers gave the same results. In the absence of driers A on ageing showed enhanced (I) and (III) with little change in (II), whereas B showed increased (I), decreased (II), and unchanged (III).

S. M.

Absorption of water by rust-preventive paints. F. J. PETERS (Farben-Ztg., 1933, 38, 1609-1610, 1633-1634, 1658-1659).—The wt. increase of typical rustpreventive paint films on immersion in H<sub>2</sub>O for varying periods was studied, attention being given to film thickness (I), no. of coats, age of film before immersion, and type of paint. (I) has negligible effect in the early stages, although the thick films ultimately absorb the more  $H_2O$ . Although blistering and rusting are greatly favoured if films are immersed when too fresh, the results on dried films show considerable variation according to type. The incorporation of linseed stand-oil was not found to diminish  $H_2O$ -absorption.

S. S. W.

Livering and its prevention by use of [acid] catalysts. CHICAGO PAINT & VARNISH PRODUCTION CLUB (Amer. Paint & Varnish Manufrs.' Assoc., Nov., 1933, Circ. No. 445, 459-468).-The consistencies of 8 tung oil-ZnO enamels made with various resins and having the same approx. degree of polymerisation and  $\eta$  but varying acid val. (A) were measured with a Ford cup at intervals during storage. Thickening (T) was proportional to the tung oil content and inversely proportional to A and the content of CaO-hardened resin. Congo-resin enamel thickened more slowly, whilst an ester-gum enamel rapidly reached a max. Lithopone induced T at proportionately slower rates. Where A is high T is related to: (a) the increase in η obtained by addition of NaOH after 2 months' storage ; (b) the reduction in  $\eta$  obtained by addition of a polar solvent, e.g., cellosolve. Incorporation of tribasic acids  $(H_3BO_3, \text{ citric})$  reduced T to some extent, but the rapid livering of "4-hr." varnishes made from modified PhOH-CH,O resins was unaffected. S. M.

Valuation of paints for acoustical treatments. F. C. ATWOOD (Amer. Paint & Varnish Manufrs.' Assoc., Nov., 1933, Circ. No. 445, 443—453).—A table is given to show the relative sound-absorption coeffs. of 12 acoustic building materials when painted or unpainted. Painting does not reduce the acoustic efficiency unless, as in the case of brick and some plasters, the surface pores of the material are so fine that they are bridged by the paint. Irregular pores tend to increase the absorption at the lower, and decrease it at the higher, frequencies. Tests with casein–H<sub>2</sub>O, shellac, and glyptal coatings on plaster covered with burlap indicate that many ordinary paints have insufficient porosity to be acoustically efficient. S. M.

Causes of and remedies for paint failure on plastic [plaster] surfaces. II. MONTREAL PAINT & VARNISH PRODUCTION CLUB (Amer. Paint & Varnish Manufrs.' Assoc., Nov., 1933, Circ. No. 445, 425-431; cf. B., 1933, 976) .- Tables are given to show the effects of H<sub>2</sub>O immersion on a series of plaster panels coated with paints containing varying amounts of Na2SO4,10H2O and made with various media. The free alkali in a plaster should be > the equiv. of 0.25% of NaOH. Blown linseed oil induces blistering (I) without improving the alkali-resistance (II); blown or S-treated tung oil shows increased (II), but (I) is also increased. S<sub>2</sub>Cl<sub>2</sub>treated linseed oil retards "spotting," but does not increase (II). Blending linseed oil with tung, pilchard, and soya-bean oils is without advantage. Of 22 white pigments and fillers examined, TiO2, TiO2-CaSO4, gypsum, MgCO3, and asbestine are most efficient on wet alkaline surfaces; the last two promote (I) in composite pigments, which should contain a high % of  $CaSO_4$ . S. M.

Effect of high temperatures on some paint materials. DETROIT PAINT & VARNISH PRODUCTION CLUB (Amer. Paint & Varnish Manufrs.' Assoc., Nov., 1933, Circ. No. 445, 505-507).—The behaviour of 35 pigments, varnishes, and resins when heated to 538° in a special furnace is described. S. M.

Comparison of various pigments and vehicles in rust-inhibitive primers. PITTSBURGH PAINT & VARN-ISH PRODUCTION CLUB (Amer. Paint & Varnish Manufrs.' Assoc., Nov., 1933, Circ. No. 445, 469—476).—Results are tabulated of  $2\frac{1}{2}$  months' exposure of steel panels coated with 10 basic, neutral, and metallic pigments ground into raw linseed oil, varnishes made with ester gum, PhOH and glyptal (A) resins, and into iodised oil. On the whole A and ZnCrO<sub>4</sub> appear to be as yet the most protective, but variations occur with the conditions of exposure and with application of a top coat of Al-bronze paint. S. M.

Effect of addition agents on floating of pigments in paints and enamels. CLEVELAND PAINT & VARNISH PRODUCTION CLUB (Amer. Paint & Varnish Manufrs.' Assoc., Nov., 1933, Circ. No. 445, 481-497).-Four series of linseed oil paints were prepared each containing 2 differently coloured pigments and sufficient of each of 5 basic org. compounds (I) to satisfy the predetermined absorptive capacity (A) of the pigments. The effect of (I) on the floating tendency (F) was determined by photomicrographing each paint after exposure to an electrostatic field for several hr. The pigment particles in a paint film of normal concn. possess electrical charges (cf. B., 1932, 195) which are neutralised by (I) according to the amount added and A; although decrease of Ftook place the effect was not proportional. Reversal of the charge by excess of (I) may aggravate F. Variation in the  $\eta$  of the paint during evaporation also affects the vortex movement of the particles and hence F. A does not vary so widely for basic as for acidic agents ; it depends on the solvent employed, but not on the d of the pigment. S. M.

Use of ferrous chloride in the manufacture of Prussian-blue and Mars-yellow. G. ZERR (Farbe u. Lack, 1933, 533—534, 544).—Prussian-blue can be made from FeCl<sub>2</sub> by treatment with hot or cold  $K_4$ Fe(CN)<sub>6</sub> solution according to the texture required. It possesses 25—30% greater staining power than that obtained from FeSO<sub>4</sub> and some varieties are deeper bronze. It is softer and can be more easily ground into printing media; the print has a darker, more violet tone. The prep. of FeCl<sub>2</sub> solution is described and the variation of its d with concn. tabulated. S. M.

Ultra[-violet] light and nitrocellulose films. A. KRAUS (Farbe u. Lack, 1933, 545—546, 556—557).— The extent of chemical change in nitrocellulose films containing 7 common plasticisers (I) was investigated by determining, after exposure to ultra-violet light, the  $COMe_2$ -insol. portion and the N content (II) of both the sol. and insol. portions. (I) were extracted from the exposed films with  $Et_2O$  and their (II) was also determined. They usually showed considerably increased nand acid val. (tabulated), in contrast to their behaviour when irradiated in the free state. Comparison of these data with the variations in the mechanical properties and degree of yellowing of the films shows that whilst the absorptive capacity of (I) for the N-decomp. products has an important influence, the variations are not parallel (cf. B., 1933, 199). Similarly, destruction of the film is not simply related to loss of solubility in  $COMe_2$  or to degree of denitration, although max. destruction and denitration occurred together. S. M.

