# BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

NOV. 11 and 18, 1932.\* or as four as to for of as or beginning of the second se

# I.-GENERAL; PLANT; MACHINERY.

Use of trisodium phosphate in boilers not provided with water-softening plant. F. KROEMER (Chem.-Ztg., 1932, 56, 722—724).—A general account is given of the use of  $Na_3PO_4$  in boiler practice for (1) removal of scale both during normal working and when the boiler is shut down; (2) softening the  $H_2O$ in the boiler; and (3) treating the feed- $H_2O$  between the softening plant and boiler. H. F. G.

Phosphate determination in boiler water. A. SULFRIAN (Chem.-Ztg., 1932, 56, 650-653).-Medinger's method, in which  $P_2O_5$  is indicated by a turbidity produced by adding the sample to a solution containing  $(NH_4)_2MoO_4$  and strychnine nitrate, gives good results up to 10 mg./litre  $P_2O_5$  content. Its disadvantage is interference by SiO<sub>2</sub>. Other methods have the same objection; the only one suitable for practical use is Feigl's micro-method. This depends on the oxidising action of phosphomolybdates etc. on benzidine, giving a blue colour. The author's modification of this method permits the identification of 1 mg.  $P_2O_5$  per litre in presence of a great excess of SiO<sub>2</sub>. The method is made quant. by dilution and colour-matching, the filter-paper used being placed upon a milk-glass plate. As the desirable limits of  $P_2O_5$  concended in boiler water are 20-50 mg./litre, sufficient accuracy is readily attained.

Determining thermal conductivity coefficients of various insulating materials. T. F. MAXIMOV and S. P. BOITZOV (Izvest. Tepl. Inst., 1932, No. 3, 256-278).-Heat-transfer coeffs. of various insulating compositions placed on pipes were determined. CH. ABS.

Application of physico-chemical principles to design of liquid-liquid contact equipment. I. General theory. T. G. HUNTER and A. W. NASH (J.S.C.I., 1932, 51, 286-297T).-Processes for bringing about contact between two liquids are classified and described. Formulæ for calculating the amount of solute remaining after a given treatment are developed for batch, concurrent, and countercurrent processes. These are used to show the advantages of countercurrent operation over other methods. The two-film theory of solute transfer between two countercurrent immiscible phases in contact is applied to liquid-liquid systems and the rate of extraction and calculation of the individual film extraction coeffs. is discussed. The use of overall extraction coeffs. is shown to be applicable to isothermal processes where the simple distribution law holds for normal solutes at small concns., and a relationship between overall and individual film coeffs. is developed. The mechanism of the process of extraction when chemical reaction takes place between the solute and a reagent in solution in the extracting liquid is described for both reversible and irreversible reactions. The dependence of extraction coeffs. on the dynamic conditions prevailing in the apparatus is stressed and formulæ relating these coeffs. with liquid velocity, kinematic viscosity, the linear dimensions of the apparatus, and the diffusion coeffs. are suggested. Methods of computation applicable to spray and plate tower systems are dealt with, and a graphical means for determining the height of towers employing the countercurrent system of operation is described. The theories which summarise most of the existing ideas of the mechanism of diffusion processes are discussed and it is pointed out that they are open to criticism in certain respects.

**Evaporation from large surfaces.** J. DOMINGO Y QUÍLEZ (Anal. Fís. Quím., 1932, **30**, 492-504).--Existing formulæ for calculating the rate of evaporation from a free surface are criticised, and an equation is derived which does not involve the determination of empirical consts. and necessitates only measurements of temp. and pressure. H. F. G.

Technical sedimentation analysis. H. GROHN (Chem. Fabr., 1932, 5, 325—327).—A detailed description is given of an apparatus for measuring rates of sedimentation which has the particular advantage that it can be used satisfactorily with coloured or foaming suspensions. Typical measurements with various pigments etc. with the new and old forms of apparatus are given. H. F. G.

Design calculations for vapour recovery and rectifying units. G. G. BROWN and M. SOUDERS, JUN. (Refiner Nat. Gas. Mfr., 1932, 11, 376).—Raoult's law is eliminated and ideal solutions are assumed. The towers designed are as efficient at 400 lb. pressure as at 40 lb. CH. ABS.

Orifice meter; expansion factor for gases. E. BUCKINGHAM (Bur. Stand. J. Res., 1932, 9, 61-79).-The discharge coeff. of an orifice meter, determined with  $H_2O$ , is applicable to the measurement of a flow of gas provided that the differential pressure is insufficient to change the *d* appreciably, otherwise an expansion factor must be used; this factor is shown to depend on the form of the meter, the ratio of down- to up-stream pressure, and the sp.-heat ratio of the gas, and an empirical equation is deduced for computing this factor in certain cases. A. R. P.

See also A., Oct., 1007,  $H_2$  from Fe and  $H_2O$ [in colloid mills]. 1012, Continuously-operating laboratory furnaces.

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

# PATENTS.

Furnace [for low-grade fuel]. W. A. GILCHRIST (U.S.P. 1,839,960, 5.1.32. Appl., 14.10.27).—A step- or other form of grate is provided to produce a steeply sloping bed of fuel, and the arch and back wall are arranged so as to reflect as much as possible of the heat produced by the combustion of volatile matter back on to the bed of fuel. B. M. V.

Method of controlling [exothermic] reactions. R. P. COURTNEY and V. E. MEHARG, ASSTS. to BAKELITE CORP. (U.S.P. 1,840,186, 5.1.32. Appl., 12.5.28).— The reaction vessel (preferably a bundle of vertical tubes) is surrounded by a molten salt bath which is agitated and maintained at the correct temp. by injection of a finely-divided cooling agent, e.g.,  $H_2O$  and steam. Its use in the prep. of  $CH_2O$  by catalytic oxidation of MeOH is indicated. B. M. V.

Carrying out catalytic reactions [e.g., hydrogenation or cracking of petroleum oils]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 379,335, 26.5. and 6.10.31).—The catalysts comprise sulphides of metals in groups V and VI of the periodic system prepared by heating thio- or thioxy-salts of the metals in H<sub>2</sub>S or in an atm. of S at  $> 300^{\circ}$ . 22 examples of the prep. and use of the catalysts are given. A. R. P.

Heating vacuum evaporators and boilers. E. SCHUSTER (B.P. 379,085, 17.8.31).—A steam coil is placed in the jacket, which is filled with glycerin or other non-volatile liquid. The apparatus is suitable for preventing scorching during low-temp. vac. evaporation with steam at normal temp., also for evaporation at high temp., the high-pressure steam being confined to a small-bore coil. B. M. V.

Drying of liquids containing solids [e.g., fruit juices]. C. W. WILSON (B.P. 379,081, 12.8.31).—An irreversible colloid is added to the liquid before evaporation. Na pectate, alginate, or other carbohydrate acid or salt thereof is suitable, and, after incorporation, a salt of a heavy metal or of Mg may be added to promote the pptn. of the gel. B. M. V.

Cooling tower. G. H. LIFUR (U.S.P. 1,839,104, 29.12.31. Appl., 1.8.27).—Wind-directing means, and adjustable baffles for the  $H_2O$  are disposed so as to encourage horizontal draughts of air. B. M. V.

Liquid for use with refrigeration systems. H. J. SCULLEN, ASST. to COPELAND PRODUCTS, INC. (U.S.P. 1,841,258, 12.1.32. Appl., 21.7.28).—Brine for use as the heat-absorption medium comprises a eutectic solution of an inorg. salt adapted to freeze in the operating range of the refrigerator, *e.g.*, a solution of  $23 \cdot 2$  pts. of BaCl<sub>2</sub> in 76  $\cdot$  8 pts. of H<sub>2</sub>O, which has f.p. —8°. L. A. C.

Manufacture of thermal insulating structure. H. B. LINDSAY (U.S.P. 1,842,875, 26.1.32. Appl., 14.12.27).—An aq. solution of  $Na_2SiO_3$  and  $Na_2B_4O_7$  is heated at 320—370° for a time sufficient to cause intumescence but not calcination. Sheets of the product are formed by running the solution into a suitable mould. L. A. C.

Grinder. L. F. CALHOUN, ASST. to W. R. HODGES (U.S.P. 1,839,532, 5.1.32. Appl., 9.10.29).—The stationary grinding element of a disintegrator comprises a drum without ends, an aperture being left at the top and perforations through the lower part for entry and exit, respectively, of material. The ends of the drum are formed of discs rotating with the shaft, which also carries flexible beaters, *e.g.*, chains. B. M. V.

Kneading, grinding, and mixing machines. L. and J. EIRICH (B.P. 379,265, 10.5.32. Ger., 20.6.31). —A rotating pan, having grinding and mixing devices distributed over the bottom, is provided with a central outlet guarded by a stationary cylinder having an adjustable port, and with a fixed guide extending from the circumference of the pan to the port in the outlet device. B. M. V.

Mixing of finely-divided solid materials. E. ANDERSON, ASST. to INTERNAT. PRECIPITATION CO. (U.S.P. 1,839,456, 5.1.32. Appl., 23.5.27).—The materials are charged into a tall container and are agitated by compressed gas injected at first near the surface and thereafter at lower points until the whole mass is sufficiently agitated. Discharge is effected through a bottom outlet while the mass is still fluid. B. M. V.

Consistency regulator. M. O. SCHUR, Assr. to BROWN Co. (U.S.P. 1,838,710, 29.12.31. Appl., 1.10.28).—A paddle or other detecting device is placed in the stream of pulp in its delivery condition, the variations in torque necessary to drive the paddle at const. speed being caused to adjust the amount of diluting liquid added to the pulp at some point before the detecting device. B. M. V.

Separation of materials [by flotation]. F. TSCHUDY (U.S.P. 1,840,267, 5.1.32. Appl., 9.7.28).—The process is suitable for coarsely granular material, e.g., coal. Three streams of fluids are injected into the pulp, viz., (1) circulating  $H_2O$  to control the pulp thickness and if desired to carry in the flotation oil, this stream tending to move the gangue matter to the bottom outlet; (2) a stabilising agent to hold the floated granules at the surface of the bath, which in the case of coal would be a pulp of finely-divided coal; and (3) air or gas previously ionised to a controllable extent by means of a corona or other type of electrical discharge and to a polarity opposite to that of the material to be floated. B. M. V.

Lixiviation of raw materials in stages. E. BÖHM (B.P. 378,880, 8.5.31. Ger., 18.12.30).—The material passes in succession through a no. of superposed annular trays in a closed vessel, rotation of the trays causing the material to be scraped off at one point and drop into the next tray. A preferred form of tray comprises a fixed outer wall, and an inner wall and perforated bottom rotating together. The liquid percolates downwards through the material in any tray, but is lifted upwards from tray to tray so that a countercurrent effect is produced. B. M. V.

Screw-press for dewatering and washing diluted substances. R. T. LANG, ASST. to AMER. VOITH CON-TACT CO., INC. (U.S.P. 1,838,996, 29.12.31. Appl., 27.2.29).—The material emerges from the press through an annular space between the fixed casing and an unthreaded extension of the screw. The material passes between stationary knife blades which remove any rotation and is then cut up by rotating blades supported by a member rotating with the end of the shaft and adjustable thereon so as to afford, as desired, more or less obstruction to the pressed material. B. M. V.

**Centrifuges.** INTERNAT. GEN. ELECTRIC Co., INC., Assees. of ALLGEM. ELEKTRICITÄTS GES. (B.P. 379,666, 25.6.32. Ger., 26.5.31).—An electric motor is placed within the basket or bowl of the centrifuge. The central part of the motor is the stator. B. M. V.

Rotary[-drum] vacuum filters and like machines. MANLOVE, ALLIOTT & CO., LTD., and F. TITTERTON (B.P. 379,053, 14.7.31).—A method of attaching cloth to the panels of the drum is described. B. M. V.

Sedimentation apparatus. W. W. TRIGGS. From DORR Co. (B.P. 378,890, 13,4.31).—In a settling tank' which may have an irregular bottom, the sludge is raked to one or more outlets by means of drags composed of chains stiffened with wire rope, the upper end of the drag rope being attached to a weighted carriage which is pulled around the circumference of the tank (of rectangular or other shape) by means of an endless chain.

B. M. V.

Apparatus for straining or filtering fluids. F. C. FULCHER (B.P. 378,968, 22.5.31).—A laminated filter on the principle of apparatus described in B.P. 367,379 and 367,458—9 (B., 1932, 372) is constructed in the form of a wheel with the laminations forming the rim, which is preferably of reduced diam. in the middle of the "tread." Separate scrapers are provided for cleaning the narrow spaces and for removal of the mud collected outside. B. M. V.

Adding a fluid to a stream of a fluid or of an air-borne pulverulent solid. J. A. M. TELIET (B.P. 379,214, 19.1.32. Fr., 9.2.31).—The main flow of, e.g., Portland cement in air is passed in a solid stream through the centre of an annular supply device for the other fluid, e.g.,  $H_2O$ . The partial mixture then spreads around a pear-shaped baffle from the interior of which a further supply of additive fluid is drawn, the final mixture then forming a solid stream for delivery to any desired point. B. M. V.

Apparatus for evaporating industrial liquids. B. S. HUGHES, ASST. to ZAREMBA CO. (U.S.P. 1,840,234, 5.1.32. Appl., 27.4.29).—The heating element comprises a horizontal bundle of tubes inserted at one end into a vertical chamber for liquid in the lower part and for separation of liquid and vapour in the upper part, no separating wall being provided between the zones. The liquid makes four passes through the tubes, the no. of tubes in each pass increasing in succession; the hoods for returning the liquid and vapour to the next pass are of such a size as to permit expansion.

#### B. M. V.

Centrifugal fractionating apparatus. J. B. HEID, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,839,097, 29.12.31. Appl., 31.5.28).—The vapours flow parallel to the horizontal axis of a casing; liquid is admitted and removed through perforations in the top and bottom of the casing. A rotor with longitudinally extending radial blades is provided. B. M. V.

Dephlegmator. L. C. HUFF, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,839,030, 29.12.31. Appl., 4.10.22. Renewed 26.12.28).—The vapour inlet is at the bottom of the tower below a set of parallel corrugated plates; in the upper part of the tower a cooling medium flows downwards within a pipe coil, then upwards to a no. of sprays, and again downwards in the form of spray in contact with the vapour. B. M. V.

Apparatus for separating liquids. B. D. COMYN (B.P. 379,092, 25.8.31 and 10.5.32).—The mixed liquids of different sp. gr. are passed into a vertical cylindrical vessel through an aperture in the shell extending over a considerable portion of the height. After entry the liquid passes between spiral guides to the centre, gradually separating into heavier and lighter layers, which are drawn off through axial outlets. B. M. V.

Preventing loss from tanks containing readily vaporisable fluids. S. J. R. STEWART and F. V. W. SWANTON (B.P. 379.051, 13.7.31. Union of S. Afr., 14.3.31).—The atm. is removed from the top of the tank, passed through a cooler, and returned to a lower point by means of thermo-siphon circulation. A gasometer device is provided to take up expansion and contraction due to changes of temp., but for filling and emptying purposes breather valves are provided after and before the cooler, respectively. B. M. V.

Friction material. G. F. MEEHAN and A. L. KENNEDY, Assrs. to W. DORT (U.S.P. 1,840,909, 12.1.32. Appl., 30.7.24. Renewed 27.5.31).—A mixture of asbestos fibre with a heavy-metal salt of the kelp leaf (prepared, *e.g.*, by dissolving the dried leaves in aq.  $Na_2CO_3$  and adding a Cu, Fe, Zn, or Al salt), with the addition, if desired, of scrub palmetto root extract and aq.  $NH_3$ , is moulded under pressure and dried.

L. A. C.

[Electric boiler for] vapour pumps such as diffusion pumps. GEN. ELECTRIC CO., LTD., and E. GALLIZIA (B.P. 380,471, 4.6.31).

Spray nozzles for liquids. SIMON-CARVES, LTD., and V. H. ADAMS (B.P. 380,581, 31.8.31).

[Clamping device for the covers of] centrifugal separators. HOPKINSONS, LTD., and R. A. HOPKINSON (B.P. 379,096, 27.8.31).

# II.—FUEL; GAS; TAR; MINERAL OILS.

Coal classification. H. BODE (Proc. III Int. Conf. Bit. Coal, 1932, 2, 878-896).—A discussion.

CH. ABS.

Petrographic character of coking coals. A. DUPARQUE (Compt. rend., 1932, 195, 52-53).-Good coking coals are intimate mixtures, in suitable proportions, of active elements (vitrain and gelatinised wood) and an inert element (fusain), which latter, however, plays an important part in the carbonisation process. A. B. M.

Stability of kukkersite. K. LUTS (Chem.-Ztg., 1932, 56, 721-722).—The heat of combustion of a finely powdered specimen of bituminous limestone fell from 8918 g.-cal. per g. in 1928 to 8659 g.-cal. per g. in 1931. Contrary to reports of rapid oxidation etc., therefore, the mineral is fairly stable. The H content of the powder falls by only about 0.01% per hr. when the sample is heated at 105°, and the yield of oil by

about 0.1% per hr. The mineral is more stable than, e.g., lignite (cf. B., 1930, 595). H. F. G.

Rôle of humic acids in briquetting brown coals. I. L. BLUM (Proc. III Int. Conf. Bit. Coal, 1932, 2, 646—665).—Only coals containing appreciable amounts of free humic acids are briquettable without binder. Proximate analytical vals. are given for coals, peat, and wood. CH. ABS.

Determination of moisture in coal. H. A. J. PIETERS and H. KOOPMANS (Chem. Weekblad, 1932, 29, 509—513).—The method of drying in air at 105° is satisfactory for ordinary technical purposes, but more accurate results are obtained by drying at 105° in a stream of  $CO_2$  and weighing the evolved  $H_2O$ , or by distillation with xylene. Extraction of the  $H_2O$  by MeOH or EtOH, and determination of the  $H_2O$  content of the alcohol either by observing the miscibility with petroleum, or preferably, since the alcohol can then be recovered, with tetralin, gives accurate results; extraction with EtOH, however, must be carried out at the b.p., and should not be employed for very soft coals. Full details are given of the various methods examined. H. F. G.

**Determination of the alkali-soluble ulmins in** coal. E. STANSFIELD and K. C. GILBART (Fuel, 1932, 11, 347-349).— $\frac{1}{2}$  g. of the finely-pulverised coal is fused with 1 g. of KOH with exclusion of air, the cooled mass is boiled with 50 c.c. of H<sub>2</sub>O, and the solution is diluted and filtered from the undissolved coal. The extracted ulmins are determined either by pptn. with HCl and weighing or by boiling with 0·1N-KMnO<sub>4</sub> and titrating the excess of the latter. The determination of the alkali-sol. ulmins in a series of Alberta coals differentiates the lower-rank coals far more strikingly than does ordinary chemical analysis. A. B. M.

Tendency of coal to spontaneous combustion. VI. D. J. W. KREULEN (Chem. Weekblad, 1932, 29, 518 -521; cf. B., 1932, 757).—The initial temp. (I.T.) of active oxidation of coal is not raised by extraction with solvents; after extraction with  $C_6H_6$  at 275° and 55 atm. the I.T. in one case was lowered by 11°. The I.T. is very much higher for wood charcoal and activated C (norit) than for coal. S. I. L.

Sulphur distribution in the combustion of coal and coke in the light of recent investigations on the behaviour of sulphur compounds of calcium and magnesium. I. TRIFONOV (Brennstoff-Chem., 1932, 13, 328–329).—SO<sub>2</sub>, which is absorbed by CaO or CaCO<sub>3</sub>, giving CaSO<sub>3</sub> (which at temp. above 650° decomposes into CaSO<sub>4</sub> and CaS), may be evolved again at temp. above 850°, e.g.,  $3CaSO_4 + CaS = 4CaO +$  $4SO_2$ , and, with excess of air, CaS +  $3O = CaO + SO_2$ . MgO and MgCO<sub>3</sub> have little binding power for S since any MgSO<sub>3</sub> formed decomposes again at relatively low temp.; MgSO<sub>4</sub> and MgS are also more easily decomposed than the corresponding Ca compounds. A. B. M.

Continuous vertical retorts. C. MOTHON (Gas J., 1932, 199, 393—399).—Tests have been carried out at the Clichy gasworks (Paris) on three settings of continuous vertical retorts comprising : (A) sixteen 6½-ton recuperative retorts of firebrick construction, (B) a similar setting of  $SiO_2$ -brick construction, and (C) twenty 61-ton regenerative retorts of SiO2-brick construction. The regenerative system of setting C, which was especially designed to give the max. possible degree of regeneration for preheating the secondary air and producer gas, is described in detail. The waste gases left the regenerators at  $155^{\circ}$ . Settings A and B, but not C, were fitted with waste-heat boilers. The vertical expansion of the brickwork during the drying out and heating up of the retorts of setting C, which finally amounted to about 120 mm., is shown graphically. The replacement of firebricks by  $SiO_2$  bricks enabled the throughput of a given blend of coals (Saare and Yorkshire) to be increased from 6.5 to 9.2 tons per retort per day; when the blend contained 40% of Courrières coal the max. throughput was 8.1 tons. The regenerative setting had the following advantages over the recuperative setting : (a) increased yield (by about 5%) of therms per ton, (b) 5-8% greater throughput, (c) lower fuel consumption even when account is taken of the heat content of the waste-heat steam from the latter setting, (d) greater ease of control, and (e) greater uniformity in operation and in temp. A. B. M.

Sulphite-pulp residue burnt in powdered-fuel installations. E. BLAU (Chem.-Ztg., 1932, 56, 649– 650).—The crude liquor is conc. in stages to a syrup containing 40% H<sub>2</sub>O and the latter is passed through a rotary dryer of Cu, yielding "cellulose pitch powder" of H<sub>2</sub>O content 6.8%, calorific val. 3600—3800 kg.-cal./kg. It cakes on heating to 150° or on long storage in a damp atm. It can be handled in the same way as powdered coal. It does not burn well if sprinkled on a bed of solid fuel; it must be burned by intermixture with O<sub>2</sub> while in suspension. Structural details are discussed. C. I.

Heating sawdust under pressure in presence of reduced iron. H. BERGSTRÖM and K. N. CEDERQUIST (Iva, 1931, No. 2, 44-45).—Dry sawdust (200 g.), reduced Fe (200 g.), and H<sub>2</sub>O (80 g.) were heated in a rotary autoclave at 425° and a final pressure of 158 atm. The gas contained CO<sub>2</sub> 39·0,  $C_nH_{2n}$  1·0,  $O_2$  0·2, CO 2·0, H<sub>2</sub> 38·3, and CH<sub>4</sub> 15·8%. When heated at 180° the residue gave 13 g. of oil and 83 g. of aq. solution. Extraction of the autoclave residue with Et<sub>2</sub>O gave 70 g. of thick tar, of which 53% was insol. in 10% NaOH solution and consisted of unsaturated hydrocarbons. The yield of oil and tar was 41·5% on the wt. of dry wood. CH. ABS.

Improving the quality of Upper Silesian coke. IV, V. W. SWIENTOSŁAWSKI, B. ROGA, and M. CHORĄŻY (Przemysł Chem., 1932, 16, 121—128, 141—150; cf. B., 1932, 534).—IV. The Wölbling oven yields cokes having the same properties as those prepared from the corresponding coals on a technical scale.

V. The quality of coke obtained from a mixture of agglutinating coals of the same type is the mean of that given by each constituent separately. The addition of semi-coke or of coal of high swelling pressure, and in which the plastic phase is relatively resistant to heat, to ordinary agglutinating coal gives coke of higher quality than would follow from this rule. The quality of Upper Silesian coke might thus be improved by relatively

small admixtures of high-quality coking coal to Silesian coal. R.T.

**Properties of natural and artificial graphite.** I. A. SHAPIRO and V. S. VESELOVSKI (Min. Suir., 1931, 6, 265-274).—Fine graphite dust has a higher electrical resistance than coarse; the ash content has only a slight influence. The temp. at which oxidation begins in air is the lower the longer the heating has been continued. Cryst. graphites have practically the same temp. of oxidation (730°), whilst amorphous graphite begins to oxidise at about 537°. Ch. Abs.

**Regeneration of bone black.** N. N. KUDELYA and I. R. TUGAI (Nauk. Zapiski Tzuk. Prom., 1931, **13**, 731— 745).—The material is boiled for 1—1.5 hr. with 0.2%of its wt. of soda, and, if necessary, neutralised with acid; thermal regeneration is unnecessary. CH. ABS.

Pressure hydrogenation of solid and liquid hydrocarbons in the laboratory. I. Apparatus. F. LAUPICHLER (Chem. Fabr., 1932, 5, 305-311).--Various types of bomb are described and their advantages and disadvantages are discussed. Particular attention is given to the problems of determining and controlling the temp. distribution in the bomb, and of mixing the gas and raw material efficiently during hydrogenation. A. B. M.

Processing of coal and oil with special regard to the catalytic high-pressure hydrogenation. H. G. GRIMM (Proc. III Int. Conf. Bit. Coal, 1932, 2, 49-65).—A review. CH. ABS.

Diagrams for the processes of combustion of industrial gases. M. CZYZEWSKI (Przeglad Tech., 1929, 68, 769—777).—The diagrams indicate the products of combustion of producer, blast-furnace, and coke-oven gases, the amount of gas obtainable from 1 kg. of pit coal, its calorific val., the air necessary for combustion of 1 cu. m., the amount of resulting flue gas, and the heat loss in the flue gas. CH. ABS.

Naphthalene content of gases from horizontal retorts and vertical chamber ovens. O. HOLMQVIST (Gas- u. Wasserfach, 1932, 75, 700—701).—On changing over from horizontal retorts to vertical chamber ovens in the Örebro gasworks (Sweden) the  $C_{10}H_8$  content of the gas fell from approx. 30 g. to 13 g. per 100 cu. m. The comparison was made under conditions, *e.g.*, of oven temp., arrangement of condensing system, etc., which were as nearly as possible the same in the two tests. A. B. M.

How far is it possible to remove naphthalene from gas in the benzol scrubber ? K. BRÜGGEMANN (Brennstoff-Chem., 1932, 13, 329—334).—In the first distillation for the recovery of the benzol from the wash oil some of the latter is carried over and is separated from the benzol by a second distillation. Before returning this wash oil (" pan oil ") to the system it is usually cooled to separate as much of the accompanying  $C_{10}H_8$ as possible. In such a system a state of equilibrium is eventually reached with appreciably const. concns. of  $C_{10}H_8$  in the circulating oil and in the treated gas; in one case the latter concn. amounted to 7—8 g./100 cu. m. It may happen that no solid  $C_{10}H_8$  separates from the " pan oil"; nevertheless an equilibrium is again reached,  $C_{10}H_8$  being removed from the system in the second benzol distillate; the concn. of  $C_{10}H_8$ in the treated gas is higher under these conditions, *e.g.*, 10—20 g./100 cu. m. This concn. can be reduced to a negligible val. by submitting the "pan oil" to steam-distillation before returning it into circulation. A. B. M.

Tetralin process [for prevention of naphthalene deposits in gas mains]. F. SCHUSTER (Gas- u. Wasserfach, 1932, 75, 693-694; cf. B., 1931, 6).-Separation of liquid tetralin in the mains near the point of introduction is negligible. Although the tetralin may remove the inner coating from asphaltcoated mains, a protective layer of tetralin itself is formed and prevents corrosion. The v.p. and solubility relations of tetralin and C10H8 are such that mixtures which condense from the gas on cooling are always liquid, even when the gas contains other hydrocarbon vapours (cf. De Voogd, Het Gas, 1931, 246); in this respect tetralin is preferable to other possible solvents. The concn. of C10H8 in gases in equilibrium with solutions of  $C_{10}H_8$  in tetralin can be calc. by means of the Raoult-van't Hoff rule (cf. Gluckauf, 1930, 1334). During 4 years' operation of the process at Stuttgart gasworks, no deleterious action of the tetralin vapour on the rubber packing or on the meter leathers has been observed. The action on rubber depends greatly on the quality of the latter (cf. B., 1932, 519). A. B. M.

Asphalt from Great Okha Asphalt Lake. S. S. NAMETKIN and S. N. PAVLOVA (Neft. Choz., 1931, 20, 86).—The asphalt, which is intermediate between Trinidad and Bermudez lake asphalt, contained bitumen  $89 \cdot 7$ , ash  $1 \cdot 18$ ,  $H_2O 9 \cdot 03\%$ , and gave Kraemer–Sarnow softening point  $73 \cdot 0^\circ$ , penetration 17, asphalt  $42 \cdot 8$ , resins  $19 \cdot 4$ , oil  $37 \cdot 8\%$ . The resins were adsorbed by SiO<sub>2</sub> gel. CH. ABS.

Thermal behaviour of neutral oil from browncoal tar. A. HAGEMANN and K. I. SKÄRBLOM (Braunkohle, 1932, 31, 152-157, 171-175; Chem. Zentr., 1932, i, 2262) .-- Concurrent reactions are: (1) polymerisation of unsaturated and S-containing compounds which takes place at moderate temp., (2) fission of apparently saturated compounds to benzine and gas, (3) elimination of H2 and H-rich groups from the polymerides with production of coke. Unsaturated hydrocarbons are formed from monomeric compounds. H<sub>2</sub> under high pressure (above 390°) saturates the double linkings of the monomeric compounds and arrests the elimination of H<sub>2</sub>. Polymerides produced at low temp. are unsaturated, but have a low tar no. and a sufficiently flat viscosity curve for lubricating oil requirements. A. A. E.

Pressure-hydrogenation of low-temperature tar. G. FREE (Brennstoff-Chem., 1932, 13, 321-328). —Data are given relating to the hydrogenation of a low-temp. tar, obtained by carbonising a bituminous coal in a rotary (K.S.G.) retort, in a small high-pressure autoclave. At 425°, under an initial pressure of 100 atm. and in the absence of catalysts, there was a marked increase in the low-boiling constituents and a considerable evolution of  $CH_4$ . The greatest effect was produced in the neutral oil fraction boiling above

 $300^{\circ}$ ; about 50% of the tar acids was converted into neutral oil; the tar fractions boiling below  $300^{\circ}$  were little affected. The catalytic effect of  $NH_4$  molybdate was very marked even at  $300^{\circ}$ ;  $Fe_2O_3$  had very little catalytic effect at that temp. A method of solvent analysis is described which proved suitable for the examination of the products. A. B. M.

Extraction of phenols from carbolic oils. J. DANKOWSKI (Przemysł Chem., 1932, 16, 151—155).— The solubility of neutral oils in aq. NaOPh containing free phenols increases with relative concn. of free phenols in the NaOH layer, and with the relative concn. of carbolic oil to aq. NaOH taken. If 6% NaOH is taken for extraction, the amount may be 85% of theoretical without dissolution of neutral oil in the aq. layer; with 12% NaOH a 10% deficit causes dissolution of neutral oil, whilst even the theoretical quantity of 18% NaOH gives bad results. Aged oils give unsatisfactory extraction, owing to their higher content of polymerised bituminous substances, which stabilise emulsions of neutral oil in aq. NaOPh. R. T.

Determination of sulphur in crude petroleum. M. A. PLONSKIER (Ann. Chim. Analyt., 1932, [ii], 14,154— 156).—A sample (about 10 g.) is burned in a lamp and cooled by  $H_2O$ , a slow stream of  $O_2$  being delivered from a forked tube on opposite sides of the flame in order to obviate the usual difficulties of soot formation and the extinction of the flame by the deposition of tar on the wick. The products of combustion are collected and examined in the usual way. The wick is raised from time to time during the combustion, which is allowed to proceed for 3 hr., and, in the calculation, allowance is made for the wt. of wick consumed. H. J. D.

Aniline-point method for determining aromatic hydrocarbons [in petroleum]. E. A. ROBINZON (Neft. Choz., 1932, 22, 226—230).—The method employing total turbidity gives slightly higher vals. than that employing the appearance of a ring, but the difference is within the experimental error. That of determining the NH<sub>2</sub>Ph point by using equal amounts of gasoline and NH<sub>2</sub>Ph gives vals. differing from those obtained when a max. of NH<sub>2</sub>Ph is applied, but the vals. are identical for gasolines free from aromatic hydrocarbons. Both methods can be used when different coeffs. are applied. CH. ABS.

Advisability of "doctor" treatment [of petroleum]. A. Y. KUBULNEK (Neft. Choz., 1932, 22, 175— 176).—The treatment does not remove all the S compounds, but renders them inactive; they are reactivated under the conditions existing in internal-combustion engines, and cause corrosion. S compounds can be removed with ZnCl<sub>2</sub> or OCl'. CH. ABS.

Determination of unsaturated compounds in "Crackbenzin." P. A. SMIRNOV (Neft. Choz., 1928, 15, 217-221).—Treatment with NO at  $-15^{\circ}$  to  $-20^{\circ}$ gave 3 layers; the lower two layers were treated with 20% NaOH and the upper with 10% NaOH, and the solutions distilled with steam. The measured gasoline distillate was diluted with light petroleum and purified with SiO<sub>2</sub> gel. The gel was extracted with Et<sub>2</sub>O, the evaporated residue measured, and its vol. subtracted from that of the benzine distillate. The greatest difference between parallel determinations was 1.42% of unsaturated compounds. CH. Abs.

Selection of [oil-]cracking stocks involves accurate analysis. A. W. TRUSTY (Refiner Nat. Gas. Mfr., 1932, 11, 335).—Three criteria are : distillation range, C residue or tar no., and NH<sub>2</sub>Ph no. Vals. are given for various stocks. An ideal cracking stock is a heavy closely fractionated gas oil with a low C residue, low tar no., and low NH<sub>2</sub>Ph no. CH. ABS.

Vapour-phase cracking in the Vickers unit. E. F. KOROVATZKI (Neft. Choz., 1932, 22, 217-222).-Results obtained with a kerosene distillate produced from cracked oil unsuitable for illuminating or motor fuel are described. CH. ABS.

Cracking of Manchurian shale oil. G. EGLOFF and E. F. NELSON (J.S.C.I., 1932, 51, 326-327 r).--Comparison of the product yields from Manchurian shale oil cracked by the flashing and non-residuum methods shows that the choice of method depends on the type of product required. The flashing method gave  $52 \cdot 6 - 55 \cdot 9\%$  of cracked gasoline and the coking process  $67 \cdot 5\%$ . The amount of gas of high B.Th.U. content produced by the non-residuum method is >by the flashing method. The remaining cracked product from the flashing method is  $26 \cdot 3 - 32 \cdot 6\%$  of cracked residuum and no coke, whilst the other method produces coke and no fuel-oil residuum. E.S.H.