Purification of extractive colophony. A. G. JEVDOKIMOV (J. Appl. Chem. Russ., 1933, 6, 933–938).— 50% solutions of crude colophony (I) in benzine (II) of b.p.  $\gg 100^{\circ}$  are diluted with (II) to 10%, when a ppt. containing most of the coloured impurities separates. The filtrate is extracted 3 times with furfuraldehyde, and (II) is distilled off from the upper layer, leaving a residue of high-quality (I). R. T.

"Liquid resin" and its utilisation. W. SCHMID (Zellstoff u. Papier, 1933, 13, 571-572).—The uses are described of waste sulphate-pulp liquors for the utilisation of their Na to produce soaps, the extraction of oils, and for de-inking newspaper. D. A. C.

Resin size.—See V. Analysis of leather finishes. —See XV.

See also A., Dec., 1302, Cannabis indica resin.

#### PATENTS.

Manufacture of a pigmentary base product. A. V. BLOM (U.S.P. 1,899,132, 28.2.33. Appl., 5.4.27).— An alloy of Pb 85, Sb 13, Sn 2% is oxidised at 250—280°. B. M. V.

Quick-drying [writing] ink. G. B. OTTLEY (U.S.P. 1,897,071, 14.2.33. Appl., 31.10.29).—To 3 pts. of a known ink 1 pt. of a mixture of diacetone alcohol 10, EtOAc 30, COMe<sub>2</sub> 30, and OH·[CH<sub>2</sub>]<sub>2</sub>·OEt 30 pts. is added. B. M. V.

Coating compositions. IMPERIAL CHEM. INDUS-TRIES, LTD., A. HILL, R. HILL, and E. E. WALKER (B.P. 401,309, 9.5.32).—Compounds from o-cresol (2 mols.) and ketones or aldehydes (except  $CH_2O$ ) (1 mol.) are condensed with  $CH_2O$  or other reactive  $CH_2$  compound in acid medium. The products are dissolved in drying or semi-drying oils to give lacquers etc. Suitable starting materials are  $\alpha\alpha - 4: 4'$ -dihydroxy-3: 3'-ditolyl-ethane, -propane, or -cyclohexane, and  $\beta\beta - 4: 4'$ -dihydroxy-3: 3'-ditolylpropane (from COMe<sub>2</sub>). H. A. P.

[Preparation and] use of oil-soluble artificial resinous materials. IMPERIAL CHEM. INDUSTRIES, LTD., A. HILL, R. HILL, and E. E. WALKER (B.P. 401,290, 28.4.32).—p-Alkylphenols, with the exception of p-cresol, are condensed with CH<sub>2</sub>O in acid medium. The phenol may contain a further o-alkyl or -aralkyl substituent. Paint vehicles and varnish media comprising solutions of the resins in drying oils are claimed. Suitable phenols are p-isobutyl-, p-tert.-amyl-, p-benzyl-, and 2: 4-dibenzyl-phenols. H. A. P.

Manufacture of artificial [resinous] masses. Soc. CHEM. IND. IN BASLE (B.P. 401,535, 6.2.33. Switz., 5.2.32).—Aromatic bases containing  $\leq 2$  nuclei directly or indirectly linked by  $\leq 2$  mols. of CH<sub>2</sub> or substituted CH<sub>2</sub> groups for each 2 mols. of aromatic residue, and at least as many  $\rm NH_2$  or  $\rm NH_2 + OH$  groups as the no. of nuclei, are condensed with aldehydes or substances that generate them under the conditions of reaction. Suitable bases are  $\rm NH_2 \cdot C_6 H_4 \cdot CH_2 \cdot NHPh$ ,  $\rm CH_2(C_6 H_4 \cdot NH_2)_2$ ,  $\rm CH_2 Ph \cdot NH \cdot C_6 H_4 \cdot NH_2$ , and intermediate products from these,  $\rm NH_2 Ph$ ,  $\rm NH_2 \cdot C_6 H_4 Me$ , etc., and  $\rm CH_2 O$ ,  $\rm CH_2 \cdot CH \cdot CHO$ , furfuraldehyde, etc. Thermoplastic and thermohardening resins are obtained. H. A. P.

Dental impression material. L. E. HARRISON and S. E. NOVES, Assrs. to S. E. NOVES (U.S.P. 1,897,034, 14.2.33. Appl., 10.11.30. Renewed 31.5.32).—A mixture is made, with the aid of heat, of the following (or materials of the same classes) : mineral and drying oils 40-72 pts., beeswax and paraffin 24-40, Al stearate 40-56 pts.; optional additions are rubber, guttapercha, or balata or their latex  $\gg 1$  pt., starch 8-24 pts., glycerin  $\gg 2$  pts., S, resin, finely-divided fibrous material, flour, and scent. The softening point is adjusted to  $35-55^{\circ}$ . B. M. V.

Condensation products.—See III. Colour base. Ice colours.—See IV. Cellulosic compositions. Safety paper.—See V. Laminated glass.—See VIII. Covering Fe surfaces.—See X.

# XIV.-INDIA-RUBBER; GUTTA-PERCHA.

Determination of rubber in rubber-bearing plants. D. SPENCE and M. L. CALDWELL (Ind. Eng. Chem. [Anal.], 1933, 5, 371–375).—The finely-divided sample (5 g. of known  $H_2O$  content), after being heated with boiling 1%  $H_2SO_4$  for 3 hr. and then steamed for 3 hr. at 30 lb./sq. in., is leached with  $H_2O$  at 60° for 3 hr. The leached material is at once extracted with COMe<sub>2</sub> for 12 hr., dried, and extracted with  $C_6H_6$  for < 16 hr. To the solution of rubber thus obtained there are added 5 c.c. of a 1% solution of dimethyl-*p*-phenylenediamine in  $C_6H_6$  to serve as antioxidant (suitable correction being made later) and the solution is evaporated to dryness on a  $H_2O$ -bath, being finally dried at 105°. The determination is always made in duplicate.

D. F. T.

Ageing process of rubber. I. Effect of irradiation with ultra-violet light on the surface tension of rubber solutions. B. DOGADKIN and G. PANT-SCHENKOV (Kolloid-Z., 1933, 65, 350—355).—In air the surface tension ( $\gamma$ ) or rubber solutions falls when irradiated with ultra-violet light, due to the formation of a surface-active oxidation product. The change in  $\gamma$  is parallel to that of  $\eta$ . In N<sub>2</sub> no change in  $\gamma$  occurs, but  $\eta$  alters. Depolymerisation of the rubber mols. is believed to occur. E. S. H.

Comparison of eighteen [vulcanisation] accelerators in a non-loaded [rubber] stock. T. R. DAWSON (India Rubber J., 1933, 86, 711-717).—Tests were made as to tensile strength, extensibility, permanent set, hardness (measured in 3 different ways), and ageing at 70° of mixtures of rubber (100), ZnO (3), and suitable proportions of S and org. accelerator, representing a range of degrees of vulcanisation. The results agree with the commercial practice of vulcanising for approx.  $\frac{2}{3}$  of the time necessary for an immediate optimum result. Accelerators of the aldehyde-amine class had antioxidant qualities, but methylenedianilide and "form" aniline "effloresced at 70°. No general advantage was discernible in the use of mixed accelerators. D. F. T.