Knock ratings of  $\Delta^{a}$ -olefines. Significance of olefines in cracked petrol. F. H. GARNER, R. WILKINSON, and A. W. NASH (J.S.C.I., 1932, 51, 265-271r).—The knock ratings of the  $\Delta^{\alpha}$ -olefines from C<sub>2</sub>H<sub>4</sub> to nonene have been determined on the S.30 engine, at cooling-jacket temp. of 100° and 150°, when blended with a standard reference petrol to the extent of 20 vol.-%. The effect of PbEt<sub>4</sub> on these blends has been examined in the cases of the liquid members of the series. C2H4 and C3H6 were tested as wt.-% blends with the reference petrol, and on this basis, with a jacket temp. of 100°, are superior to  $C_6H_6$  as anti-knocks. The results show that the "blending octane nos." of the hydrocarbons increase from C<sub>2</sub>H<sub>4</sub> to butene, and then decrease progressively as the length of the C chain increases. The fall in octane no. caused by the use of the 150° jacket temp. progressively decreases with increased length of the mol. until with octene and nonene the "blending octane nos." are greater at 150° than at 100°. The addition of unit concn. of PbEt<sub>4</sub> raises the octane no. of the olefine-petrol blend by an amount which is greater, the longer is the olefine mol.

Fluid fuels to-day and to-morrow. A. E. DUNSTAN (Chem. and Ind., 1932, 822-831, 846-855).—Jubilee memorial lecture.

Transport of benzene during the winter in tank cars. V. S. YABLONSKI, P. P. SHUMILOV, and V. M. POKROVSKI (Neft. Choz., 1931, 20, 76–85). CH. ABS.

Swedish light benzines. B. HOLMBERG (Iva, 1930, No. 2, 26-34).—Light oil from the hydrogenation of C and from shale gas was examined. CH. ABS.

Acid treatment of kerosene. A. G. ZAKHARENKO and M. I. MIRZAKULIEV (Neft. Choz., 1931, 20, 203-206).—The colour of distillates treated with NaOH  $(d \ 1 \cdot 06)$  was unchanged. Colours of distillates subjected to various treatments with  $H_2SO_4$  (d 1.81) and NaOH are recorded. CH, ABS.

Modification of Lachman's method of treating gasoline with zinc chloride. A. Y. KUBULNEK (Neft. Choz., 1932, 22, 104-106).-To avoid corrosion due to the formation, in presence of mercaptans, H<sub>2</sub>S, etc., of HCl and ZnS, S is first removed with 5% NaOH solution and the gasoline subsequently treated with excess of H<sub>2</sub>O. CH. ABS.

Preparation of low-knocking gasoline. A. D. PETROV (Neft. Choz., 1932, 22, 95-101).-A discussion. The thermal condensation of olefines present in gases from vapour-phase cracking proceeds at atm. pressure and in presence of ZnCl<sub>2</sub>, in part according to the scheme :  $2CH_2:CH_2 \rightarrow CH_2:CMe_2$ ;  $2CH_2:CMe_2 \rightarrow CMe_3:CH:CMe_2$ . The olefines of high mol. wt. are then cracked on further heating, forming diolefines and aromatic hydrocarbons.

CH. ABS.

Mercury and thiocyanate numbers of cracked gasoline. P. S. PANYUTIN and N. V. MILOVIDOVA (Coll. Rep. Motor Fuel Res., U.S.S.R. Trans. Sci. Automobile-Tractor Inst., 1931, 21, 33-44).-Characteristics of Baku, Grozni, and Petrograd cracked gasoline, and the I and CNS vals. after treatment with Hg acetate (Tausz, modified) or thiocyanate (Kaufmann) are recorded. CH. ABS.

Preventing escape of mercaptans in reclaiming caustic soda [from oil refining]. W. MENDIUS (Refiner Nat. Gas. Mfr., 1932, 11, 370).-Mercaptans, mercaptals, sulphides, and disulphides are removed from spent NaOH by countercurrent scrubbing with steam or dry S-free gas. CH. ABS.

Contact filtration of lubricating oils. P. D. PETTI (Neft. Choz., 1931, 20, 90-94) .- Natural clays of the fuller's-earth type require preliminary activation with H<sub>2</sub>SO<sub>4</sub> for decolorising. Acids are removed similarly by these and by clays of the bentonite type. "Gumbrin " clay from Tiflis has at 200° a higher decolorising power than fuller's earth. Treatment with H2SO4 quintupled the activity of a Tiflis bentonite. CH. ABS.

Laboratory control in dewaxing lubricating oils. N. VASILIEV and V. SHILE (Azerbaid. Neft. Choz., 1932, No. 4, 63-68) .- The pressability of wax distillates is measured by the rate of filtration through laboratory CH. ABS. vac. filters.

Edeleanu method applied in the treatment of lubricating oil distillates of heavy crude oil from Binagadi. A. S. VELIKOVSKI and I. V. POZNYAK (Neft. Choz., 1931, 20, 474-482).-Characteristics of machine and cylinder oil distillates after treatment with liquid  $SO_2$  at  $-10^\circ$  are recorded. CH. ABS,

Regeneration of spent lubricating oils. A. I. VORONOV and M. D. SUIRENKOV (Neft. Choz., 1931, 21, 156-161).-Lubricating oil bottoms (65%) and gas oil (35%) were treated with the same amount of spent oil (consts. recorded); the oil was then treated with acid and distilled, yielding 61-63% of an automobile oil CH. ABS. distillate (consts. recorded).

Utilisation of [oil-]refinery residues. V. REIS-NER (Azerbaid. Neft. Choz., 1932, No. 4, 58-63) .- High-

grade lubricating oils can be recovered from refinery residues by extraction with solvents. Cracked residuum is a possible source of lubricating oils and asphalt; highvac. distillation offers commercial possibilities.

CH. ABS.

Chemical treatment of rotary drilling fluids. H. C. LAWTON, H. A. AMBROSE, and A. G. LOOMIS (Physics, 1932, 2, 365-375).-Low viscosity with const. d is desirable. Suitable adjustment of the NaOH concn. in reagents employed for lowering viscosity imparts desirable thixotropic properties to the clay dispersion. CH. ABS.

Breaking up alkaline water-oil emulsions under pressure, A. I. VORONOV (Neft. Choz., 1931, 21, 162-164).-About 30% better yield of oil results when the emulsions are treated at 1-2 atm. and 120-130°. CH. ABS.

Appraisal of mineral oil bleaching-earths. W. SCHAEFER (Chem. Umschau, 1932, 39, 177-179) .--Differences in bleaching power are not necessarily parallel to the "hydrolytic acidities" (cf. Utermöhlen, B., 1931, 881). The tendency of a bleached oil to redarken can be gauged from the colour developed after storage in contact with an Fe plate for 10-30 hr. at 60-100°. E. L.

Rectifying units.-See I. Alcohols etc. from hydrocarbons. Oxidation of higher hydrocarbons. AcOH from charcoal-kiln gases. Pine-wood tars. Heavy wood-spirit oils.—See III. Asphalt in varnish .- See XIII. Gas-mask charcoal for COCI, and HC1.-See XXIII.

See also A., Oct., 1001, Flame movement. Combustion of hydrocarbons. 1012, Continuously operating laboratory furnaces.

#### PATENTS

Retorting of carbonaceous material. H. N. HERRICK, ASST. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,838,622, 29.12.31. Appl., 30.4.25).-The material is carbonised in a vertical retort (A) by passing through it the hot gases from an adjacent combustion chamber (B). A device, e.g., a reciprocating ram working in a horizontal channel passing through the centre of the charge in the lower part of A, is provided for continuously feeding a part of the carbonised material on to the grate of B. Part of the hot gases from the latter are passed through an annular space surrounding the upper part of A and serves to preheat the material. The gases and volatile products are withdrawn through passages placed at different levels along the carbonising zone and are passed to a condensing system. The uncondensed gases are led to B, and the heavy constituents of the condensed oil may be returned to the carbonising zone of Ato be cracked therein. The air for combustion is used to cool the lower part of A and is itself thereby preheated. A. B. M.

Coke-oven battery. J. BECKER, Assr. to KOPPERS Co. (U.S.P. 1,838,294, 29.12.31. Appl., 12.6.26) .-The gas mains which are located at the sides of the battery are so arranged that those supplying coke-oven gas lie within the larger mains supplying producer gas. A. B. M.

Manufacture of coke and combustible gas. A. JOHNSON, ASST. to COMBUSTION UTILITIES CORP. (U.S.P. 1,841,036, 12.1.32. Appl., 28.1.25).—The blow gases are burnt by a supply of secondary air introduced above the fuel bed, the heat liberated being used to superheat the water-gas during the run. Two chequerbrick regenerators are equipped with air, steam, and gas inlets and are used alternately for preheating the blast air, regenerating the sensible heat of the waste blast gases, and superheating the steam for the run periods. The water-gas which is thus superheated without the use of an external vessel is then passed through a vertical shell containing coal, whereby it is enriched by the volatile matter of the coal, coke being formed in the process. R. N. B.

Apparatus for production of carbon black. G. C. LEWIS, ASST. to COLUMBIAN CARBON CO. (U.S.P. 1,838,316, 29.12.31. Appl., 24.6.25).—A current of CH<sub>4</sub> is mixed with the vapour of  $C_{10}H_8$ , creosote, or other tar oil, and the mixture is burned with an insufficient supply of O<sub>2</sub>, the C formed being deposited in known manner on metal plates or channels. The tar oil etc. is passed through preheating tubes arranged above the depositing plates to a saturator through which the CH<sub>4</sub> is passed. A. B. M.

Oil-cracking still. H. E. WIDDELL (U.S.P. 1,841,072, 12.1.32. Appl., 14.8.26).—The temp. of an oil-cracking furnace is controlled by passing a heat-absorbing fluid through pipes adjacent to the furnace walls. The oil to be cracked is then exposed to the cooled gases at preferably 705—870°. R. N. B.

Cracking of hydrocarbons. N. E. LOOMIS and M. R. MEACHAM, ASSIS. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,842,104, 19.1.32. Appl., 22.7.25).—Preheated petroleum oil is passed into a horizontal cylindrical still (A) the vapours from which pass through a dephlegmator (B) to a condenser. The residuum and condensate from B are heated under pressure in a cracking coil and passed into digesting chambers from which they are passed with reduction of pressure to either A or B. D. K. M.

Cracking of hydrocarbons. E. C. HERTHEL, Assr. to SINCLAIR REFINING CO. (U.S.P. 1,842,753-4, 26.1.32. Appl., 18.8.26).—(A) A hydrocarbon oil, of which  $\geq 25\%$ distils below 260° and < 80% distils below 315°, is heated to cracking temp. under pressure by circulating it from a main supply, A, through heating tubes and back to A. The vapours from A are removed under pressure during the heating operation and are refluxed in direct contact with fresh oil of the same character and an oil of the character corresponding to that of the distillate taken off. The admixed, refluxed, and unvaporised components of the oil are returned to A, while at the same time sufficient of the tar-laden oil therein is withdrawn so that throughout the major portion of the operation the amount of oil taken off as final distillate is 50-60% of the total oil to be cracked. (B) The process is conducted at pressures > 125 lb./sq. in. H. S. G.

Cracking of mineral oil. L. MELLERSH-JACKSON. From SUN OIL Co. (B.P. 379,759, 5.6.31).—Oil is passed under high pressure in a continuous stream of restricted cross-section through a succession of confined spaces in each of which the temp. of the oil is fixed by heat exchange with Hg vapour. The oil is heated rapidly through the cracking-temp. range to within 10° of its crit. temp., and during the cracking operation the temp. is raised as the crit. temp. of the oil rises. H. S. G.

**Treatment [cracking] of hydrocarbons.** E. W. BEARDSLEY and M. W. COLONY, ASSTS. to PETROLEUM CONVERSION CORP. (U.S.P. 1,842,318, 19.1.32. Appl., 31.10.25. Cf. U.S.P. 1,715,239; B., 1929, 744).—Oil from which light hydrocarbons are removed by passing down rectifying columns (A) is preheated and then evaporated with the help of steam or part of the fixed gas formed in the process, and the vapours, with or without superheating, are passed into a reaction chamber in which they are cracked by mixing with more of the fixed gas heated in hot-blast stoves. After leaving the preheater the products from the chamber pass into A, the vapours from which are condensed.

D. K. M.

Control of pyrolysis of hydrocarbon oils. F. A. HOWARD and N. E. LOOMIS, ASSTS. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,842,096, 19,1.32. Appl., 31.12.26).—Oil is heated by passage through a heating coil (A) and is discharged into a digestion drum (B) held under pressure where cracking is completed. A substantial quantity of oil is vaporised and enters an auxiliary drum (C) wherein a part is condensed and returned to B for the purpose of regulating the temp. The uncondensed portion is drawn from the top of Cand is discharged to a separating zone together with a stream of oil from the lower portion of B. Feed to Ais preheated by passage through a coil in C, H. S. G.

Treatment of petroleum oil. R. CRoss, Assr. to GASOLINE PRODUCTS CO., INC. (U.S.P. 1,841,363, 19.1.32. Appl., 22.7.22).—Oil is raised to cracking temp. in a heating coil and passed to an enlarged reaction zone (maintained under sufficient pressure to prevent substantial distillation) where additional heat to promote cracking is added by electrical heating elements. The converted products are discharged into a cooling stage where the contained heat is used to preheat oil to be treated. H. S. G.

Conversion of kerosene into lower-b.p. hydrocarbon liquids. H. C. WADE (U.S.P. 1,842,221, 19.1.32. Appl., 24.3.25. Renewed 21.4.31).—A mixture of atomised kerosene and  $H_2O$  (the  $H_2O$  vapour being 17 vol.-% of the oil +  $H_2O$  vapour) is heated to  $< 649^\circ$  by passing through a coil in the outlet of which is a metal catalyser, *e.g.*, nichrome wire. D. K. M.

Oil-refining still. O. E. ANDRUS and S. HERMAN-SON, ASSTS. to A. O. SMITH CORP. (U.S.P. 1,840,305, 12.1.32. Appl., 13.7.28).—A thin sheet of corrosionresisting material is united to a thick sheet of steel by a no. of spaced electric welds. The composite sheet is rolled or bent to the required shape at preferably 705°. R. N. B.

Refining of [petroleum] distillates. P. BORGSTROM, Assr. to L. L. REEVES (U.S.P. 1,840,269, 5.1.32. Appl., 18.3.30).—Cracked petroleum distillates are sweetened and degummed by treatment with distillates in which org. peroxides have developed in the presence of fuller's earth in the liquid or vapour phase. S may then be removed by washing with H<sub>2</sub>O. D. K. M.

Treatment [purification] of hydrocarbons. R. CROSS, ASST. to CROSS DEVELOPMENT CORP. (U.S.P. 1,840,158, 5.1.32. Appl., 22.10.25).—Hydrocarbons produced by the cracking of petroleum are desulphurised and degummed by passing in the vapour (direct from the cracking plant) or liquid phase through a bed of a metal, its oxide, or a salt with an affinity for S, e.g., CuO, and then through a bed of absorbent clay, e.g., bentonite. D. K. M.

Refining of hydrocarbons [lubricating oils]. F. A. APGAR, ASST. to SINCLAIR REFINING CO. (U.S.P. 1,841,271, 12.1.32. Appl., 10.2.28).—Lubricating oil vapours are passed through an adsorbent catalyst, e.g., fuller's earth or SiO<sub>2</sub> gel, at a pressure < 20 in. Hg and the product is condensed. Any wax present is then in a potentially crystallisable form and is easily removed by chilling. R. N. B.

Decolorisation of lubricating oils. T. T. GRAY, Assr. to GRAY PROCESSES CORP. (U.S.P. 1,839,388, 5.1.32. Appl., 7.2.28).—Lubricating oil vapours are passed through fuller's earth in the presence of the same oil either in the liquid form produced by partial condensation or introduced as a spray. D. K. M.

Imparting a green fluorescence to lubricating oil. J. C. BLACK, W. D. RIAL, and J. R. MCCONNELL, Assrs. to RICHFIELD OIL CO. OF CALIFORNIA (U.S.P. 1,842,856, 26.1.32. Appl., 18.5.27).—Coal-tar pitch (1 pt.) is extracted with a solvent (1—4 pts.), e.g., gasoline or  $C_6H_6$ , near its b.p., or with lubricating oil stock at 110—149° in the presence or absence of a decolorising clay. When the extract is mixed with lubricating oil, volatile hydrocarbons and/or clay being removed before or after mixing, a green fluorescence is imparted to it. If clay is not used in making the extract, it may be added directly to the original lubricating oil and removed later. D. K. M.

Reclaiming used lubricating oil. J. F. HANKE, Assr. to UNITED OIL REFINERTES (U.S.P. 1,842,983, 26.1.32. Appl., 18.4.27).—Used lubricating oil heated to 38—54° is mixed with  $H_2SO_4$  ( $d \ 1.84$ ; 5—7 lb. per barrel) for several hr., the acid neutralised with aq. NaOH ( $d \ 1.26$ —1.30) or milk-of-lime, and the oil washed. The clear oil (150 gals.) at 49—54° is mixed with ZnCl<sub>2</sub> (0.25 lb.) and NaOH (0.25 lb.) or with ZnCl<sub>2</sub> (0.25 lb.) and aq. NaOC1 containing 4.5%(approx.) of available Cl (1 pint), slowly heated to 166°, and after the evaporation of all  $H_2O$  is distilled under vac. D. K. M.

Lubricating oils. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 379,717, 29.4.31).—When a small quantity (>0.5%) of a hydrocarbon of mol. wt. >800, preferably >2000, which does not undergo appreciable decomp. when heated at 200° for 5 min., e.g., polymerised styrene or its hydrogenated derivative, hydrogenated polymerised diolefines, is added to a lubricating oil its lubricating properties are improved and its viscosity is increased. Alternatively, the basis hydrocarbon, e.g., styrene, may be added to the oil and then polymerised and hydrogenated (if desired) therein. D. K. M. Pressure treatment for oils and the like. J. H. HIRT, Assr. to L. J. HIRT (U.S.P. 1,840,164, 5.1.32. Appl., 23.4.29).—Colloidal matter is removed from oils (etc.) by filtering under pressure (500 lb. per sq. in.) through a bed of finely-divided porous material, *e.g.*, volcanic ash, compressed to about 700 lb. per sq. in. D. K. M.