See also A., Dec., 1300, Antioxidants of rubber latex.

# PATENT.

[Rubber-like] condensation products.-See III.

# XV.-LEATHER; GLUE.

Valonia and its tannin. L. TODESCHINI (Boll. Uff. Staz. Sperim. Ind. Pelli, 1933, 11, 541-547).—The composition of valonia and its extract, the best means of extraction, and the uses in tanning are described.

T. H. P.

Use of fluorides in hide-curing. D. J. LLOYD (Collegium, 1933, 698—700).—The increase in NaF content of gelatin made from hides cured with  $Na_2SiF_6$  and NaCl is negligible and its edible property is not affected thereby. D. W.

Simplified filter [bell] method [of tannin analysis]. J. VON SCHROEDER (Collegium, 1933, 631— 633).—The diam. of the upper half of the bell is 1.8 cm. and of the lower half 3.5 cm. Comparative results are given by both old and new methods. D. W.

Determination of chromium and iron in [chrometanning] liquors and leather. M. BERGMANN and F. MECKE (Collegium, 1933, 609–612).—The liquor or leather is heated (Kjeldahl) with  $\text{HClO}_4$ , cooled,  $\text{H}_2\text{O}$ added, the  $\text{Cl}_2$  boiled off, the Cr determined volumetrically with aq.  $\text{FeSO}_4,(\text{NH}_4)_2\text{SO}_4$  or iodometrically, and the Fe determined with  $\text{K}_2\text{Cr}_2\text{O}_7$  after reduction with ZnCl<sub>2</sub>. D. W.

Detection and determination of the degree of sulphiting in quebracho tanning extracts. O. GERN-GROSS and H. HERFELD (Collegium, 1933, 602 - 609).— The Auerbach (B., 1932, 852) and Stather-Lauffmann (B., 1932, 853) methods have been compared. Sulphitecellulose in the quebracho extracts should be pptd. with NH<sub>2</sub>Ph and HCl and the above methods used on the filtrate. Corrections are given for each method.

D. W.

Theory of mineral tannage. E. ELÖD and T. SCHACHOWSKOY (Collegium, 1933, 701-719).—The effect of well-defined Co complexes on gelatin solutions has been investigated by means of their absorption spectra, from which it is deduced that tanning properties are associated with the instability of the complexes. Stable complexes do not tan. Similar changes in the absorption spectra curves of Co tanning compounds were observed in  $H_2O$  as well as in gelatin, so that the changes in both are analogous. D. W.

Brittah Chemical Abstracts-B

73

obtained on leathers of low fat content. Spewing is prevented by absence of  $H_2O$  or very high storage temp. No connexion has been traced between the I val. of the oil and its spewing propensity.

VIII. Fatty acids are liberated from the soaps and oils used in fat-liquoring chrome leather (I), oxidation taking place with consequent diminution of the I val. and formation of oxy-fatty acids. It is not possible to judge what oils are selectively irreversibly combined with (I) from the nature of the extracted oil as compared with that of the original oil used. D. W.

Effect of mineral acidity on vegetable-tanned sole leather. Committee Report [of the American Leather Chemists' Association]. T. BLACKADDER (J. Amer. Leather Chem. Assoc., 1933, 28, 532—541).— Electrometric determinations of the  $p_{\rm H}$  of aq. extracts of the leather give more information on the effect of mineral acidity than do Procter-Searle determinations, and these  $p_{\rm H}$  vals. should be determined. D. W.

Determination of acid in chrome-tanned leather. C. RIESS and A. PAPAYANNIS (Collegium, 1933, 719—722). —The leather is heated for 1 hr. at 60° with 0.1N-aq. NH<sub>3</sub> (I) under reflux, the extract filtered into a measured quantity of 0.1N-HCl, CH<sub>2</sub>O added to the filtrate, and the NH<sub>4</sub> salts are determined by titration with 0.1N-NaOH. A blank test should be made. The protein constituents of leather are unaffected by (I). D. W.

Analysis of leather finishes. Committee Report [of the American Leather Chemists' Association]. A. C. ORTHMANN (J. Amer. Leather Chem. Assoc., 1933, 28, 541—544).—Coagulable albumins are pptd. by heat and filtered off, the filtrate is acidified, the ppt. dried, wax and oil are extracted therefrom with  $CCl_4$ , and shellac or other resin is extracted from the residue with EtOH.  $H_2O$ , ash, N, alkalinity, and solvent extractive may be determined on the finish. D. W.

Adhesives for paper. Artificial leather.-See V.

## XVI.—AGRICULTURE.

Soil survey of the Hundreds of Laffer and Willalooka, S. Australia. J. K. TAYLOR (Counc. Sci. Ind. Res., Australia, Bull. No. 76, 1933, 41 pp.).— Mechanical and chemical data are recorded and soil types discussed. A. G. P.

Complex colloids of soil. V. C. ANTONIANI and I. POLITI. VI. I. POLITI (Rend. R. Ist. Lombardo Sci. Lett., 1933, [ii], 66, 187–195, 253–256; Chem. Zentr., 1933, ii, 593–594).—Analytical data for Italian soils high in  $P_2O_5$  are given.  $\gg 70-80\%$  of the  $P_2O_5$  in the soils examined was associated with the humic substances. A. A. E.

Comparative determinations of the phosphoric acid in soils by Mitscherlich's pot-culture method and the rapid method of Dirks and Scheffer. W. MEIMBERG and W. SAUERLANDT (Z. Pflanz. Düng., 1933, 12, B, 489–498).—Agreement between the results of the two methods was obtained only in 55—60% of the cases examined. Dirks and Scheffer's method yields only qual. results. A. G. P.

Factors affecting the accumulation and loss of nitrogen and organic carbon in cropped soils. R. M. SALTER and T. C. GREEN (J. Amer. Soc. Agron. 1933, **25**, 622–630).—The action of crop residues in conserving N and org. C in soils was in the order : oats > wheat > maize. A. G. P.

Assimilation of fixed nitrogen by grasses and clovers. A. B. BEAUMONT, W. S. EISENMENGER, and W. J. MOORE, JUN. (J. Agric. Res., 1933, 47, 495-503). -Comparison is made of NaNO3 (I), (NH4)2SO4 (II), and urea (III) as N sources for plants. In early growth in H<sub>o</sub>O cultures the N of (II) was the most readily assimilated by the grasses. Over the whole growth season, however, (I) gave best results for grasses and (III) for clovers. The appearance of root decay in unsterilised media is attributed to NH3 accumulation in roots, whereby the C: N ratio of the tissue was narrowed sufficiently to encourage the activity of micro-organisms. The order of assimilability by grasses was (I) > (III) >(II), and by clovers (III) > (I) > (II), irrespective of the presence or absence of CaCO<sub>3</sub>. A. G. P.

Effect of lime and certain fertiliser constituents on yield and composition of herbage from pasture plots at Beltsville, Maryland. H. L. WILKINS and H. N. VINALL (J. Amer. Soc. Agron., 1933, 25, 603—611).—In general, yields were influenced more than was chemical composition. Rainfall is an important factor. Liming appreciably affected only the Ca content of the herbage. Superphosphate increased both the Ca and P contents. A. G. P.

Nitrogen and mineral contents of the pasture, hay, and aftermath of grasses grown in a mixture, pure plots, and pure drills. T. W. FAGAN and W. E. J. MILTON (Welsh J. Agric., 1933, 9, 93—109).— The N content of the grasses (I) (cocksfoot, timothy, meadow foxtail, fine-leaved fescue) when grown in drills (II) is consistently > when grown in mixture (III) or in pure plots (IV), whether cut as pasture (V), hay, or aftermath (VI). The (I) grown in (II) have a higher proportion of stem to leaf, but the stems have a N content > that of the same (I) grown in (III) or (IV). The CaO content of (V) and (VI) is lower when grown in (II) than when grown in (III) or (IV), whilst the K<sub>2</sub>O content of (I) is consistently higher when grown in (II) than as (V). The P<sub>2</sub>O<sub>5</sub> content of the (VI) of (I) grown in (IV) is always > that of the same (I) grown in (III) or in (II). NUTE. ABS. (m)

Influence of manuring on the after-ripening of malting barley. O. HEINISCH (Z. Pflanz. Düng., 1933, 12, B, 503—524).—The period of after-ripening varies with the variety, and is influenced more by climatic conditions than by manurial practice. Balanced manuring produces the earliest ripening. Deficiency of P delays this. A. G. P.

Factors affecting the yield and quality of canning maize. J. E. METZGER (J. Amer. Soc. Agron., 1933, 25, 583-587).—Optimum yields were obtained by application of fertilisers containing N:P:K = 1:4:2. Higher proportions of N or K delayed maturity and induced excessive growth of husk and multiple ear formation. With a N:P ratio of 1:2 increased gross yields were obtained, but max. amounts of cut maize were not produced however great the amount of fertiliser applied. High proportions of P hastened maturity, shortened the period during which maize retained optimum canning properties, and accelerated the formation of a toughened pericarp. A. G. P.

Factors affecting experimental error in fieldplot tests with maize. A. A. BRYAN and R. M. HUGHES (Iowa Agric. Exp. Sta. Res. Bull., 1933, No. 163, 243—280).—Statistical treatment of results of cropping trials is discussed. A. G. P.

Effect of different colloidal soil materials on efficiency of superphosphate. P. L. GILE (U.S. Dept. Agric. Tech. Bull., 1933, No. 371, 49 pp.).-The action of superphosphate (I) is examined in pot cultures of millet grown in sand to which varying proportions of soil colloids are added. In certain cases the soil addition reduced the response to (I) to an extent which increased with the proportion of soil used, and decreased with increasing amounts of (I) applied. In such soils max. efficiency of (I) occurs at  $p_{\rm H}$  4.5-5.0, with a min. at  $p_{\rm H}$  7.0 (approx.). In other instances soil additions improved the action of (I). In general, clay colloids of high SiO, : sesquioxide ratio increase, and those of low ratio decrease, the effects of (I) in comparison with pure sand cultures. Activated C and peat increase the efficiency of (I). The action of (I) is influenced by soil factors to extents which decrease in the order: degree of saturation of colloids with PO4", SiO2: sesquioxide ratio, effect of colloids on  $p_{\rm H}$  of medium, org. matter content. The efficiency of rock phosphates is primarily controlled by the  $p_{\rm H}$  of the substrate.

A. G. P.

Comparison of different nitrogenous fertilisers (Kalkammonsalpeter and ammonium sulphate) for turnips. O. ENGELS (Z. Pflanz. Düng., 1933, 12, B, 499–502).—Kalkammonsalpeter produced the greater gross yield, but the quality (% sugar) of the roots was slightly lower than when  $(NH_4)_2SO_4$  was used.

A. G. P

Determination of volatile acids in silage. W. LEPPER (Landw. Versuchs-Stat., 1933, 117, 113—123).— Wiegner's method is unsuitable for determining free AcOH (I) and  $PrCO_2H$  (II) in presence of lactic acid or of (I) and (II) combined. A modified process is described for determining the total (I) and (II).

A. G. P.

Effect of soil fertility, boll-maturation period, and early or late production of bolls on the length of cotton fibres. G. M. ARMSTRONG and C. C. BENNETT (J. Agric. Res., 1933, 47, 467—474).—Small, ill-nourished plants on poor soils (K- and N-deficient) produced lint of similar length to, but with less uniform distribution of fibre lengths than, vigorous plants on rich soils.

A. G. P.

Ammonium hydroxide versus calcium nitrate for cotton seedlings. J. W. TIDMORE (J. Amer. Soc. Agron., 1933, 25, 619—622).—The absence of  $NH_3$ injury to cotton grown in culture media to which relatively large proportions of aq.  $NH_3$  were added (cf. Tiedjens, B., 1932, 856) is attributed to the high [PO<sub>4</sub>'''] and therefore high buffer capacity of the media used. With culture solutions approximating in composition to those of natural soil solutions aq.  $NH_3$  produced very poor growth in comparison with that obtained with  $Ca(NO_3)_2$ . A. G. P.

Effect of certain crops on succeeding crops. T. E. ODLAND and J. B. SMITH (J. Amer. Soc. Agron., 1933, 25, 612—618).—The growth of a no. of crops is related to the changes in soil  $p_{\rm H}$  induced by the preceding crop and by the amounts of basic materials removed thereby. A. G. P.

Preparation of Derris extract sprays. H. H. RICHARDSON (J. ECON. Entom., 1933, 26, 995).—On dilution to spray concn. (1 to > 250) of COMe<sub>2</sub> extracts of Derris (2·3—5 g. of rotenone and 16—18% of total extractives per 100 c.c.) much solid matter separates, clogging the nozzles of the machines. A milky dispersion is obtained by addition of an equal vol. of COMe<sub>2</sub> to the extract before dilution with H<sub>2</sub>O or solutions of spreading agents. The toxicity of the spray residue is much more persistent when sulphonated castor oil (I) is used as a wetting agent than when K-coconut oil soap is used. (I) effectively wets foliage of gladiolus, rose, and onion at dilutions of 1 in 400. A. G. P.

Effect of accessory substances on the adherence of lime-sulphur spray to the integuments of pineleaf scale, *Chionaspis pinifoliæ* (Fitch). W. W. YATES (J. Econ. Entom., 1933, 26, 989–994).—The amount of S remaining on insects sprayed with CaO-S was decreased by additions of 0.5% of casein, slightly improved by 0.1%, and reached a max. with 0.05%. Blood-albumin produced max. covering at a concn. of 0.1% and produced inferior results in either greater or smaller proportions. A. G. P.

Mode of action of copper [spray] mixtures at the time of use. J. BRANAS and J. DULAC (Compt. rend., 1933, 197, 938—941).—The min. concn. of dissolved Cu (0.005% as  $CuSO_4,5H_2O)$  in acid, neutral, or alkaline Burgundy and Bordeaux mixtures is adequate, at the time of application, to ensure parasiticidal action. A. C.