Purifying [removing phosphorus from] oils. J. M. MICHEL, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,841,754, 19.1.32. Appl., 7.11.30. Ger., 13.11.29).— Oils (e.g., transformer oils, tar oils used as sealing medium in gasometers) contaminated with P are maintained in contact with a finely-divided metal that reacts therewith, e.g., Cu, at a temp. not far removed from the b.p. of the oil and in the presence of an inert gas. The metal phosphide is subsequently separated from the oil. H. S. G.

Transformer oil. B. W. STORY, Assr. to VACUUM OIL Co., INC. (U.S.P. 1,841,070, 12.1.32. Appl., 31.3.26). —The sludging tendency of transformer oils is retarded by addition of cetyl alcohol (0.1%). R. N. B.

Oil-distilling system. J. B. HILL, Assr. to ATLANTIC REFINING CO. (U.S.P. 1,842,756, 26.1.32. Appl., 14.5.26). —Combustible, O<sub>2</sub>-containing, and inert gases are mixed and burned while submerged in and in direct contact with oil contained in a still. The products of combustion are collected and at least a portion is used with the combustible mixture. H. S. G.

Apparatus for making gasoline. O. BEHIMER, Assr. to TEXAS Co. (U.S.P. 1,840,012, 5.1.32. Appl., 30.1.23. Can., 3.12.21).—Petroleum oil heated to 371— 510° is passed into a vertical drum and the vapours are passed to an air condenser the condensate from which is mixed with the raw oil passing to the heating coil, while the vapours are condensed yielding light hydrocarbons. The apparatus is operated under 100—400 lb. per sq. in, D. K. M,

Separation of hydrocarbon vapours. F. A. HOWARD and N. E. LOOMIS, ASSIS. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,842,097, 19.1.32. Appl., 18.11.27) .- Hydrocarbon vapours are subjected to fractional condensation under rectifying conditions to produce a first vapour phase (A) having an end-point of 149—182°, and a first liquid residue (B) which is treated to remove undesirable constituents therefrom. A is further fractionated to produce a second liquid residue (C) and a second vapour phase, comprising hydrocarbons normally gaseous, which is passed into countercurrent relationship to an absorption medium consisting of Bafter treatment, the process being carried out under 50 lb./sq. in. The absorption medium is heated and fractionated under 5 lb./sq. in. to obtain gases and a liquid, substantially free of constituents of lower b.p. than that of  $C_4H_{10}$ , which is combined with C.

H. S. G.

Removing dust from coal [on vibrating screen]. SIMON-CARVES, LTD., and V. H. ADAMS (B.P. 380,196, 11.7.31).

Apparatus for drawing off gas by suction in chamber ovens. C. STILL (B.P. 380,405, 23.5.32. Luxemb., 23.5.31).

Gas fires. South METROPOLITAN GAS Co., D. CHANDLER, and E. V. EVANS (B.P. 380,352, 15.2.32).

Gas burners. COMMERCIAL GAS Co., and W. R. PARRIS (B.P. 380,118, 9.6.31).

Oil-burning system. PEABODY, LTD. From PEA-BODY ENG. CORP. (B.P. 380,117, 9.6.31).

Furnace for low-grade fuel. Separation of materials [coal]. Cracking etc. of petroleum oils. —See I. Phenol etc. from tar. Ketones from shale. Anthracene.—See III. Plant waste as fuel.—SeeV. Recovery of Mo. Solid CO<sub>2</sub>. H<sub>2</sub> from hydro-carbons.—See VII. Bitumen suspensions etc. Road-paving material.—See IX. O<sub>2</sub>-H<sub>2</sub> generator. —See XI, Bituminous paints.—See XIII.

# **III.—ORGANIC INTERMEDIATES.**

Preparation of alcohols, aldehydes, and acids from gaseous hydrocarbons. E. F. KOROVATZKI (Neft. Choz., 1931, 20, 483—491).—A stripped natural gas containing air 1—5,  $CO_2 \ 0.1$ ,  $CH_4 \ 52$ —56,  $C_2H_6 \ 10$ —12,  $C_3H_8 \ 17$ —20, and higher hydrocarbons 10—12% was mixed with air and passed through a furnace, the treatment then being repeated. NO<sub>2</sub> has a scarcely noticeable catalytic effect; Cu causes much decomp. The yields (up to 32% of liquid products) of alcohols, aldehydes, and acids depend on the concn. of O<sub>2</sub> and the velocity of the reaction. In presence of sufficient O<sub>2</sub> the optimal temp. is 400—500°. At 600—700° CO,  $CO_2$ , H<sub>2</sub>O, and unsaturated compounds are formed. The condensate contains up to 10% CH<sub>2</sub>O. CH. ABS.

Autoxidation of higher hydrocarbons and their mixtures. K. IVANOV (Neft. Choz., 1932, 22, 85-94). -For identical oxidation conditions paraffin-base oils yield more fatty and less OH-acid than naphthene-base oils. By oxidation of valueless light oils with  $O_2$  at 15 atm. for 3 hr. in presence of Na, Li, Mn, Fe, Pb, and Cu naphthenates, 30% was converted into acids of high mol. wt., 66% of the product being fatty acids suitable for soap manufacture. Data concerning the oxidation of acid-treated Grozni and Balakhani gas oils are recorded. Oil and distillates washed with tap H<sub>2</sub>O before oxidation were oxidised more completely than those washed with distilled H<sub>2</sub>O. As a rule, catalysts increase the oxidisability of slightly refined oils, whilst highly refined oils are little affected. CH. ABS.

Recovery of acetic acid from pine charcoal kiln gases by direct extraction with tar oils. H. BERGSTRÖM and K. N. CEDERQUIST (Iva, 1932, No. 2, 28-36).—The AcOH was extracted (90%), after separation of the tar, with oils distilled from wood tar. Continuous fractional distillation gives an oil-free product; the oils are not readily separated when the  $H_{2}O$  content is very low. CH. ABS.

Phenol and guaiacol in pine-wood tars. H. BERGSTRÖM and K. N. CEDERQUIST (Iva, 1931, No. 1, 14—16).—Distillation of wood tar in a column at 175° and 20 mm. pressure gave 65% distillate and 5% loss; extraction of the distillate with 2N-NaOH afforded PhOH (0.25% of the tar). About 40% of the crude phenols was guaiacol. CH. ABS. Phenols in heavy wood-spirit oils. H. BERG-STRÖM and K. N. CEDERQUIST (Iva, 1931, No. 1, 18).— The oil (2% of the 100% MeOH produced) contained 1.6 g. of phenols per 100 kg. of dry wood treated.

CH. ABS.

[Colour] reaction for recognising primary cyclic amines and its application to medicaments. J. A. SANCHEZ (Ann. Chim. Analyt., 1932, [ii], 14, 152-154). The reagent consists of a saturated aq. solution of furfuraldehyde containing AcOH (5 drops per 10 c.c. of aldehyde). A few drops when added to a similar amount of a primary cyclic amine give a red coloration. Apparently negative cases require a drop of HCl. When the NH2-group is acid-substituted, hydrolysis is first effected by heating to dryness about 0.05 g. of the amine with a small vol. of 30% NaOH, and testing when cool. An explanation of the probable reaction is offered together with examples of its application to the identification of medicaments, e.g., the differentiation of novocaine and tutocaine from stovaine and cocaine, or the recognition of atoxyl and arsenobenzenes. H. J. D.

Determining aromatic hydrocarbons. Phenols from carbolic oils.—See II. Crude oil of turpentine.—See XIII. Fermentation [products].—See XVIII.  $C_6H_2Me(NO_2)_3$ .—See XXII.

See also A., Oct., 1004, Vapour-phase oxidation of alcohols. 1005, Prep. of formates from  $CO_2$ . Electro-org. reductions and oxidations. 1017, Prep. of alcohols from fats. 1019, Prep. of keten from  $COMe_2$  and of  $COMe_2$  from AcOH. 1029, Prep. of o-chlorobenzoic acid. 1035, Complex salts of hydroxyquinones. 1051, Determination of EtCO<sub>2</sub>H. Test for phenols.

#### PATENTS

**Production of formaldehyde [from methane].** GUTEHOFFNUNGSHÜTTE OBERHAUSEN A.-G. (B.P. 375,314, 20.1.31. Ger., 20.1.30).—CH<sub>4</sub>, with O<sub>2</sub> or air or steam and nitrogen oxides (<1%), is led over a metal catalyst (Pt, W, Cr, Cu, Ag, Ni, Zn, Mn, or Si, or oxides thereof) at temp. below the ignitic n point of the mixture. The yield of CH<sub>2</sub>O is 20—35 g, per cu. m. of CH<sub>4</sub>. C. H.

Production of paraformaldehyde. DEUTS. GOLD-U. SILBER-SCHEIDEANSTALT VORM. ROESSLER (B.P. 375,744, 21.3.32. Ger., 21.3.31).—The water is removed from paraformaldehyde by means of an entraining liquid, e.g., EtOAc, PhMe, COMEEt,  $C_2H_4Cl_2$ , etc., whereby a  $H_2O$ -sol. product free from higher polymerides is obtained. Thus 40% CH<sub>2</sub>O is added through a column to boiling EtOAc at the same rate as  $H_2O$  is entrained by the ester; paraformaldehyde gradually separates in the distillation vessel. C. H,

Production of acetaldehyde and acetic acid from acetylene. GUTEHOFFNUNGSHÜTTE OBERHAUSEN A.-G. (B.P. 376,045, 1.8.31. Ger., 2.8.30).— $C_2H_2$  and  $O_2$  in substantial absence of steam are passed with small amounts of N oxides over inert materials (quartz, porcelain) at temp. (e.g., 287°, 407°) below the ignition point. C. H.

**Production of acetic anhydride.** A. H. STEVENS. From E. BERL (B.P. 377,442, 5.8.31).—AcOH and COMe<sub>2</sub> are passed over  $V_2O_5$ , e.g., at 675°; or the COMe<sub>2</sub> is first converted by  $V_2O_5$  at 675° into keten, which is then led into the vapours or condensate produced from AcOH anhydrised at 730° in contact with pumice. C. H.

Manufacture of reaction products of keten. H. E. POTTS. From CARBIDE & CARBON CHEMICALS CORP. (B.P. 377,574, 27.1.32).—Keten, with other pyrolysis products of COMe<sub>2</sub> etc., is led immediately after formation into a reactive absorbent, *e.g.*, glycol Et ether or AcOH; *e.g.*, COMe<sub>2</sub> is passed over Cu at 600—750° with a contact time of < 0.75 sec., and the keten etc. is quenched > 0.03 sec. later with the reactive liquid. C. H.

Oxidation of ethylenic compounds. IMPERIAL CHEM. INDUSTRIES, LTD., and H. L. RILEY (B.P. 376,306, 8.4.31).— $C_2H_4$  is oxidised to glyoxal by SeO<sub>2</sub> or H<sub>2</sub>SeO<sub>3</sub> at 50—350°; propylene yields methylglyoxal. C. H.

Manufacture of ethylenediamine. G. O. CURME, JUN., and F. W. LOMMEN, ASSTS. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,832,534, 17.11.31. Appl., 12.1.24).—C<sub>2</sub>H<sub>4</sub>Cl<sub>2</sub> is treated with large excess of NH<sub>3</sub>, the product is continuously removed, and the recovered NH<sub>3</sub> is returned to the reaction chamber. C. H.

Manufacture of chlorohydrins [from olefines]. H. TROPSCH and R. KASSLER (B.P. 377,595, 18.2.32. Czechoslov., 18.2.31).—In the reaction between olefines,  $Cl_2$ , and  $H_2O$ , excess of olefine is used (8 vols. to 1 vol.  $Cl_2$ ), and, starting with pure  $H_2O$ , the chlorohydrin concn. is not allowed to pass 20%. Formation of  $C_2H_4Cl_2$ is thus suppressed. C. H.

Production of alcohols from olefines. N. V. DE BATAAFSCHE PETROLEUM MAATS., Assees. of W. ENGS and R. MORAVEC (B.P. 377,583, 8.2.32. U.S., 9.2.31).— An olefine above  $C_3$  (butylene) is contacted with 1 mol. of 90—100% H<sub>2</sub>SO<sub>4</sub> below about 15°, under sufficient pressure to keep the olefine liquid, and the alkyl H sulphate produced is hydrolysed. C. H.

Manufacture of dialkyl sulphates from olefines. N. V. DE BATAAFSCHE PETROLEUM MAATS., Assees. of W. ENGS and R. Z. MORAVEC (B.P. 376,925, 18.6.31, U.S., 20.6.30).—Straight-chain olefines above  $C_3$  are agitated, preferably in liquid phase, with < 0.5 mol. of 99—100% H<sub>2</sub>SO<sub>4</sub>. C. H.

Production of xanthates. H. D. ELKINGTON. From N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 377,077, 14.12.31).—Metal (Na) sec. -or tert.-alkoxides, or mixtures of metal hydroxide and a sec.- or tert.-alcohol, are treated with  $CS_2$  to give xanthates, e.g. Bu<sup>y</sup> xanthates, useful as flotation agents, insecticides, or vulcanisation accelerators. C. H.

Recovery of ketones [from shale oil etc.]. J. DÖDERLEIN (B.P. 376,862, 28.4.31. Ger., 5.5.30).—The crude ketone oil from shale etc. is treated with  $H_2SO_4$ , the slime is diluted, *e.g.*, with the water distilled from the shale, and distilled. Ketones are recovered from the distillate either by salting-out, fractional distillation, or as NaHSO<sub>8</sub> compounds. C. H.

**Production of furfural [from wood pulp].** G. A. RICHTER, ASST. to BROWN CO. (U.S.P. 1,838,109, 29.12.31. Appl., 6.6.30).—The acid wood-pulp cooking liquors are separated from the pulp, without relieving the pressure,

and digested at 121–216°. The vapour (consisting of  $H_2O$ and furfuraldehyde) is blown off from time to time and condensed. The digester relief gas may also be condensed and added thereto. If the spent liquor is free from Ca it is neutralised, conc., and re-acidified before digesting as above. Furfuraldehyde recovered is 60% of the pentosan content of the wood. A. G.

Manufacture of derivatives of higher fatty acids [for use as wetting etc. agents]. J. Y. JOHNSON, From I. G. FARBENIND. A.-G. (B.P. 378,002, 29.4.31. Cf. B.P. 341,053, 343,524; B., 1931, 386, 622).—Fatty acid ( $>C_6$ ) amides are treated with halogenosulphonic acids, or the amide (e.g., oleic amide, dissolved in  $C_6H_6$ ) is converted into the N-alkali (alkaline-earth or Mg) compound, which is heated at 100—250° with a halogenosulphonic acid or its Na salt. The dark, hygroscopic, waxy products form foaming aq. solutions, which are stable to CaO and acids, and disperse Ca soaps etc.

E. L.

Manufacture of derivatives of polyhydric alcohols [wetting, cleansing, and dispersing agents]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 375,842, 26.3.31).—Hydroxyalkyl ethers of aliphatic polyhydric alcohols above  $C_2$  and free from CO groups are etherified or esterified with aliphatic compounds above  $C_5$  and preferably of  $C_{10}$ — $C_{18}$  in such a way as to leave at least 2 OH groups free. Examples are sorbitol hexa-( $\beta$ -hydroxyethyl) ether with lauric acid, linoleic acid, coconut oil acids, or acids  $C_8$ — $C_{11}$  from oxidised paraffin wax, or with  $\alpha$ -dodecylene chlorohydrin. C. H.

Production of aminopyridines. V. V. UTGOFF, Assr. to ROOSEVELT CHEM. Co. (U.S.P. 1,832,799, 17.11.31. Appl., 4.5.27).—In the manufacture of 2-amino- and 2: 6-diamino-pyridines from  $C_5H_5N$  and NaNH<sub>2</sub> the latter is finely divided before reaction, *e.g.*, by agitation of the molten NaNH<sub>2</sub> with a cold oil. C. H.

Manufacture of double compounds of the pyridine series. Soc. CHEM. IND. IN BASLE (B.P. 377,132, 5.3.32. Switz., 7.3.31).—Double compounds of pyridinecarboxylic derivatives with  $H_2O$ -sol. salts of alkalineearth metals are obtained by mixing alcoholic solutions of the constituents. Examples are compounds from CaCl<sub>2</sub> with nicotinic acid, acid chloride, Et ester, diethylamide, picolinic acid, 2-hydroxynicotinic acid; from nicotinic diethylamide with Ca(SCN)<sub>2</sub>, CaBr<sub>2</sub>, CaI<sub>2</sub>, Ca salicylate, MgCl<sub>2</sub>, SrCl<sub>2</sub>; Ca(SCN)<sub>2</sub> with nicotinic methyln-propylamide, picolinic diethylamide, Et collidine-3:5-dicarboxylate, 2-chloronicotinic diethylamide, and Ca picolinate. C. H.