Injury to apple by petroleum oil sprays. P. A. Young and H. E. MORRIS (J. Agric. Res., 1933, 47, 505– 522).—Various forms of leaf injury are described. Injury by oil usually increased with the sulphonatable fraction of the oil, but the various sulphonatable substances present in different oils affected the leaves to varying extents. Injury was increased by drought conditions. Emulsions made with "Cresoap" were more easily diluted, more uniformly applied, and less injurious than those prepared with Ca caseinate. For Hibernal apples oils should contain  $\geq 15\%$  (5–10%), and for dormant use  $\geq 45\%$  (30–40%), of sulphonatable matter. The speed at which oil kills cells or causes plesionecrotic symptoms on leaves is a valuable index of its insecticidal action. A. G. P.

Control of gladiolus scab. L. E. MILES (Phytopath., 1933, 23, 802-813).—The efficiency of materials examined was in the order "Calogreen" (I) (a Hg<sub>2</sub>Cl<sub>2</sub> prep.) > HgCl<sub>2</sub> (1 in 1000) > "Semesan" > CH<sub>2</sub>O and AcOH, the last-named being very ineffective. "Calochlor" (containing HgCl<sub>2</sub> and Hg<sub>2</sub>Cl<sub>2</sub>) reduced scab to a moderate extent, but delayed germination and retarded growth. No varietal differences in the response of corms to disinfectants were observed. Soil treatment

with (I) effectively reduced infection by soil-borne organisms. Bordeaux mixture was superior to Cu-CaO dust in preventing foliage infection. A. G. P.

Waste products of agriculture : their use as humus. A. HOWARD (J. Soc. Arts, 1933, 82, 84—121). —A lecture.

Disposal of sludge.-See XXIII.

See also A., Dec., 1252, Soils containing  $CO(NH_2)_2$ . 1261, Determining  $PO_4^{\prime\prime\prime}$ . 1262, Determining Mg. 1269, Saskatchewan soil profiles. Red Mediterranean soils of France. 1333, Bacterial oxidation of S in soil.

## PATENTS.

**Production of [arsenical] poison.** W. D. KIL-BOURN, Assr. to WHITE IRON ORES & PRODUCTS CO. (U.S.P. 1,898,189, 21.2.33. Appl., 25.5.28).—Minerals containing As, S, and/or Fe are mixed with hard wood containing volatile matter (e.g., sage plants) and burned upon a bed of coke. The fume that is collected is substantially insol. in  $H_2O$  and is claimed to be a compound of As and S rather than a mixture of  $As_0O_0$  and S.

B. M. V.

Hydrolysis of cellulose [farm waste etc.].— See V.

# XVII.—SUGARS; STARCHES; GUMS.

Determination of sulphur dioxide in molasses. B. DREWS (Z. Spiritusind., 1933, 61, 221-222).-A mixture of 50 g. of the molasses with 500 c.c. of H<sub>2</sub>O and 15 c.c. of 25% H<sub>3</sub>PO<sub>4</sub> is distilled to half-vol. in a current of  $CO_2$ , the distillate being collected in a receiver (R) containing excess of 0.1N-I (10-50 c.c.), which is connected with a Peligot tube (T) containing a further 5 c.c. After the distillation the contents of R and T are acidified with HCl, heated until excess of I has evaporated, and pptd. with  $BaCl_2$ , the  $BaSO_4$  being weighed. For routine analysis the contents of R and T are titrated with 0.1N-Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> after acidification with 10 c.c. of 2N-HCl, and the SO2 is calc. from the amount of I reduced by the distillate ; in this method no current of CO2 should be employed, and the vol. of I used must be measured accurately. The results by the two methods may differ by several %. J. H. L.

Purification and acidification of polysaccharide solutions by electricity. V. R. HARDY (Ind. Eng. Chem., 1933, 25, 1395—1398).—Apparatus is described for purification of fructose syrup, prepared by hydrolysis of inulin from the Jerusalem artichoke, by electrodialysis. K and Ca salts are thereby removed with reduction of  $p_{\rm H}$ , and colloid matter is pptd. The interrelationship of c.d., rate of flow of solutions through the cells, inorg. matter removed, and final  $p_{\rm H}$  are determined. H. A. P.

Technical reactions [in sugar degradations].— See II. Sugar beet.—See XVI. Mannitol in musts.—See XVIII. Apple juice concentrates.— See XIX.

See also A., Dec., 1240 and 1246, Molasses formation. 1278, Sugar determination. 1279, Solutions of invertase for determination of sucrose. Potato starch. 1330, Starch-digesting and sugar-forming enzymes. Liquefaction and saccharification of starch.

## PATENTS.

Hydrolysis of cellulose.—See V. Chocolate syrup. —See XIX.

# XVIII.—FERMENTATION INDUSTRIES.

Some factors affecting the solubility of nitrogenous substances in [malt] mashing. II. Effect of various salts in the mashing liquor. H. E. KELLY and T. S. BREMNER (J. Inst. Brew., 1933, 39, 622-639; cf. B., 1932, 957).—The variations of permanently sol. (I), total sol. (II), coagulable (III), and NH2-N (IV) with  $p_{\rm H}$ , temp. of mashing, and concn. of salt are examined. (III) decreases with rise of temp. and is greatest in the NaCl sets. The salts are classified into 2 groups according to their action on (I) and (II): (A), containing CaCl<sub>2</sub>, CaSO<sub>4</sub>, NaCl, and K<sub>2</sub>SO<sub>4</sub>, restricts the optimum  $p_{\rm H}$  for (I) and (II); (B), containing Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, FeSO<sub>4</sub>,  $CaSO_4$ , and  $MgSO_4$ , produces a well-defined dip at  $p_H$ 4.6. The optimum  $p_{\rm H}$  for (IV) is 4.6—5.2 at a mashing temp. of 65°, using a Scotch malt. The temp. optimum for (III), (II), and (I) are 40-50°, 50°, and 50-60°, respectively. R. H. H.

Formation of mannitol in musts dialysed at low temperature in presence of chloroform. G. BARBERA (Annali Chim. Appl., 1933, 23, 470–473).—When conc. grape musts containing CHCl<sub>3</sub> are dialysed through oxbladder at 8–10°, mannitol is formed in appreciable quantity. T. H. P.

Browning of white wine. HERZBERG (Schweiz. Wein-Ztg., 1933, 41, 373—374; Chem. Zentr., 1933, ii, 628).—The coloration, which is probably due to oxidases, is prevented by heavy sulphuring or by Möslinger's method. Blending is subsequently necessary.

A. A. E.

Determination of citric acid in wine. T. VON FELLENBERG (Mitt. Lebensm. Hyg., 1933, 24, 142-156; Chem. Zentr., 1933, ii, 1102-1103).-20 c.c. are boiled with 3 c.c. of 20% aq. Pb(OAc)2, cooled, and centrifuged until pptn. is complete. The ppt. is washed, shaken with 1 c.c. of  $H_2SO_4$  (1 + 4), boiled, and 10 c.c. (red wine) or 3-5 c.c. (white wine) of Br-H<sub>2</sub>O in excess are added, H<sub>2</sub>O to 10 c.c. being added in the latter case. After centrifuging, the liquid is separated, and that remaining with the ppt. determined. The liquid is heated with 3 c.c. of Br-H2O to 40°, and 1 drop of 5% aq. KMnO4 is added; after decolorisation this addition is repeated, Br being maintained in excess above the liquid. Finally the cooled liquid is decolorised with NaHSO3. A white ppt. indicates citric acid (I). The ppt. is extracted with CHCl<sub>3</sub>, the extract dried with Na<sub>2</sub>SO<sub>4</sub>, evaporated, and the residue weighed. For a yield of 90 o of pentabromoacetone the factor is 0.52. Wine usually contains 0.38-0.94 g. of (I) per litre. A.A.E

Corrosion-resistance of metallic apparatus in breweries. III. P. SCHAFMEISTER and W. TOFAUTE (Schweiz. Brauerei-Runds., 1933, 44, 101-103; Chem. Zentr., 1933, ii, 947).