Manufacture of condensation products [triazines] containing nitrogen. I. G. FARBENIND. A.-G. (B.P. 376,603, 6.11.31. Ger., 10.11.30).—An aldehydeammonia above  $C_1$  is heated with a carbamide or guanidine;  $H_2O$  may be removed during reaction by means of a dehydrating agent or an entraining liquid. OH·CHMe·NH<sub>2</sub> and CO(NH<sub>2</sub>)<sub>2</sub> at 70—75° give 2 : 4-dimethyl-6-ketohexahydro-1 : 3 : 5-triazine, m.p. 190.° Similar compounds are obtained from OH·CHMe·NH<sub>2</sub> and guanidine nitrate (m.p. 156—157°) ; OH·CHMe·NH<sub>2</sub> and CO(NH<sub>2</sub>)<sub>2</sub>; aldol-ammonia and  $CO(NH_2)_2$ ;  $OH \cdot CHMe \cdot NH_2$  and  $CO(NHMe)_2$ ;  $CH_2Ph \cdot CH(OH) \cdot NH_2$  and  $CO(NH_2)_2$ . C. H.

Production of pure phenol, cresols, and benzene hydrocarbons from phenols of tar derived from low-temperature carbonisation. OBERSCHLESISCHER BERG- U. HÜTTENMÄNNISCHER VEREIN E.V. (B.P. 376,509, 22.7.31. Ger., 22.7.30).—The crude phenols are heated with  $H_2$  under pressure in absence of catalysts. C. H.

Preparation of metalliferous sulphurised derivatives of phenols. CHEM. FABR. VORM. SANDOZ. (B.P. 375,885, 7.4.31. Ger., 8.4.30. Addn. to B.P. 365,534; B., 1932, 496).—The process of the prior patent is carried out in presence of accelerators, viz., weak inorg. salts (nitrites, hyposulphites, thiosulphates, thionates), org. salts (acetates, formates, lactates) of alkali metals, or I. C. H.

Manufacture of cresol ethers alkylated in the nucleus. RHEINISCHE KAMPFER-FABR., G.M.B.H. (B.P. 375,678, 10.11.31. Ger., 10.11.30).—A tolyl Me or Et ether is condensed with an alcohol above  $C_2$  in presence of 50—80%  $H_2SO_4$  at 20—120°; alkyl enters probably para to the Me. Any SO<sub>3</sub>H groups in the product may be removed by hydrolysis. *tert.*-Bu (m.p. 22°, b.p. 228°) and  $Pr^{\beta}$  derivatives of *m*-tolyl Me ether are described. C. H.

[Manufacture of] aniline-crotonaldehyde condensation products [vulcanisation accelerators]. C. O. NORTH and C. W. CHRISTENSEN, ASSTS. to RUBBER SERVICE LABORATORIES CO. (U.S.P. 1,832,415, 17.11.31. Appl., 18.2.27).—NH<sub>2</sub>Ph is heated with 1 mol. of crotonaldehyde and then with further quantities of the same aldehyde. C. H.

Manufacture of 3-methoxy- and 3-ethoxy-4-hydroxybenzaldehydes [vanillin]. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 376,610, 10.11.31). -4- Hydroxy-3 - methoxy- (or ethoxy-)phenyltrichloromethylcarbinol is converted smoothly into the aldehyde by NaOMe in boiling MeOH or by a boiling solution of NaOH in MeOH. C. H.

Manufacture of halogenated 4'-amino-o-[2-pamino-]benzoylbenzoic acid. E. T. HOWELL, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,832,211, 17.11.31. Appl., 23.2.28).—The acid is treated in conc.  $H_2SO_4$  with  $Cl_2$  or Br at 15—45°. The halogen enters ortho to the  $NH_2$ -group. C. H.

Manufacture of 1-m-hydroxyphenyl-2-aminopropanol-1[ $\beta$ -amino- $\alpha$ -m-hydroxyphenyl-n-propyl alcohol]. I. G. FARBENIND. A.-G. (B.P. 376,457, 12.6.31. Ger., 13.6.30). — m-Hydroxyphenylacetylcarbinol, ArCH(OH) COMe, or an ether is converted via the oxime into the aminoalcohol. m-Benzyloxybenzaldehyde bisulphite compound is treated with KCN and the cyanohydrin reacts with MeMgI to give the keto-alcohol; the oxime is reduced and debenzylated with H<sub>2</sub> and Pd in alcoholic HCl, yielding  $\beta$ -amino- $\alpha$ -m-hydroxyphenyln-propyl alcohol hydrochloride, m.p. 180° (decomp.). C. H.

[Manufacture of bromo- and chloro-]derivatives of p-hydroxydiphenyl. E. F. GRETHER, Assr. to Dow CHEM. Co. (U.S.P. 1,832,484, 17.11.31. Appl., 11.10.28). —p-Hydroxydiphenyl is treated with NaOCl or NaOBr. The 3-Cl- (m.p. 77°; Me ether, m.p. 91—92°), 3-Br- (Me ether, m.p. 61—63°), and 3-NH<sub>2</sub>- (m.p. 79°) derivatives are described. C. H.

Manufacture of nitroaminodiphenyl sulphides. Soc. ANON. DES MAT. COL. ET PROD. CHIM. DE ST. DENIS, and R. LANTZ (B.P. 376,961, 25.7.31. Fr., 8.8.30).— An o- or p-chloronitrobenzene is caused to react with a sol. sulphide (2—5 mols.) and the resulting aminothiophenol, with or without isolation, is treated with a further mol. or an o- or p-chloronitrobenzene. The following nitroaminodiphenyl sulphides are described : 4:4'- (m.p.  $143^{\circ}$ ), 2:4'- (m.p.  $103^{\circ}$ ), 4:2'- (m.p.  $87\cdot4^{\circ}$ ), 2:2'-(m.p.  $85\cdot4^{\circ}$ ); 2:4':4-, 4:4':2-, 2:2':4-, and 4:4':2'-sulphonic acids; 4:4':2- [m.p. 215— $216^{\circ}$ (decomp.)] and 4:2':2- [m.p.  $243^{\circ}$  (decomp.)] carboxylic acids; 4-chloro -2:4'- (m.p.  $133^{\circ}$ ) and 4:4'-dichloro 2:2'- (m.p.  $139^{\circ}$ ); 2:4-dinitro-4'-amino- (m.p.  $168\cdot5^{\circ}$ ).

Manufacture of terpene [camphene and pinene]. SCHERING-KAHLBAUM A.-G. (B.P. 375,395, 25.3.31. Ger., 15.4.30).—Inorg. or inorg.-org. acids which do not give additive products with pinenes catalyse the direct conversion of nopinene into pinene and of pinene into camphene. Examples are  $HVO_3$  at 100°,  $Sb_2O_5$  at 120°, and phosphomolybdic acid at 20–30°. C. H.

Isolation or purification of menthols. HowARDS & SONS, LTD., J. W. BLAGDEN, and W. E. HUGGETT (B.P. 376,356, 10.4.31).—A mixture of menthols, obtained, e.g., by hydrogenation of thymol, menthones, menthenols, menthenones, etc., is treated with  $H_3PO_4$  and the cryst. products are separated by crystallisation, e.g., from light petroleum. C. H.

Manufacture of substituted aromatic ketones. Soc. CHEM. IND. IN BASLE (B.P. 377,464, 24.8.31. Switz., 28.8.30).-tert.-Aminoalkyl ethers of hydroxyaryl ketones are synthesised, e.g., by treatment of the ketone with tert.-aminoalkyl chlorides and alkali. Examples are : (p-β-diethylaminoethoxy)acetophenone, b.p. 167-168°/5 mm.; 4-methoxy-2-β-diethylaminoethoxyacetophenone, b.p. 186—187°/5 mm.; 4-methoxy-2- $\beta$ -diethylaminoethoxyphenyl benzyl ketone, b.p. 210— 211°/0·1 mm.; p- $\beta$ -dicyclohexylaminoethoxybenzo-phenone, m.p. 157—158°; 4-methoxy-2- $\beta$ -benzylmethylaminoethoxyacetophenone, b.p. 197.5-198°/0.12 mm., from the 2-β-bromoethoxy-compound, m.p. 65-68°, and CH<sub>2</sub>Ph·NHMe; 4-methoxy-2- $\gamma$ -diethylamino-iso-butoxyacetophenone, b.p. 174—175°/0·5 mm.; m- $\beta$ -diethylaminoethoxy- (b.p. 166—167°/6 mm.); 5-nitro-4-methoxy-2-β-diethylaminoethoxy- [m.p. 124-125°; amine, m.p. 69-72° (Ac derivative, m.p. 121-122°)], 2-β-diethylaminoethoxy-4-n-butoxy- (b.p. 166-167°/ 0.07 mm.), 4-benzyloxy-2-\beta-diethylaminoethoxy- (b.p. 206.5-207°/0.2 mm.), and 4-methoxy-2-β-di-n-butylaminoethoxy- (b.p. 179-180°/0.24 mm.) -acetophenones; 1-B-diethylaminoethoxy-2-naphthyl methyl ketone, b.p. 151—152°/0·15 mm. ; 4-methoxy-2- $\beta$ -diethylamino-ethoxybutyrophenone, b.p. 196—199°/4 mm. ; p- $\beta$ diethylaminoethoxyphenyl (m.p. 36-37°, b.p. 207-208°/3 mm.), and 4-benzyloxy-2-β-diethylaminoethoxyphenyl (picrate, m.p. 158-159°) benzyl ketones. The

following intermediates are also described : 4-methoxy-2β-hydroxy- (m.p. 65-67°), 2-hydroxy-4-n-butoxy- m.p. 40-41°, b.p. 154-155°/6 mm.), and 2-hydroxy-4benzyloxy- (m.p. 105-106°) -acetophenones; 2-hydroxy-4-methoxybutyrophenone, m.p. 32-33°, b.p. 143-145°/5 mm.; 2-hydroxy-4-benzyloxyphenyl benzyl ketone, m.p. 107-108°; and NEt2 CH2 CH2 CHMeCl, b.p. 67-70°/15 mm. (from the sec.-alcohol, b.p. 85-87°/22 mm.). C. H.

Production of lactones [glucono-y-lactone]. R. PASTERNACK and G. O. CRAGWALL, Assrs. to C. PFIZER & Co. (U.S.P. 1,830,618, 3.11.31. Appl., 12.4.30).—Aq. gluconic acid in equilibrium with its lactone is boiled with 4 pts. of an alcohol, e.g., Bu<sup>a</sup>OH, until clear, the solution is conc., and lactone separated as crystals.

C. H.

C. H.

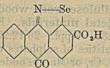
Manufacture of 1-amino-2-alkoxynaphthalenes. Soc. CHEM. IND. IN BASLE (B.P. 376,667, 3.2.32. Switz., 9.2.31).-A 1-halogeno-2-alkoxynaphthalene is heated with  $NH_3$ , preferably in presence of Cu or  $ZnCl_2$ . 1-Amino-2-methoxy- and -2-ethoxy-naphthalenes, m.p. 54° and 50-51°, respectively, are thus prepared.

Purification of crude anthracene. L. C. DANIELS, Assr. to SELDEN Co. (U.S.P. 1,831,059, 10.11.31. Appl., 27.7.28) .- Phenanthrene is dissolved out of crude anthracene by means of o-C6H4Cl2 or other chlorinated aromatic bydrocarbon at 15°. At 80° some carbazole C. H. is also removed.

Production of aminoanthraquinone derivatives. IMPERIAL CHEM. INDUSTRIES, LTD., R. J. LOVELUCK, and J. THOMAS (B.P. 375,848, 30.3.31).-A 1-benzamido-4-, 5-, or 8-arylsulphonamidoanthraquinone is treated with conc. H<sub>2</sub>SO<sub>4</sub> or weak oleum at about 28°, whereby the C. H. arylsulphonyl group only is removed.

Manufacture of carboxylic acid amides. IMPERIAL CHEM. INDUSTRIES, LTD., and R. F. GOLDSTEIN (B.P. 375,883, 7.4.31).—A carboxylic acid is heated with a primary or sec. amine and a dehydrating agent (PCl<sub>3</sub>) in presence of AlCl<sub>a</sub> and, if desired, an inert medium (PhMe, PhCl). Examples are anthraquinone-2-carboxylic and salicylic anilides. С. Н.

[Manufacture of] anthraquinone derivatives [containing selenium]. E. I. DU PONT DE NEMOURS & Co. (B.P. 375,437, 30.3.31. U.S., 29.4.30).-Selenazolanthrone-3-carboxylic acid (annexed formula), preferably



N-Se as chloride, is condensed with an aminoanthraquinone, e.g., with 1-CO,H or 2-aminoanthraquinone (yellow vat dye), 1:5-diamino- (yellow), 1:4-diamino- (yellow-brown), 1amino-5-benzamido-(red-yellow),

or 6-chloro-1-amino- (green-yellow) -anthraquinones, or with 1-aminoanthraquinone-6-carboxylic acid and there-C. H. after with further amines.

Preparation of stable diazo compounds. IMPERIAL CHEM. INDUSTRIES, LTD., A. DAVIDSON, and W. G. REID (B.P. 376,307, 8.4.31).-A Zn salt is added to a diazo solution from a 4-p-aminoarylamino-1-alkyl- (or dialkyl-) aminoanthraquinone. The stable double salt may be dried at 40-45° or mixed with Na<sub>2</sub>SO<sub>4</sub> etc. Examples

are: 4-p-aminoanilino-1-methylamino- or -1-dimethylamino-anthraquinone and the 4-(4'-amino-2' : 5'-dimethoxyanilino)-, 2-bromo-4-p-aminoanilino-, and 4-(3'chloro-4'-aminoanilino)-1-methylaminoanthraquinones. C. H.

Controlling reaction.-See I. Hydrogenation catalysts .- See X. Lactic acid etc.-See XVIII.

# IV.-DYESTUFFS.

Leishmann's dye. I. V. KULIKOV (Khim. Farm. Prom., 1932, No. 2-3, 64-68).-H<sub>2</sub>O (2800 c.c.) and methylene-blue (150 g.) are treated at 60-65° with NaHCO<sub>3</sub> (75 g.) in H<sub>2</sub>O (200 c.c.) and stirred for 30 min.; H<sub>2</sub>O (2800 c.c.) and Na eosinate (135 g.) are added, and the mixture is again stirred for 30 min. The product (200-210 g.) is collected by suction.

CH. ABS.

#### Gambier catechins.-See XV.

See also A., Oct., 1028, Diphenyl-green dyes. 1035, Complex salts of hydroxyquinones. 1038, Dye from 10-hydroxy-9-phenyl-y-anthranol. 1043, Dye from phenanthraquinone.

#### PATENTS.

Manufacture of azo dyes insoluble in water [green pigments and ice colours]. I. G. FARBENIND. A.-G. (B.P. 377,024, 6.10.31. Ger., 6.10.30).-The monodiazo compound from 7:7'-diaminothioindigo is coupled in substance or on the fibre with an acetoacetic or aroylacetic arylamide free from solubilising groups, particularly acetoacetyl-p-aminophenylbenziminazole or bisacetoacetyltolidine. Other coupling components are, e.g., benzoylacetic o-chloroanilide, p-toluoylacetic anaphthylamide, anisoylacetic anilide, acetoacetic or terephthaloylbisacetic 5-chloro-o-anisidide. C.H.

Manufacture of disazo dyes. I. G. FARBENIND. A.-G. (B.P. 376,438, 28.5.31. Ger., 28.5.30).-Level reddish-blue to greenish-blue shades on viscose silk are obtained with dyes of the type: p-phenylenediaminesulphonic acid  $\rightarrow$  1 mol. of  $\gamma$ -acid or an alkyl- or aryl-y-acid and 1 mol. of the same or another alkylarylamine or diarylamine of the C6H6 or C10H8 series, both couplings being in acid medium. Examples are: 4-nitroaniline-2-sulphonic . acid  $\rightarrow \gamma$ -acid, reduced,  $\rightarrow \gamma$ -acid or methyl- $\gamma$ -acid; 6-acetamido-m-anisidineacid  $\rightarrow$  phenyl- $\gamma$ -acid, hydrolysed,  $\rightarrow$ 4-sulphonic methyl- \beta-naphthylamine-7-sulphonic acid ; 6-acetamido*m*-anisidine-4-sulphonic acid  $\rightarrow \gamma$ -acid, hydrolysed,  $\rightarrow$  methyl- $\gamma$ -acid or *n*-butyl- $\beta$ -naphthylamine-7-sulphonic acid or NMe,Ph. C. H.

Production of polyazo [direct] colouring matters. W. M. RALPH, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,832,322, 17.11.31. Appl., 3.1.27).-The excess of diazobenzene remaining after coupling benzidine  $\rightarrow$  H-acid (1 mol.) with diazotised NH<sub>2</sub>Ph is removed by coupling with 4'-hydroxydiphenylazo-Hacid (made by boiling benzidine  $\rightarrow$  H-acid); the product is then coupled as usual with m-phenylene- or -tolylene-diamine, and the green-black dye is obtained in improved yield free from dyes of the chrysoidine type. The admixed dark green disazo dye does not interfere

A. J. H.

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with the dyeing, is readily discharged, and scarcely stains acetate silk. C. H.

Anthraquinone derivatives containing Se.—See III.

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

Comparison of the properties of true and wild silk. R. INOUYE and K. KITAZAWA (Bull. Sericult., Japan, 1932, 5, 7–8).—The % sol. N removed from silks by treatment with boiling H<sub>2</sub>O, NaOH, H<sub>2</sub>SO<sub>4</sub>, aq. Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>, and aq. Na<sub>2</sub>SiO<sub>3</sub>, and of the material sol. in H<sub>2</sub>O, increases in the order true silk, Yamamai, tussah, and *Attacus cynthia*. After treatment with H<sub>2</sub>O, H<sub>2</sub>SO<sub>4</sub>, HCl, AcOH, NaOH, aq. NH<sub>3</sub>, CH<sub>2</sub>O, Et<sub>2</sub>O, etc., the Yamamai variety has the highest tensile strength and extensibility when wet, and the wild silks are more attacked than the true, although the former are more insol. in acids and alkalis than the latter. Conc. aq. NH<sub>3</sub> has a very pronounced effect on the strength of true silk. B. P. R.

Physical properties of raw silk. I. Density and modulus of elasticity. II. Thermal expansion coefficient. J. KUBOTA (Bull. Sericult., Japan, 1932, 5, 9–10).—The modulus of elasticity of raw silk is 115,000,000 g./sq. cm. and its coeff. of linear expansion -0.0000098. Hence, silk contracts if the temp. rises and elongates if it falls, and thus behaves like caoutchouc. B. P. R.

Composition of the fibre and waste of Agave Leuchuguilla. R. A. GREENE (Bot. Gaz., 1932, 93, 484-491).—Analytical data are similar to those for *A. americana*. The possible uses of the material as a fertiliser, or for cattle food, paper pulp, or alcohol production are discussed. A. G. P.

Straw. H. BACH (Chem.-Ztg., 1932, 56, 701-703).-The objectionable nature of the effluent produced during the manufacture of strawboard from straw by treatment with CaO is due to the S-compound content of the straw. The S may be determined by heating the straw with reduced Fe, treating the residue with HCl, and absorbing the evolved H<sub>2</sub>S in aq. NaOH; a sample of straw contained 0.25% S, and one of strawboard made from it only about 0.1%. The  $p_{\rm H}$  of a straw-H<sub>2</sub>O mixture falls within 2-3 days from 6.4 to 5.2, and thereafter remains nearly const., although increasing quantities of  $H_2S$  are evolved. If the  $p_H$  is maintained const., by addition of NaOH, at 7.0-7.6 for 2-3 weeks, much more S, org. and inorg. N, and other substances are removed from the straw, which is unchanged in appearance and may be used satisfactorily for strawboard manufacture; the effluent from the process is much less objectionable than when raw straw is used. The liquor from the NaOH treatment should be of val. as a fertiliser; in commercial practice Ca(OH)<sub>2</sub> could be used. H. F. G.

Manufacture of ethyl- and benzyl-celluloses. A. G. JAEGER (Kunstseide, 1932, 14, 332).—For ethylcellulose, cotton linters are treated with 50% aq. NaOH, pressed or hydro-extracted until the alkali-cellulose contains 30—35% H<sub>2</sub>O, distilled with C<sub>6</sub>H<sub>6</sub> to reduce the H<sub>2</sub>O content to  $1\cdot 3\%$ , and treated for 8—10 hr. at 180-200° with 3 pts. of EtCl; excess EtCl is then removed and the product freed from HCl, by washing with cold  $H_2O$ , and from insol. org. impurities, by dissolution in EtOH,  $C_6H_6$ , or COMe<sub>2</sub>, filtration, and pptn. by dilution with  $H_2O$ . Benzylcellulose is obtained by treating alkali-cellulose with BzCl; it is highly resistant to 20% NaOH,  $H_2SO_4$  (d 1·4), and heat, contains normally 0.5--1.0%,  $H_2O$ , softens at 180°, and may be moulded at 120--160° under pressure.

Sulphite-pulp residue as fuel.-See II.

See also A., Oct., 992,  $[H_2O]$  absorption [by fibre boards]. 1022, Acetolysis of cellulose. Action of FSO<sub>3</sub>H on cellulose.

#### PATENTS.

**Comminution of fibres.** P. SCHIDROWITZ, R. M. UNGAR, and M. W. PHILPOTT (B.P. 379,099, 28.8.31).— To reduce the tendency to slip on the grinding rolls, the fibres are pretreated with a natural or artificial aq. dispersion, vulcanised or unvulcanised, of rubber, guttapercha, or balata, or with a solution or dispersion of gelatin, resin, cellulose ester, glucose, gum arabic, or dextrin, and dried at 150°. F. R. E.

Opening up [pulping of] resiniferous wood. H. HEIMANN, E. OPFERMANN, I. PETERSEN, K. BITTERFELD, and A. BAYERL, ASSTS. to I. G. FARBENIND. A.-G. (U.S.P. 1,840,162, 5.1.32. Appl., 26.3.31. Ger., 12.4.30).— The raw material, e.g., pine shavings, is impregnated with dil. (30%) HNO<sub>3</sub> under reduced pressure, kept at  $30-50^{\circ}/1$  atm. for 2 hr., and, after removal of excess acid, is treated with H<sub>2</sub>O or highly dil. HNO<sub>3</sub> for 2—4 hr. at 60—80°. The raw pulp is then boiled for 2 hr. with 2—3% aq. NaOH, bleached with 4—5% of Cl<sub>2</sub>, and treated with cold 8% aq. NaOH for 1—2 hr. After further bleaching with 1—2% of Cl<sub>2</sub> in a slightly alkaline medium a pulp is obtained containing 95% of  $\alpha$ -cellulose and  $\geq 0.3\%$  of fats and resins. D. J. N.

Treatment [digestion] of cellulose-containing materials. BRIT. CELANESE, LTD. (B.P. 378,722, 16.11.31. U.S., 24.11.30).—The raw material (wood chips, straw, etc.) is mixed with digestion liquor [NaOH,  $Ca(HSO_3)_2$ , or  $Na_2S + NaOH$ ] and pumped continuously through a pipe or coil maintained at the required cooking temp. and of such a length that the material during its passage is cooked to the desired degree. Apparatus is described. D. J. N.

Production of high-grade cellulose from wood, straw, grasses, and other vegetal materials. E. SCHMIDT (U.S.P. 1,840,413, 12.1.32. Appl., 26.12.29. Czechoslov., 11.7.28).—Small quantities of sulphates of Fe, Ag, Cu, Al, Zn, Sn, Pb, Co, Ni, Cr, or Mn are added to the normal sulphite lye used in boiling the materials. F. R. E.

Refined wood pulp of low pentosan content. G. A. RICHTER, ASST. to BROWN CO. (U.S.P. 1,838,326, 29.12.31. Appl., 14.3.30).—Wood pulp containing > 96% of  $\alpha$ -cellulose and < 1% of pentosans is obtained by cooking first with aq. H<sub>2</sub>SO<sub>4</sub> or aq. H<sub>2</sub>SO<sub>3</sub>, with or without acid salts, and then with aq. NaOH and Na<sub>2</sub>S. The tear strength is > that of kraft pulp and the cuprammonium viscosity very low. A.G.

Preparation of high a-cellulosic pulp. E. R. DARLING, ASST. to CORNSTALK PRODUCTS CO., INC. (U.S.P. 1,838,367, 29.12.31. Appl., 31.3.30).-In preparing pulp of 95-96% a-cellulose content from plants of annual growth, e.g., cornstalks, the bleached pulp is boiled with  $H_2O$  under pressure (5 lb. > atm.) before treatment with aq. NaOH. A. G.

Multiple-step process of fibre liberation. G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,841,551, 19.1.32. Appl., 6.5.30) .- Wood chips are submitted to successive cooks with aq. H<sub>2</sub>SO<sub>3</sub>, NaHSO<sub>3</sub>, and NaOH, Na<sub>2</sub>S, etc. under such conditions that complete liberation of the fibre occurs only during the final alkaline cook. B. P. R.

Utilisation [as fuel] of waste arising from depulping of fibrous foliate plants. F. KRUPP GRUSON-WERK A.-G. (B.P. 378,718, 10.11.31. Ger., 27.4.31) .--The waste pulp, e.g., from sisal leaves, is passed through successive pairs of fluted crushing rollers (to remove H<sub>2</sub>O) and can then be used as fuel, optionally after drying. D. J. N.

Manufacture of cellulose esters. A. G. BLOXAM. From Soc. CHEM. IND. IN BASLE (B.P. 379,658, 23.6.31). -NaOAc may satisfactorily replace KOAc in the process of B.P. 353,978 (B., 1931, 921) if the reaction temp. is raised by, e.g., substituting xylene for  $C_6H_6$ D. J. N. in the acetylating bath.

Production of cellulose acetate free from haze. C. J. MALM, Assr. to EASTMAN KODAK CO. (U.S.P. 1,840,404, 12.1.32. Appl., 10.8.29) .- The parts of the reaction vessels and the implements which come in contact with the pretreatment, acetylation, and hydrolysis liquids, which may if desired contain acid catalysts, are composed of an alloy containing, approx., 18% Cr, F. R. E. 8% Ni, and 74% Fe.

Cellulosic composition containing (A) butyl propionate, (B) cyclohexanol, (c) nitro-p-xylene. S. J. CARROLL, ASST. to EASTMAN KODAK CO. (U.S.P. 1,841,311-3, 12.1.32. Appl., 23.9.29).-(A) 10-30 pts. of Bu propionate, (B) 10-30 pts. of cyclohexanol, or (c) 10-50 pts. of nitro-p-xylene are used as plasticisers F. R. E. for each 100 pts. of cellulose acetate.

Making soda-cellulose for conversion into viscose. G. A. RICHTER, Assr. to BROWN CO. (U.S.P. 1,839,774, 5.1.32. Appl., 27.7.29).—High-a-cellulose pulp prepared from kraft or other soda-pulp gives an alkali-cellulose which shows substantially no loss of a-cellulose on ageing (even when ageing is accelerated by raising the temp. to 25°), and is therefore more suitable for viscose manufacture than refined sulphitepulp. D. J. N.

Rayon manufacture. G. A. RICHTER, Assr. to BROWN CO. (U.S.P. 1,839,773, 5.1.32. Appl., 21.1.27).-High-a-cellulose pulp of high (cupremmonium) viscosity (> 10) is used for viscose manufacture, the reduction of viscosity necessary for the prep. of a spinnable viscose solution being effected by suitably increasing the time and/or temp. of ageing of the alkali-cellulose. The resulting products are superior as regards strength, stretch, and colour to those produced from sulphite-pulp the initial viscosity of which has been reduced to 3-6 by, e.g., over-cooking. D. J. N.

Treatment of viscose solutions. F. W. SPERR, JUN., Assr. to KOPPERS Co. (U.S.P. 1,841,420, 19.1.32. Appl., 8.1.27).-Liberation of H<sub>o</sub>S during the spinning of viscose into an acid bath is prevented by previously aërating the solution for several hr. after the addition of a catalyst (quinol, pyrocatechol, etc.) which promotes the conversion of the S compounds into thiosulphate. B. P. R.

Manufacture of shaped artificial sponge masses from viscose. I. G. FARBENIND. A.-G. (B.P. 379,235, 7.3.32. Ger., 7.3.31).-Viscose containing salts as pore-forming agents is pressed into a tubular mould, coagulated by heat, freed from salts, and cut into pieces perpendicularly to the long axis of the mass.

F. R. E.

"Production of artificial materials by wet-spinning processes. BRIT. CELANESE, LTD., W. A. DICKIE, and P. F. C. Sowter (B.P. 378,858, 12.5.31. Addn. to B.P. 340,324 and 340,436; B., 1931, 1043).-The solvents or swelling agents used in the coagulating bath, after-treatment bath, or spinning solution consist of lower aliphatic acid esters of di- or poly-hydric alcohols, other than mono- or di-acetin, or their partial ethers, e.g., glycol and methylglycol mono-acetates. F. R. E.

Production of a loosely distended mass of fibrous material in the form of a layer. F. B. DEHN. From CELLUFOAM CORP. (B.P. 379,119, 28.9.31).-Fibrous cellulosic material, mixed, if desired, with long hairlike fibres, is beaten with H<sub>2</sub>O together with a bubbleforming ingredient, e.g., saponin. The distended mass is then formed into a uniform layer, subjected to suction at the surface to form a skin, and finally dried by heat without application of pressure. Apparatus is claimed. F. R. E.

Manufacture of tracing paper. F. W. J. ALLOTT (B.P. 379,575, 3.12.31).-By using a mixture of petroleum and resins (dammar, copal), optionally with addition of traces of vegetable oils (castor, poppyseed) as the "transparentising" agent, a paper is obtained which does not discolour or become brittle with age.

D. J. N.

Manufacture of tracing paper and transparent paper. J. KNAGGS, J. C. MARSDEN, and PORTALS, LTD. (B.P. 380,008, 30.3.32) .- Paper made from cotton or flax is treated with an emulsion of oil, fat, or wax with a protein (gelatin, albumin, etc.) and a hygroscopic substance (glycerin, MgCl<sub>2</sub>), and the protein is rendered insol. by subsequent treatment with CH<sub>2</sub>O. B. P. R.

Manufacture of sized paper. G. T. LANE, Assr. to EASTMAN KODAK CO. (U.S.P. 1,840,399, 12.1.32. Appl., 1.3.29).-A soap of a saturated fatty acid above  $C_9$  and having an I val. < 40 is added to paper pulp of  $\alpha$ -cellulose content > 90% during beating, and is pptd.  $\alpha$ -cellulose content  $> 00_{10}$  damped and  $\alpha$ , or CuSO<sub>4</sub>. thereon by means of alum, MgSO<sub>4</sub>, or CuSO<sub>4</sub>. F. R. E.

[Sized] paper manufacture. E. SUTERMEISTER and J. A. WARREN, Assrs. to S. D. WARREN Co. (U.S.P. 1,838,969, 29.12.31. Appl., 7.4.28).-The objectionable properties of CaCO3 and other alkaline compounds of alkaline-earth metals when used as fillers for sized paper are overcome by adding an emulsion of paraffin wax in aq. Na resinate and silicate. A. G.

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**Production of papers for security purposes.** less fast to light when dyed by this method. Cotton V. BAUSCH, SEN., and T. and F. BAUSCH (F. SCHOELLER & BAUSCH) (B.P. 378,694, 7.10.31. Ger., 7.10.30) .---Paper (of  $p_{\rm H}$  3-5) containing acid-sensitive reagents, e.g., Pb<sub>2</sub>Fe(CN)<sub>6</sub> and a Fe<sup>"</sup> salt, is impregnated with a solution of a carboxylic acid derivative of C<sub>6</sub>H<sub>6</sub> or C10Hg and/or a derivative or compound thereof (e.g., a 1:1 mixture of pyrogallolcarboxylic acid and pentagalloylglucose) the  $p_{\rm H}$  of which has been adjusted to 3-3.5 by the addition of alkali. Such a paper reacts to acids and oxidising agents (including dil. KMnO4) giving stains difficult to remove with reducing agents.

D. J. N.

Mineral-coated paper. H. E. Potts. From CHAMPION COATED PAPER CO. (B.P. 379,833, 10.8.31).-- An aq. composition containing clay, satin-white, etc. and an adhesive (casein, glue, etc.) is applied to the moving paper sheet and the excess is removed by suitable means (described). B. P. R.

Silicating [of fibre board]. O. B. ANDREWS (U.S.P. 1,841,775, 19.1.32. Appl., 15.8.29).-Aq. Na2SiO3 is applied to the surface of the boards, allowed to penetrate slightly into the material, and the final drying performed at 21°. B. P. R.

Method and apparatus for applying batching solution to fibre such as jute. W. J. NICOLL (B.P. 380,291, 12.11.31).

Means for supporting the funnels employed in machines for spinning artificial silk. KIRKLEES, LTD., A. BENNETT, and W. TONG (B.P. 380,569, 17.8.31).

Device for washing and after-treating bobbins of artificial silk by the stacking system. VEREIN. GLANZSTOFF-FABR. A.-G. (B.P. 380,395, 22.4.32. Ger., 27.4.31).

Printing and waxing of paper. E. C. FRANCOISE (B.P. 380,244, 2.9.31).

Furfuraldehyde from wood pulp .-- See III. Coatings for films.-See XIII. Rubber-fibre products. -See XIV. Lactic acid etc.-See XVIII. Surgical sewing materials.-See XX. [Light-sensitive] regenerated cellulose.-See XXI.

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

Mordanting of vegetable fibres, viscose, and cuprammonium silks for dyeing with acid and chrome colours. J. P. SISLEY (Rev. gén. Mat. Col., 1932, 36, 334-337).—The fibre is mordanted with a condensation product prepared from a primary or sec. amine (o-toluidine, m-xylidine, o-anisidine, cresidine, NHEtPh, thiocarbamide) and an aliphatic aldehyde (CH<sub>2</sub>O, MeCHO, crotonaldehyde) in presence of HCl. In particular, anhydroformaldehyde-o-toluidine gives insol. lakes with acid and chrome dyes, and with sulphanilic acid, H-acid, J-acid, etc. It has direct affinity for cotton and viscose from a 0.50-0.70 vol.-% bath at 15-60°. The mordanted fibre is dyed below 80° with acid or chrome colours from a bath acidified with AcOH or HCO,H. The dyeings are faster to washing and acid than corresponding dyeings on unmordanted silk, but certain colours (Chrome-fast-yellow G, Bengal-pink, Violamine R, Carmine-blue A, Milling-orange N) become

mordanted with the new mordant takes up H-acid, Y-acid, J-acid, sulphonated Naphthol AS, etc., and may then be developed with a diazo compound; sulphonated diazo compounds may similarly be fixed on the fibre and afterwards developed with Naphthols, but the shades are weak. The mordant may be used on wool, silk, leather, or furs, but not on cellulose acetate.