Bromatological chemistry [analysis] of highly alcoholic beverages. M. TAVEIRA, J. E. A. FILHO, and J. B. DE MELLO (Rev. Soc. Brasil. Quím., 1933, 4, 106—144; cf. B., 1933, 166, 842).—Methods in use in the Bromatological Laboratory of the National Dept. of Public Health (Brazil), and proposed for "standard" use, are detailed; identification of colouring matters is included. E. L.

Fractionation and analysis of spirits. G. VEGEZZI and F. HALLER (Mitt. Lebensm. Hyg., 1933, 24, 21–35; Chem. Zentr., 1933, i, 4060).—Analytical results are recorded. More exact differentiation is possible if the sample is first treated with increasing quantities of C.

## Fodder from wood.-See XIX.

See also A., Dec., 1330, Amylosynthease. Starchdigesting and sugar-forming enzymes. 1333, Hydrogenase and hydrogenlyase.

#### PATENTS.

Ergosterol.—See III. Chocolate syrup. Supplemental food prep.—See XIX.

## XIX.—FOODS.

Adulteration of wheat flour. G. ISSOGLIO (L'Ind. Chimica, 1933, 8, 1359—1368).—Adulteration, e.g., by adding bran, may not cause the ash and cellulose contents to exceed the legal max., but it reduces greatly the plasticity as measured by the ergometer, and the flour is therefore unsuitable for baking. It is urged that a legal min. plasticity should be specified.

H. F. G.

A. A. E.

Flour-water farinograph curve. H. L. B. DE JONG (Z. ges. Getreide- u. Mühlenw., 1933, 20, 118—120; Chem. Zentr., 1933, ii, 798—799).—The % H<sub>2</sub>O bound is for various flours of the same fineness smallest at the  $p_{\rm H}$ vals, at which the gluten is max. The effect of addition of small quantities of electrolytes is described. The differences between the flours are best examined by means of the farinograph. A. A. E.

Nitric acid reaction of flour. G. KLEMT (Z. ges. Getreide- u. Mühlenw., 1933, 20, 92—95, 116—118; Chem. Zentr., 1933, ii, 800).—The Mohs and Kühl coagulation reaction with HNO<sub>3</sub> is a sp. protein reaction of the albumin leucosin. A. A. E.

Examination of soya-bean lecithin products. M. AUERBACH (Fettchem. Umschau, 1933, 40, 218—219). —The content of soya-bean oil in products such as cacao fat containing soya-bean lecithin may be determined approx. by saponifying a COMe<sub>2</sub> extract of the material, separating the "liquid" acids (Pb-salt separation), and submitting them to the hexabromide test.

E. L.

Chemical analysis of casein. R. S. SNYDER and H. C. HANSEN (Ind. Eng. Chem. [Anal.], 1933, 5, 409— 412).—Existing methods are modified to give more accurate and consistent results more rapidly.

R. S. C.

Bromatological chemistry [analysis] of coffee. M. TAVEIRA and F. F. CÔRTES (Rev. Soc. Brasil. Quím., 1933, 4, 145—160).—Methods in use in the Bromatological Laboratory of the National Dept. of Public Health (Brazil) and proposed for "standard" use are detailed. Analyses of 50 samples are tabulated. E. L. Determination of caffeine in coffee. F. F. CÔRTES Rev. Soc. Brasil. Quím., 1933, 4, 105; cf. preceding abstract).—The powdered coffee (1 g.) is treated on the  $H_2O$ -bath with 2 c.c. of conc.  $H_2SO_4$  (15 min.) followed by 50 c.c. of boiling  $H_2O$  (15 min.); the hot mixture is then filtered, boiling  $H_2O$  being used for washing, and the cooled filtrate made alkaline with conc. NaOH and extracted with CHCl<sub>3</sub>. The caffeine, quantitatively recovered by evaporation of the extract, is of a high degree of purity. E. L.

Losses of vitamin-A in drying fresh raw carrots and sweet potatoes and canned spinach. G. S. FRAPS and R. TREICHLER (J. Agric. Res., 1933, 47, 539-541).—Carrots lost approx. 80%, spinach 65%, and sweet potatoes 29% of their vitamin-A activity on drying. A. G. P.

Effects of time of planting and of fertiliser mixtures on the curly-top resistant sugar-beet variety, U.S. No. 1, in Idaho. A. W. SKUDERNA, C. E. CORMANY, and L. A. HURST (U.S. Dept. Agric. Circ., 1933, No. 273, 16 pp.).—Artificial fertilisers containing high proportions of P produced higher yields of beet and of sugar per acre than those in which K or N predominated. Fertilisers containing > 8% N markedly reduced the % stand of plants. A. G. P.

True and apparent digestibility of oat hulls and lucerne meal by swine, with special reference to the ability of swine to digest cellulose and crude fibre. H. H. MITCHELL and T. S. HAMILTON (J. Agric. Res., 1933, 47, 425-435).—The total digestible nutrient content of oat hulls (I) was  $26 \cdot 8\%$  and of lucerne meal (II)  $31 \cdot 3\%$ . Metabolisable energy vals. were  $1 \cdot 166$ and  $1 \cdot 624$  kg.-cal. per kg., respectively. At low levels of protein feeding the absorbed protein from (I) and (II) was completely metabolised. The digestibility of the crude fibre of (I) and (II) was 2%, and of paper pulp and cellophane  $0-9 \cdot 2\%$  (average  $3 \cdot 5\%$ ). A. G. P.

Feeding value for milk production of pasture grasses when grazed, when fed green, and when fed as hay or silage. R. R. GRAVES, J. R. DAWSON, D. V. KOPLAND, and T. W. MOSELEY (U.S. Dept. Agric. Tech. Bull., 1933, No. 381, 47 pp.).—Comparative feeding-trials are recorded with immature grass cut at varying intervals. A. G. P.

Wet beet pulp compared with maize silage in the dairy ration. G. Q. BATEMAN and G. B. CAINE (Utah Agric. Exp. Sta. Bull., 1932, No. 239, 40 pp.).— No significant differences were observed in the effects of the two feeding-stuffs on milk or butterf at production. A. G. P.

Fodder from wood. C. G. SCHWALBE (Angew. Chem., 1933, 46, 707-710).—A historical survey. The chopped green wood without separation of the bark is treated with alkali, and then with lactic acid, or with a lactic ferment and sugar, in an edge mill, the product being 100% digestible by animals. The product obtained with other swelling agents is attacked by moulds, whilst that from HCl treatment tends to contain furfuraldehyde. A. A. L.