C. H.

Dyeing of wool and cotton by means of basic substantive dyes. A. E. PORAI-KOCHITZ, D. M. ZELETSEV, and R. M. RATMAN (Izvest. Textil. Prom. Torgov., 1931, 10, No. 3-4, 82-84).-In dyeing wool with  $[(\mathrm{NH}_2)_2\mathrm{C}_6\mathrm{H}_2\mathrm{Me}\cdot\mathrm{N}_2\cdot\mathrm{C}_6\mathrm{H}_3\cdot\mathrm{OMe}]_2,2\mathrm{HCl}$  and  $[(\mathrm{NH}_2)_2\mathrm{C}_6\mathrm{H}_3\cdot\mathrm{N}_2\cdot\mathrm{CH}\cdot\mathrm{CH}]_2\mathrm{CO},4\mathrm{HCl}$  the base is selectively fixed, whilst with cotton the salt is fixed.

CH. ABS.

Methods of studying the desizing of fabrics. P. M. POGOYEV and E. A. RAKLIN (Izvest. Textil. Prom. Torgov., 1931, 10, No. 3-4, 80-81).-Determination of loss in wt. is inaccurate ; preferably starch is determined before and after desizing. A method described employs enzymic hydrolysis followed by titration with I. CH. ABS.

Detergent.-See XII.

#### PATENTS.

[Wetting agents for] treating natural and artificial cellulosic fibres with alkali. CHEM. FABR. VORM. SANDOZ (B.P. 378,194, 29.7.31. Ger., 30.7.30).-The hydrogenated aromatic compounds described in B.P. 279,784 (B., 1928, 228) are replaced by alicyclic carboxylic acids such as naphthenic acids and the resin acids present in natural resins. A. J. H.

[Sulphur] dyeing process. IMPERIAL CHEM. INDUSTRIES, LTD., and M. WYLER (B.P. 379,742, 4.6.31). -The dull red-brown shade obtained by dveing cotton with the acenaphthene S dye described in B.P. 124,589 (B., 1919, 318A) is rendered faster and changed to a clear red-orange shade by after-treatment at 100° for 20 min. in a bath containing, per litre, 4 c.c. of NaOH (d 1.38), 1.5 g. of Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>, and 10 g. of an alkylating or aralkylating agent. A. J. H.

Manufacture of azo dyes on the fibre [ice colours]. W. W. GROVES. From Soc. CHEM. IND. IN BASLE (B.P. 377,271, 21.4.31).-A 2: 3-hydroxynaphthoic arylamide is coupled on the fibre with a diazotised mono(aryloxyacetyl)-p-phenylenediamine symmetrically substituted in the diamine nucleus by 2 equal alkyl or alkoxyl groups. The dyeings show improved fastness to boiling Na<sub>2</sub>CO<sub>3</sub> and to bleach. Examples are: 4-o-toloxyacetamido-2: 5-dimethoxyaniline  $\rightarrow m$ -nitroanilide (blueviolet); 5-phenoxyacetamido-p-2-xylidine  $\rightarrow p$ -anisidide (blue-red); 5-o-toloxyacetamido-p-xylidine  $\rightarrow$  p-chloroanilide (blue-red). C. H.

[Products for] improving the action of alkaline mercerising liquors on cellulosic fibres. I. G. FARBENIND. A.-G. (B.P. 378,223, 29.8.31. Ger., 24.9.30). -Ethers, particularly the Pr. Bu<sup> $\alpha$ </sup>, Bu<sup> $\beta$ </sup>, amyl, and hexyl ethers, of polyhydric alcohols (especially glycerin) are efficient wetting agents for adding to mercerising A.J.H. liquors.

Dyeing [in a sulphide bath with azo dyes having a di- or tri-sulphide group]. Soc. ANON. DES MAT. COL. ET PROD. CHIM. DE ST.-DENIS, and R. LANTZ (B.P. 377,348, 20.5.31. Fr., 21.6.30).-Azo dyes containing a di- or tri-sulphide grouping dye fibres of cellulose or its esters or ethers in full shades from a sulphide bath below 65°, preferably at 15°. Examples are: 2:2'dinitro-4:4'-diaminodiphenyl disulphide  $\rightarrow \beta$ -naphthol (2 mols.; orange) or *m*-phenylenediamine (2 mols.; brown-yellow); 4:4'-diaminodiphenyl disulphide  $\rightarrow$ its bis-2: 3-hydroxynaphthoyl derivative (2 mols.; bluered) or β-naphthol (2 mols.; chromed, red); 4-nitroo-aminophenol  $\rightarrow$  6:6'-dihydroxy-2:2'-dinaphthyl disulphide (coppered, violet); 4:4'-diaminodiphenylyl disulphide  $\rightarrow \beta$ -C<sub>10</sub>H<sub>7</sub>·NH<sub>2</sub> (2 mols.; brown; developed with  $\beta$ -naphthol, black). C. H.

Finishing wet artificial silk in the form of skeins or cakes with oil emulsions. N. V. HOLLANDSOHE KUNSTZIJDE IND., T. DOKKUM, and P. H. HERMANS (B.P. 379,196, 6.1.32. Holl., 6.1.31).—Viscose silk is oiled during the washing and bleaching stages in the manufacturing process by treatment in a bath of an emulsion containing  $\ll 4\%$  of oil, and dilution of the bath is prevented by centrifuging it and thereby removing an amount of H<sub>2</sub>O equal to that introduced by the wet silk ; H<sub>2</sub>O-sol. substances removed are again added to the residual emulsion. A. J. H.

Delustring of (A) real and (B) artificial silks. W. H. LORMER (U.S.P. 1,839,978—9, 5.1.32. Appl., [A] 21.1.29, [B] 28.8.30).—Delustring is effected by immersing the material in a liquor at 21—100° containing up to 5% (calc. on the wt. of silk) of a 1:1 mixture of alum and BaCl<sub>2</sub>. A. J. H.

[Créping of] textile yarns and fabrics. BRIT. CELANESE, LTD., H. DREYFUS, W. A. DICKIE, and R. W. MONCRIEFF (B.P. 378,910 and 379,285, 15.5.31).-(A) Threads composed of org. derivatives of cellulose are impregnated with a suitable reagent [aq. citric acid, Na citrate, Pb(OAc)<sub>2</sub>, etc.] before applying at least part of the high degree of twist, and, after final twisting and incorporating into fabric, are treated with another reagent [aq. Ba(OAc)2, Ba(OH)2] so as to form a solid or semi-solid material (Ba citrate, PbSO<sub>4</sub>, etc.) on or in the crêpe threads; the fabric is subsequently scoured with hot  $H_2O$  or soap solution, dyed, and finished. (B) The impregnating liquid consists of an aq. solution containing anions having a high coagulating or pptg. action on gels or solutions of the cellulose derivative, e.g., org. di- or poly-basic acids, hydroxy-mono-, -di-, or -poly-basic acids or their alkali salts (citric, lactic, tartaric, etc.), or ferrocyanides, thiosulphates, sulphates, F. R. E. phosphates, etc.

Increasing the elasticity and strength of artificial [viscose] silk and the like. E. WURTZ (B.P. 379,604, 11.1.22).—The viscose solution is spun into a coagulating bath (A) and drawn thence through a fixing bath (B) such that the mineral acid content  $(30-35\% H_2SO_4)$  and temp. ( $> 15^\circ$ ) of bath B are at least twice and about one half that of A, respectively; bath B is capable of plasticising the silk. A. J. H.

Incorporation of metal compounds in textile materials. BRIT. BEMBERG, LTD. (B.P. 378,760, 31.12.31. Ger., 31.12.30).—Textile material is impregnated with aq. solutions of complex metal oxide– $NH_3$ compounds combined with amphoteric metal oxides (e.g., Zn, Al, Ni, and Cu) and then dried, whereby the  $NH_3$  is removed and the finely-divided insol. metal hydroxide is left within the fibres. A. J. H.

Machines for dyeing and like treatment of textile fibres [in hank form etc.]. C. CALLEBAUT and J. DE BLICQUY (B.P. 380,637, 22.10.31).

Machines for dyeing yarn. H. GOUGH, and BRIT. COTTON & WOOL DYERS' ASSOC., LTD. (B.P. 380,192, 12.12.31).

Wetting etc. agents.—See III. Lye for cotton mercerising.—See VII. Cleaning paste.—See XII. Impregnating compound [for felt etc.].—See XIV.

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

Economics of the Petersen tower system [of sulphuric acid manufacture]. F. SCHUCH (Chem.-Ztg., 1932, 56, 664). H. PETERSEN (*Ibid.*, 664—665).—SCHUCH criticises the Petersen tower process on the grounds that it involves either excess in pumping costs or N losses according to the concn. of nitrous vitriol used, and also heavy corrosion of the Pb of the production towers. The Rothamel-Bodamer tower plant, in which gases from the upper part of the tower rich in NO are recirculated by an exhauster, escapes this dilemma. Production cost estimates are given.

PETERSEN replies that these estimates are conjectural as the Rothamel-Bodamer process has nowhere been tested in practice. The importance of gaseous NO as against liquid nitrous vitriol is questioned, a practical test being quoted. The use of very conc. nitrous vitriol does not in reality lead to N losses. Corrosion of Pb has been much reduced in modern tower systems by suitable protection, and dry NO<sub>2</sub> gas is more injurious than nitrous vitriol. C. I.

Thermal analysis of talc-carbonate rock. N. V. ANDREEV (Min. Suir., 1931, 6, 822–828).—Talc undergoes endothermic changes at  $300-400^{\circ}$ ,  $635-665^{\circ}$ , and  $1000^{\circ}$ ; at  $600-800^{\circ}$  the process is more energetic owing to decomp. of MgCO<sub>3</sub>. At 900–1000° the effect is caused by decomp. of talc and liberation of H<sub>2</sub>O. At 900° there is an exothermic effect caused by oxidation of FeO to Fe<sub>2</sub>O<sub>3</sub>. CH. Abs.

Preparation of nickel carbonate-kieselguhr mixture. L. TURBIN (Masloboino Zhir. Delo, 1930, No. 11—12, 57—59).—The principal losses of Ni during hydrogenation are due to the formation of sol. NiHCO<sub>3</sub>. To minimise these losses the NiCO<sub>3</sub>–SiO<sub>2</sub> mixture is prepared by adding to the hot mixture of NiSO<sub>4</sub> and kieselguhr Na<sub>2</sub>CO<sub>3</sub> in excess of only 5—10%, continuing the boiling for 5—10 min., and washing with hot H<sub>2</sub>O free from CO<sub>2</sub>. CH. Abs.

Preparation of manganese dioxide by electrolysis. V. P. ILINSKI and N. P. LAPIN (J. Appl. Chem., Russia, 1932, 4, 757—764).—Pyrolusite was reduced by anthracite at 850—900° to  $Mn_2O_3$ , which was dissolved in dil. (25%)  $H_2SO_4$  and electrolysed at 25° with (Pb) anode and (C) cathode c.d. respectively > 5 and 20 amp. per sq. dm. The potential fall was 3–3.6 volts. Lustrous flakes of  $MnO_2$  were obtained in 75–85% yield. CH. Abs.

Determination of iodine in cuprous iodide. A. ROUSSY (Chem.-Ztg., 1932, 56, 703-704).—The  $Cu_2I_2$  obtained from the mineral springs of Java is almost always contaminated with oil, which renders the usual analytical methods inapplicable. In the method recommended, the sample (10 g.) is wetted with aq. NH<sub>3</sub> ( $d \circ 88$ ), H<sub>2</sub>O is added, and the whole is boiled. The clear solution is decanted, the residue being further extracted with aq. NH<sub>3</sub>, an aliquot part is acidified and oxidised with Br-H<sub>2</sub>O, and excess Br is removed by PhOH. H<sub>3</sub>PO<sub>4</sub> is added to prevent interference by Fe, and the solution is titrated with KI and Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution. H. F. G.

Behaviour of S compounds of Ca and Mg.— See II. Talcs.—See VIII. Se and Te in electrolytic Cu.—See X. MnO<sub>2</sub>.—See XI. Fertilisers.— See XVI. Gas-mask charcoal for COCl<sub>2</sub> and HCl.— See XXIII.

See also A., Oct., 999, Cl<sub>2</sub>-resistant Ag-Pb electrodes. 1004, NH<sub>3</sub> catalysis. 1007, Prep. of pure salts by base-exchange. 1008, Prep. of pure ZnCl<sub>2</sub>.

#### PATENTS.

Manufacture of hydrochloric acid and apparatus [furnace] therefor. CHEM. & METALLURG. CORP., LTD., J. W. CRABTREE, and S. B. CASSON (B.P. 375,039, 21.5.31).—In a HCl furnace, means are provided for feeding  $H_2SO_4$  eccentrically to the axis of the rabbles at a point above the rabble arms, which are protected from attack by the acid by means of detachable guards fixed to each arm. A. R. P.

[Platinum etc.] catalyst for oxidation of ammonia with oxygen or gases containing oxygen. I. W. CEDERBERG (B.P. 377,974, 30.4.31. Ger., 30.4.30). —Galloon net gauzes of Pt or its alloys are claimed.

A. R. P.

Preparing lye with enhanced wetting and impregnating power [for mercerising cotton]. N. V. CHEM. FABR. "SERVO," and M. D. ROZENBROEK (B.P. 379,018, 9.6.31. Holl., 16.9.30).—The addition of 0.25-4% of a mixture of a sol. and an insol. phenol to aq. NaOH (d 1.3) is claimed. Suitable phenol mixtures are cresol or xylenol (7—8 pts.) with carvacrol or thymol (3—2 pts.); they may be diluted with small quantities of aliphatic, aromatic, or cyclic alcohols. A. R. P.

Production of alkali sulphates and sulphur [from alkali bisulphites]. METALLGES, A.-G. (B.P. 378,684, 28.9.31. Ger., 13.5.31).— $(NH_4)_2SO_4$  is obtained by the following reactions in  $H_2O$ : (a) CaS +  $6NH_4HSO_3 = (NH_4)_2SO_3 + (NH_4)_2S_2O_3 + (NH_4)_2S_3O_6 + H_2O + CaSO_3,2H_2O$  (ppt.) and, after filtration, (b)  $(NH_4)_2SO_3 + (NH_4)_2S_3O_6 = 3(NH_4)_2SO_4 + 3S$ . Reaction (b) proceeds to completion at 100—150° under pressure. The CaSO\_3,2H\_2O produced in (a) is reduced to CaS for use again by reduction with C at 1000°. The process is applicable to the utilisation of the SO<sub>2</sub> content of gases containing insufficient SO<sub>2</sub> for conversion into  $H_2SO_4$ , the SO<sub>2</sub> being removed by scrubbing with aq. NH<sub>3</sub>.

Production of big crystals [of ammonium chloride]. P. SEIDLER (U.S.P. 1,839,124, 29.12.31. Appl., 3.12.28. Ger., 10.8.26).—The solution is treated with pectin or a pectin derivative and a little HCl and allowed to cool slowly. A. R. P.

Production of solid diammonium phosphate from impure phosphoric acid. STOCKHOLMS SUPER-FOSFAT FABRIKS AKTIEB. (B.P. 376,174, 8.12.31. Swed., 9.12.30).—Crude  $H_3PO_4$  is neutralised with NH<sub>3</sub> to  $p_H 5$ under such conditions that the temp. rises to  $> 100^{\circ}$ to remove part of the  $H_2O$ ; the Ca, Al, Fe, etc. impurities are pptd. as phosphates, which are removed and the solution is treated with NH<sub>3</sub> to  $p_H 8$  while the temp. is kept at  $< 80^{\circ}$ . The hot solution is then sprayed into warm air at  $\ll 70^{\circ}$  to obtain  $(NH_4)_2HPO_4$  in the form of small rounded grains suitable for storage and distribution. A. R. P.

**Decomposition of raw phosphates.** H. WADE. From AKTIEB. KEMISKA PATENTER (B.P. 378,670, 8.9.31). —Apatite is heated with  $H_2SO_4$  and  $H_3PO_4$  to convert the Ca present into stable  $CaSO_{4,0} \cdot 5H_2O$ . The temp. required is given by the expression  $2p + t \equiv 186$ , where t is the temp. and p the %  $P_2O_5$  in the liquor when the reaction is finished. Leaching the product affords  $H_3PO_4$  relatively free from  $CaSO_4$ . A. R. P.

Manufacture of (A, D, F) acid phosphate, (B) soluble phosphates, (C, H) double superphosphate, (E, G, I, J) phosphatic fertiliser. (A-C, E-J) B. OBER, (A-J) E. H. WIGHT, and (C, E, H, J) W. H. WAGGAMAN, Assrs. to OBERPHOS Co. (U.S.P. 1,837,283-5, 1,837,304-5, 1,837,307, 1,837,328, 1,837,330-2, 22.12.31. Appl., [A] 29.5.26, [B] 7.10.26, [C] 12.3.27, [D] 29.4.26, [E] 19.10.27, [F] 8.2.27, [G] 22.12.26, [H] 17.3.27, [I] 30.3.27, [J] 26.11.27. Renewed [C] 29.1.31, [D] 6.2.29, [J] 8.10.31).-(A) Finelypowdered phosphate rock is mixed with H<sub>2</sub>SO<sub>4</sub> in an evacuated autoclave and the mixture heated until the pressure rises to several atm. ; when reaction is finished the pressure is released and the vessel again evacuated to cause the reaction product to crystallise. (B) Decomp. of the coarse rock is effected with NO·SO<sub>2</sub>·OH under pressure in a rotating autoclave in which the rock is ground during decomp. (c)  $H_3PO_4$  from a previous operation or P2O5 and H2O are used to decompose the rock in an autoclave. (D) Finely-ground rock is mixed with H<sub>2</sub>SO<sub>4</sub> and the mixture passed to an autoclave in which it is heated to 200° under pressure to complete the conversion into  $Ca(H_2PO_4)_2$ . (E)  $H_2SO_4$  (d 1.4) is used in the decomp. of the rock. (F) Crystallisation of the reaction product of any of the above procedures is promoted by externally or internally cooling the reaction vessel under reduced pressure. (G) Materials containing N or K are added to the charge of H<sub>2</sub>SO<sub>4</sub> and rock to obtain a mixed fertiliser. (H) H<sub>3</sub>PO<sub>4</sub> is used instead of  $H_2SO_4$  in the process claimed in (A). (1) The charge is cooled for a short time during mixing in the autoclave, and as it becomes viscous is heated under pressure to complete the reaction. (J) The products are dried by alternately injecting a hot gas into the autoclave and reducing the pressure. A. R. P.