Apple juice concentrates. O. GROVE (Fruit Products J., 1933, 13, 74-75, 89).—For use in the cider

industry these may be prepared by evaporation at  $>50^{\circ}/\text{vac.}$ , by a freezing method, or by a mixture of both methods. These methods are described and the resultant products compared. A special form of concentrate can be used as treacle. In this, pectin is removed, acidity partly neutralised if necessary, and sugar added before evaporation. All the above products are prepared from sub-standard apples. E. B. H.

Colour in tomato products. M. B. MATLACK and C. E. SANDO (Fruit Products J., 1933, 13, 81—82, 90).— Lycopene (I) is the pigment in both American and Italian tomatoes. Possible reasons for change in colour of tomato products during processing are discussed. Prevention of colour change and of loss of carotene, closely associated with (I), is of primary importance.

E. B. H.

Canned shrimp and the presence of vitamins-A, -B, and -D. M. C. MOORE and H. W. MOSELEY (Science, 1933, 78, 368—369).—Qual. tests with rats on wet-pack and dry-pack canned shrimp show the presence of vitamin-A and -D, but not -B, in appreciable quantity in shrimp fat. L. S. T.

Presence of tin in food products. S. BACK (Food Manufacture, 1933, 8, 381—384).—A crit. review of published work on the origin and determination of Sn in foodstuffs, and the pharmacological properties of Sn compounds. E. S. H.

See also A., Dec., 1262, Detecting Cs, Rb, and Tl [in mineral waters]. 1325, Nutritive val. of Indian vegetable foods. 1333, Cheese ripening. 1339, EtOH-extractable substances of rice.

#### PATENTS.

Manufacture of [cereal] foodstuffs. J. PROCTER & SON, LTD., T. W. HIGGINS, and H. HIGGINS (B.P. 401,555, 4.3.33).—The cereals, husked or unhusked according to the type of grain, are softened by steeping in  $H_2O$  and then cooked in steam-kettles. The material is then pressed into cakes or pellets, flattened into flakes by squeeze rolls, and finally toasted. Different grains may be blended together after the cooking process.

Preparation of supplemental food product. T. G. DYER, Assr. to STANDARD PRODUCTS Co. (U.S.P. 1,898,350, 21.2.33. Appl., 30.9.29).—Milled cereals rich in gluten are mixed with diastase and yeast starters, moistened, stored in bulk at 21—55° until lactic acid has formed, and the fermentation is then stopped by drying in a thin layer at the same temp. range,

B. M. V.

Manufacture of chocolate syrup. W. W. TRIGGS. From WALLERSTEIN Co., INC. (B.P. 401,333, 4.3.32).— Cocoa is mixed with 2—4 times its wt. of  $H_2O$  and heated until the mixture thickens. It is then cooled and a diastatic enzyme prep., with or without proteolytic enzymes, is added, and set aside until hydrolysis of the cocoa starch is complete. The necessary amount of sugar and  $H_2O$  is then added to produce a chocolate syrup, which has good keeping qualities and will not thicken on keeping. E. B. H.

Treating liquids [milk].—See I. Stabilising oils and fats.—See XII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Sterilisation of hexamethylenetetramine solutions. A. PFISTER (Pharm. Ztg., 1933, 78, 1228– 1229).—25% solutions of  $(CH_2)_6N_4$  are sterile 48 hr. after prep., but 10% solutions need heating at 40° for 15 min. Both solutions give only a weak test with Schiff's reagent on boiling for a short time, but this is intensified by prolonged boiling. S. C.

Determination of free acid in quinine salts. J. FRANGOULI and A. VASSILIOU (Praktika, 1932, 7, 293—297; Chem. Zentr., 1933, ii, 916).—25 c.c. of a solution of 4 g. of quinine salt in 500 c.c. of  $H_2O$  are treated with 40 c.c. of abs. EtOH and titrated with 0.1N-NaOH (Me-red). A second portion is treated with twice as much NaOH, and the excess alkalinity towards phenolphthalein, representing the free acid, is titrated.

A. A. E.

Formation of free nicotine in the residues from the steam-distillation of tobacco. C. PYRIKI (Pharm. Zentr., 1933, 74, 717—721).—The nicotine (I)-free residue from the steam-distillation of a mixture of tobacco and NaOH after keeping (e.g., for 10 days) and then re-distilling gives a positive reaction for (I), probably due to the slow decomp. of the glucoside tabacin. 25 successive distillations of cigar tobacco gave 0.139— 0.153% of (I). E. H. S.

Analysis of Derris root and determination of rotenone. D. R. KOOLHAAS (Bull. Jard. bot. Buitenzorg, 1932, [iii], 12, 563-574; Chem. Zentr., 1933, ii, 749-750).-H<sub>2</sub>O is determined in 20-40 g. of finelyground root by the xylene method. 50 g.  $( \ge 10\% H_2 O)$ are extracted for  $2 \times 24$  hr. with dry (Na) Et<sub>2</sub>O; the extract is evaporated to 25 c.c. and kept for 2 days at room temp. and for 2 days at 0°. After rotating the solution for 2-3 min. at 3500 r.p.m. it is poured into a weighed flask, the crystals are triturated with 10 c.c. of Et<sub>2</sub>O, kept for 1 day at 0°, and centrifuged. The crystals are dried at 80° in vac. The Et<sub>2</sub>O solution is kept over ice and somewhat diluted with Et<sub>2</sub>O; the rotenone (I) crystallising out is added to the main crop. The residue of the Et<sub>2</sub>O extract is determined. The crude (I) is finely powdered and centrifuged with Et<sub>2</sub>O at 0° until the m.p. is  $\lt$  120°. 0.042 g. is added for the (I) in each 10 c.c. of Et<sub>2</sub>O. The (I) content varies even for different specimens of the same variety. 23-24-months-old roots are richest. There is no relation between (I) content and Et<sub>2</sub>O extract. A. A. E.

Gravimetric determination of santonin in Flores Cinæ. II. M. M. JANOT and C. ESTÈVE (Bull. Sci. pharmacol., 1933, 40, 280–286; Chem. Zentr., 1933, ii, 584). A. A. E.

Behaviour of fluid extracts of medicinal plants towards Wood's light. A. SOLDI (Annali Chim. Appl., 1933, 23, 455-462).—210 extracts of medicinal plants of most common therapeutic use have been examined and their fluorescence colours classified according to Ostwald's system. A no. of the results are reproduced on a coloured plate. T. H. P.

Essential oil of Canarium Zeylanicum (Thw.). J. P. C. CHANDRASENA and H. LOURENSZ (J.S.C.I., 1933, 52, 362 r).—Fractionation of this oil,  $[\alpha]_{Hg}$  +52·31° (10—15% yield), affords  $\alpha$ -phellandrene (45%), and a

E. B. H.

main fraction (b.p.  $120-122^{\circ}/5$  mm.) from which a solid  $C_7H_{16}O$ , m.p.  $164^{\circ}$ , is deposited, leaving a polycyclic terpene  $C_{15}H_{24}$ . Dehydrogenation of the higher-b.p. fractions failed to give alkylnaphthalenes. J. W. B.

Essential and fatty oils from N. Caucasian coriander seeds. I. I. VANIN and A. A. TSCHERNO-JAROVA (J. Appl. Chem. Russ., 1933, 6, 922–932).— The seeds contained 0.85% of essential oils (chiefly linalool, and  $\alpha$ - and  $\beta$ -pinene), and 20% of fatty oils, consisting chiefly of the glyceride of petroselic acid, together with glycerides of *iso*oleic and stearic acids. R. T.

## Determining caffeine in coffee:-See XIX.

See also A., Dec., 1249, Autoxidation of aldehydes. 1256, Changes in acid solutions of adrenaline. 1260, Determining S [in pharmaceutical products]. 1262, Microchemistry [of drugs]. 1264, Detecting Bi. 1271, Prep. of CBr<sub>3</sub>·CH<sub>2</sub>·OH. 1288, Synthesis of sec. and tert. amines. 1293, Synthesis of arylacetic and -propionic acids. 1296, Substances with the odour of violets. 1298, Phenolic ketones and their reduction products. 1300, Isomeride of Bredt's 5-ketocamphor. 1311—3, Alkaloids (various). 1313, Hydrates of Na *p*-aminophenylarsinate. 1314, Colour reactions of  $m-C_6H_4(NO_2)_2$ in alkaline media. 1317, Prep. of pure heparin. 1338, Separation of sexual hormones. 1338—40, Vitamins (various).

#### PATENTS.

[Preparation of] hexamethylenetetramine zirconium citrate [medicinal]. A. J. GELARIE (U.S.P. 1,905,848, 25.4.33. Appl., 30.7.28).—To an aq.-EtOH solution of ZrCl<sub>4</sub> or ZrOCl<sub>2</sub> citric acid in EtOH is added, followed by hexamine. The pptd. substance,  $C_{12}H_{16}O_9Zr$ , is filtered off, washed with EtOH, and dried with Et<sub>2</sub>O. H. A. P.

Preparation of choline derivatives. O. DALMER and C. DIEHL (U.S.P. 1,894,162, 10.1.33. Appl., 18.4.31. Ger., 8.5.30).-Products of choline carbamate type are claimed as pharmaceuticals. The following are prepared either from  $\omega$ -chloroalkyl carbamates and tert. bases, or from the corresponding 'NAlk2-compounds and alkyl halides:  $\beta$ -dimethylaminoethyl carbamat methiodide, m.p. 200°, and methochloride, m.p. 208°  $\beta$ -dimethylaminoethyl carbamate  $\beta$ -diethylaminoethyl carbamate methiodide, m.p. 123—124°, β-hydroxyethiodide, m.p. 105-107°, β-carbamatoethiodide, m.p. 151°, and phenacobromide, m.p. 182°; y-dimethylaminopropyl carbamate methochloride, m.p. 207-208°; a-methyl-a-(dimethylaminomethyl)-n-propyl carbamate methiodide, m.p. 220°; β-[methyl-(β'-4-hydroxyphenylethyl)amino]ethyl carbamate methiodide m.p. 152-153° (from hordenine); β-diethylamino-α-phenyl-n-propyl carbamate methiodide, m.p. 220-221°. H. A. P.

[Preparation of] metal complex compounds. H. SCHMIDT and H. JUNG, ASSTS. to WINTHROP CHEM. Co., INC. (U.S.P. 1,893,872, 10.1.33. Appl., 4.6.31. Ger., 3.6.30).—Complex salts are prepared by interaction in aq. solution of metallic oxides or hydroxides of at. no. > 21 with the exception of Fe, Sb, alkalis, or alkaline earths (As, Cu, V, Sn, Ce, Mo, Co, La, and W) with polyhydroxycarboxylic acids  $\gg C_5$  containing at least 3 vicinal OH groups (saccharic, gluconic, and glucoheptoic acids) and sufficient alkali or org. base for neutralisation. The preferred product is K Na cuprisaccharate. H. A. P.

 $\alpha$ -p-Aminophenyl- $\beta$ -methylaminopropanol-1. NO<sub>2</sub>-derivative of *tert*.-butylcymene. Bactericides. —See III. Cinchona alkaloid products for pickling baths.—See X.

# XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

See A., Dec., 1234, Lines of slip in deformed AgCl crystals. 1255, Latent images. Highly disperse emulsions. Intermittency effect in exposures. 1256, The DIN degree. 1264, X-Ray powder photography. Heterochromatic photometry.

## XXII.—EXPLOSIVES; MATCHES.

Analogues of tetryl. I. Trinitrophenylnitroaminoethyl nitrate [pentryl]. L. V. CLARK (Ind. Eng. Chem., 1933, 25, 1385–1390).—The prep. of  $\beta$ -2:4-dinitroanilinoethanol, m.p. 92°, in 70% yield from C<sub>6</sub>H<sub>3</sub>Cl(NO<sub>2</sub>)<sub>2</sub> and NH<sub>2</sub>·C<sub>2</sub>H<sub>4</sub>·OH, and its nitration (H<sub>2</sub>SO<sub>4</sub>-HNO<sub>3</sub> at 20—50°) to  $\beta$ -2:4:6:N-tetranitroanilinoethyl nitrate, m.p. 128° (90% of theory), are described. In explosive properties it resembles but is more powerful than C<sub>6</sub>H<sub>2</sub>Me(NO<sub>2</sub>)<sub>3</sub> (I); it is more sensitive than (I) and may be used as a detonator. Its explosive properties are still more powerful in presence of oxidising agents. It is unaffected by H<sub>2</sub>O at room temp. and only slightly decomposed by keeping at 75° for 40 days. H. A. P.

See A., Dec., 1242, Sorption of vapours by nitrocotton. 1251, Thermal decomp. of solids.

#### PATENTS.

**Explosive [priming composition].** J. D. MCNUTT, Assr. to WINCHESTER REPEATING ARMS Co. (U.S.P. 1,906,394, 2.5.33. Appl., 27.9.32).—A priming composition consists of Pb dinitrophenylazide (I), Pb[ $O \cdot C_6 H_2(NO_2)_2 \cdot N_3]_2$ , with other components; e.g., a mixture of (I) 15, Ba( $NO_3$ )<sub>2</sub> 25, PbO<sub>2</sub> 12, diazodinitrophenol 17, trinitroresorcinol 8, ground glass 22, and gum arabic 1% is suitable. W. J. W.

Propellent powder. G. C. HALE (U.S.P. 1,899,147, 28.2.33. Appl., 1.3.32).—A smokeless and flashless explosive comprises nitrocellulose with  $C_6H_2Me(NO_2)_3$  5—15 and PhNO<sub>2</sub> 2—10%. B. M. V.

#### XXIII.—SANITATION; WATER PURIFICATION.

Disposal of sludge at water purification and softening works of the Mahoning Valley Sanitary District [Youngstown, Ohio]. W. H. DITTOE (J. Amer. Water Works' Assoc., 1933, 25, 1523---1533).--Approx. 10<sup>7</sup> gals. per day are treated with CaO, Na<sub>2</sub>CO<sub>3</sub>, and alum in the proportions  $4 \cdot 8 : 1 \cdot 8 : 1$  with the production of 160 tons of wet sludge (95% H<sub>2</sub>O). By lagooning, the H<sub>2</sub>O is reduced to 60---65%; it is hoped that the semi-dried sludge may be of use in agriculture. C. J.

#### PATENTS.

Sedimentation tanks. Supplying air etc. to liquids.—See I.