Purification of phosphatic liquids. N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 376,178, 15.12.31 Holl., 23.12.30).—The use of starch or other org. colloid for coagulating the (Fe, Al)PO<sub>4</sub> ppt. obtained in making  $(NH_4)_2HPO_4$  from natural phosphate rock is claimed. A. R. P.

Manufacture of nitrate of lime in globular form. NORSK HYDRO-ELEKTRISK KVAELSTOF A./S. (B.P. 380,007, 29.3.32. Ger., 22.2.32).—A solution of "nitrate of lime," containing 4.7% NH<sub>4</sub>NO<sub>3</sub> and 13.3% H<sub>2</sub>O, at 100° passes to a trough, one side of which constitutes a rotating cylinder, internally cooled to 20°. The layer of solidified material on the surface of this cylinder is removed by a scraper and conducted to a steam-heated vessel with helical conveyor and there heated to 96°; the resultant broth is passed to a heated rotating disc, whence it falls in the form of drops. W. J. W.

Manufacture of bleaching powder. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 375,827, 1.4.31).— Ca(OH)<sub>2</sub> with about 8% excess H<sub>2</sub>O is passed downwards at 390 kg. per hr. through a slightly inclined rotating tube up which Cl<sub>2</sub> is passed at 240—270 cu. m. per hr., the upper part of the tube being cooled and the lower part heated so that the temp. does not fall below 50° until all H<sub>2</sub>O is expelled from the CaOCl<sub>2</sub>. The Cl<sub>2</sub> may be led countercurrent to the Ca(OH)<sub>2</sub> if H<sub>2</sub>O is sprayed into the reaction zone, and in both cases part of the H<sub>2</sub>O may be replaced by CCl<sub>4</sub> or CHCl<sub>3</sub>. A. R. P.

Manufacture of calcium boride. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 378,469, 12.5.31).— A mixture of  $CaCO_3$  (50),  $B_2O_3$  (100), and coke (60 pts.) is heated to whiteness in an electric resistance furnace from which air is excluded.  $H_3BO_3$  in equiv. amount may replace  $B_2O_3$ . A. R. P.

Flux for welding iron and steel. DEUTS. EDEL-STAHLWERKE A.-G. (B.P. 378,802, 22.2.32. Ger., 25.2.31). —A fused 1:1 mixture of CaF<sub>2</sub> and Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub> is claimed. A. R. P.

Decolorising of heavy spar [barytes]. METALLGES. A.-G., and M. SCHTECHEL (B.P. 376,080, 1.9.31. Ger., 25.11.30).—Crude BaSO<sub>4</sub> containing Fe<sub>2</sub>O<sub>3</sub> and/or MnO<sub>2</sub> is roasted with Na<sub>2</sub>SO<sub>4</sub>, K<sub>2</sub>SO<sub>4</sub>, Na<sub>2</sub>SiO<sub>3</sub>, or ZnO, with or without an oxidising agent, *e.g.*, KNO<sub>3</sub> or a peroxide. A, R. P.

(A-C, E-G) Chlorination of iron sulphide-containing material and (D) recovery of sulphur therefrom. R. F. BACON, Assee. of (A, D, F) I. BENCOWITZ, (B, C) H. T. HOTCHKISS, and (E, G) R. FANELLI (B.P. 374,069-71, 374,098, 375,370, 375,795, 375,798, [A-C, E, F] 25.2.31, [G] 26.2.31, [D] 27.2.31. U.S., [A, B, G] 26.2.30, [C, E, F] 25.2.30, [D] 5.3.30).-(A) The ore is heated in a current of SO<sub>2</sub>Cl<sub>2</sub> to produce FeCl<sub>2</sub>, S, and SO<sub>2</sub>, the FeCl<sub>2</sub> is converted into FeCl<sub>3</sub> which is sublimed and burned in air to give Fe<sub>2</sub>O<sub>3</sub> and Cl<sub>2</sub>, and the SO2 and Cl2 are returned to the chlorination chamber. (B) S<sub>2</sub>Cl<sub>2</sub> at 300-550° is used in the process and regenerated by causing the recovered Cl<sub>2</sub> to react with part of the S formed in the chlorination process. (c) The ore is heated in Cl<sub>2</sub>, SO<sub>2</sub>Cl<sub>2</sub>, or S<sub>2</sub>Cl<sub>2</sub> at temp. gradually rising from 300° to 550° so that the Fe is volatilised as FeCl<sub>3</sub> in the hotter zones. (D) The ore or matte is heated at about 700° in a current of HCl and air, CO<sub>2</sub>, CO, or flue gases to product FeCl<sub>2</sub> and gases containing H<sub>2</sub>S, SO<sub>2</sub>, COS, or mixtures thereof which are then treated for recovery of S. (E) The ore is

heated with a chlorinating agent to liberate S and form  $FeCl_2$  which is treated with steam and air at 440-600° to produce HCl and  $Fe_2O_3$ . The HCl is passed with air over coke, pumice, or brick soaked in CuCl<sub>2</sub>, NiCl<sub>2</sub>, CuSO<sub>4</sub>, or NiSO<sub>4</sub> and heated at 370-400° to regenerate Cl<sub>2</sub> for use again. (F) In the process claimed in (E), the FeCl<sub>2</sub> is chlorinated to FeCl<sub>3</sub> before conversion into  $Fe_2O_3$  and HCl. (G) The ore is heated in Cl<sub>2</sub> at 300-450° to form FeCl<sub>3</sub> and S<sub>2</sub>Cl<sub>2</sub>, the FeCl<sub>3</sub> is heated in steam to form HCl and Fe<sub>2</sub>O<sub>3</sub>, the S<sub>2</sub>Cl<sub>2</sub> is used to chlorinate more ore to FeCl<sub>2</sub> and S, the FeCl<sub>2</sub> is chlorinated to FeCl<sub>3</sub> before is chlorinated to FeCl<sub>3</sub> which is decomposed with steam, and the HCl is oxidised to Cl<sub>2</sub> as described in (E). A. R. P.

Recovery of molybdenum from mixtures of molybdenum or molybdenum compounds with other metals or metal compounds. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 375,948, 18.5.31. Addn. to B.P. 350,135; B., 1931, 765).—Spent catalysts containing Mo, such as those used in the destructive hydrogenation of low-temp. lignite tar, are roasted at  $400-600^{\circ}$  and the Mo is extracted by  $(NH_4)_2CO_3$ and/or aq.  $NH_3$ , preferably with addition of a little  $(NH_4)_2S$ . A. R. P.

Recovery of molybdenum [oxide from molybdenite]. C. V. IREDELL, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,838,767, 29.12.31. Appl., 2.5.30).— Roasted molybdenite is extracted with aq. NH<sub>3</sub> to obtain a solution having  $d \ 1 \cdot 19$ . Conc. HCl is run into the clear liquor which is vigorously agitated until just neutral to Me-orange, whereby  $4(NH_4)_2O,19MoO_3$  is pptd. The ppt. is washed with 20% aq. NH<sub>4</sub>Cl, redissolved in aq. NH<sub>3</sub>, and repptd. after oxidising and filtering off any Fe etc. Ignition of the ppt. affords pure  $MoO_3$ . A. R. P.

**Production of solidified carbon dioxide.** W. L. SMITH (B.P. 378,490, 8.5.31).—Gases from the combustion of petroliferous material are purified, cooled, and treated with a suitable solution to absorb  $CO_2$ , this being separated by spraying the solution over heated coils, liquefied, and allowed to expand. W. J. W.

Manufacture of hydrogen from hydrocarbons [other than methane]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 379,014, 6.6.31).—Vapours of hydrocarbons with a low b.p. are mixed with an excess of steam and passed through a steel tube packed at the front end with a catalyst comprising a mixture of pptd. Ni, Al(OH)<sub>3</sub>, and Mg(OH)<sub>2</sub> bonded with Al<sub>2</sub>O<sub>3</sub> cement, and at the back with a catalyst obtained by reduction of a mixture of NiCO<sub>3</sub>, MgO, and kaolin. The front part of the tube is heated at 400—700° and the back at 700—750°. The issuing gases contain >75% H<sub>2</sub> and <15% CO and no soot is deposited in the catalyst even after several months' use. A. R. P.

Recovery of sulphur dioxide from gases containing the same. W. M. CLARK, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 378,464, 12.5.31).—The gases are treated with a solvent for SO<sub>2</sub>, such as an aq. solution of an alkali hydroxide and/or alkali sulphite, in presence of a salt of a weak base and strong acid, e.g., AlCl<sub>3</sub>, which by undergoing hydrolysis with rise of temp. increases the  $p_{\rm H}$ . Substantially the whole of the SO<sub>2</sub> is recovered on heating the solution to 100°. W. J. W. Simultaneous recovery of phosphorus or oxides of phosphorus and mixtures of hydrogen and carbon monoxide. C. PADOVANI (B.P. 378,226, 7.9.31. It., 5.9.30).—Mineral phosphates are heated to  $1000-1500^{\circ}$ , together with SiO<sub>2</sub> if desired, in presence of gases containing hydrocarbons, *e.g.*, natural gas, the nascent H liberated acting as a powerful reducing agent. The mixture of H<sub>2</sub> with varying amounts of CO may be used for heating or for the synthesis of hydrocarbons. W. J. W.

Thermal insulation.—See I. Concentrating Ge. —See X. Electrolytic cells. O<sub>2</sub>-H<sub>2</sub> generator.— See XI. Rinsing etc. agents.—See XII. Fertilisers. Insecticides.—See XVI. Foams.—See XX.

# VIII.—GLASS; CERAMICS.

Clay mixtures for glass-melting pots. IV. Corrosion-resisting properties. J. H. PARTRIDGE, G. F. ADAMS, and J. R. LAIT (J. Soc. Glass Tech., 1932, 16, 131-144T; cf. B., 1931, 883).-The decrease in size of test-pieces subjected to the action of the glass was measured. Siliceous materials are more readily corroded than aluminous ones, which latter tend to form a white protective layer consisting chiefly of mullite. Closeness of grain decreases the rate of corrosion. Siliceous clays tend to pit and consequently to produce stones. The quality of the glass after contamination by the refractory was judged by examination after grinding and polishing. "Cordiness" was more prevalent with aluminous clays and in the small-scale melts the strains set up by cordiness could not be removed by annealing. J. A. S.

Some special forms of corrosion of fireclay pots used for melting potash-lead oxide-silica glass. E. PRESTON and W. E. S. TURNER (J. Soc. Glass Tech., 1932, 16, 240-243T).—Several instances of corrosion of the interior of the crown and of the pot mouth were examined. The zones of corrosion (white or yellow slag) contained PbO 26%,  $Al_2O_3 + Fe_2O_3$ 17.3%. Viscous "stalactites" on the upper interior of the pot mouth contained PbO 38.9%,  $Al_2O_3 +$  $Fe_2O_3$  17%. The corrosive agent was undoubtedly the volatilised PbO, and a hard firing of the pots before use is urged as the best preventive step. J. A. S.

Volatilisation and vapour tension at high temperature of an alkali-lead oxide-silica glass. E. PRESTON and W. E. S. TURNER (J. Soc. Glass Tech., 1932, 16, 219-239T).-A glass of the composition  $SiO_2 55 \cdot 57$ , PbO 29 · 83,  $Al_2O_3 + Fe_2O_3 1 \cdot 29$ , CaO 0 · 22, Na20 5.18, K20 7.60% was heated at temp. from 900° to 1400° for periods up to 200 hr. and the rate of loss in wt. was followed. PbO is very volatile, but volatilisation of alkali oxides is appreciable only above 1200°. At 1400° all the PbO is lost from a layer of glass 3 mm. deep in < 40 hr. The relationship between log (initial rate of loss) and  $1/T^{\circ}$  (abs.) is linear and similar to that between v.p. and temp. The relationship x/(A - x) = kt(where x is amount volatilised at time t, and A is original amount present) holds true up to the time of  $\frac{1}{2}$  loss. The rate of loss also depends on the area exposed, the depth and the rate of diffusion in the glass. A long-period experiment showed that a concn. gradient is set up in a deep crucible, the relationship connecting rate of volatilisation with time (t) and the depth of glass (h) being  $\log\{(A-2x)/A\} = -2kt/h$ . V.p. measurements were made by the dynamical method and the vals. obtained from 1100° to 1350° ranged from 0.16 to 3.90 mm. Hg. The relationship between v.p. and temp. was precisely the same as that between rate of volatilisation and temp. The heat of volatilisation of PbO is calc. to be approx. -60,000 g.-cal. J. A. S.

Effect of gases on a commercial glass containing lead oxide. J. H. PARTRIDGE (J. Soc. Glass Tech., 1932, 16, 121–130r).—A white scum on the surface of a (30%) PbO glass heated in an oil-fired furnace was found to be a ppt. of  $\alpha$ -cristobalite (with small amounts of sulphates of Ca, Mg, Pb, etc.) caused by the presence of SO<sub>3</sub> in the furnace gases. As glasses less rich in alkalis are less stable in this respect, the action of the SO<sub>3</sub> is probably the effective removal of alkalis by combination with them. Parmelee has noticed a similar effect with enamels. J. A. S.

Causes of crystallisation of window glass produced by the Fourcault process. S. U. YUKOVSKI (Trans. Ukrain. Sci. Res. Inst. Silicate Ind., 1929, 1).—The crystals are tridymite and cristobalite, sometimes with a eutectic tridymite + Na<sub>2</sub>O, 3CaO, 6SiO<sub>2</sub>. The rapidity of the appearance of crystallisation is influenced by the volatilisation of alkalis, the action of furnace gases, the contact of glass with the grog particles of the walls of the tank or pot, and the surface tension of glass. Zones where conditions are favourable for crystallisation can be detected by thermal measurements. CH. ABS.

Viscosity-temperature relationship and some inferences on the nature of molten and of plastic glass. S. C. WATERTON (J. Soc. Glass Tech., 1932, 16, 244-253 T).-Applying the equations of Fulcher and Andrade to the data of English and of Lillie on the viscosity of Na<sub>2</sub>O-CaO-SiO<sub>2</sub> glasses, it was concluded that at temp. < that at which log  $10\eta = 8$  the glasses are either not associated or associated to a const. degree. At higher temp. the relationship departs from the linear, due to dissociation. An alternative equation based on those of Andrade and Frenkel for associated liquids was deduced for glasses. In the case of Na<sub>2</sub>O-B<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> glasses the degree of association increases with increase in B2O3 content. The "aggregation temp." suggested by Fulcher is considered unreal and due to the two different methods of measurement used over the two ranges of temp. J. A. S.

Tensile strength of glass. E. J. GOODING (J. Soc. Glass Tech., 1932, 16, 145—170 r).—A simple, directly loading apparatus applies the tension (through long wires, to avoid shear stresses) to a fibre of glass approx. 1 mm. diam. and constricted at its centre point. The fibre was held by blobs fused on to its ends. The type of fracture appears to be related to the tensile strength and, though the strength increases with decrease in diam. of the fibre, is decreased by matt etching, and is increased by acid polishing, it is unaffected by the method of loading, the temp. of the constriction formation, the presence of strain, and the influence of S fumes during annealing. The results, in general, are explained by the effect of surface flaws. J. A. S. Colour of selenium ruby glasses. H. P. ROOKSBY (J. Soc. Glass Tech., 1932, 16, 171—181 T).—X-Ray spectrographic examination showed that the coloration is caused by the pptn. of particles consisting of a solid solution of CdS and CdSe, and not, as in the case of Cu and Au ruby glasses, to the optical effect of colloidal particles. Facts in support of this statement are enumerated. For a colour stable against temp. of development the Cd content should be relatively high; this is ensured by the addition of CdO or CdSO<sub>4</sub> to the batch. Any required colour can then be obtained by appropriate adjustment of the Se : CdS ratio. J. A. S.

Influence of thermal treatment on opacity of fluoride opal glasses. I. KITAIGORODSKY and S. I. KUROVSKAJA (J. Soc. Glass Tech., 1932, 16, 210–218 T). —The relations between the heat treatment and the optical properties (general and unscattered transmission, reflection, and adsorption) of these glasses is in accordance with Tammann's theory on the rate of crystallisation of supercooled solutions. Every glass has its max. opacifying temp., which for a glass having the composition  $SiO_2 67.52$ ,  $Al_2O_3 3.81$ , CaO 5.86,  $Na_2O 18.54$ , F 4.0%is 650—700°. The glass must not be rapidly cooled during manufacture and sufficient time must be allowed at the crit. temp. J. A. S.

Ceramic properties of Ural talcs of industrial importance. L. V. OMININ and F. M. SINYAKOV (Min. Suir., 1931, 6, 828—849).—Freeing talc from ore and Fe affects the m.p. (approx. 1525°) little. All grades show a sharp change in the loss on heating at 800—1000°. owing to dehydration and possibly mol. regrouping with formation of clinoenstatite. Mol. changes also occur at 1300—1430°, and colour change from grey to yellow and brown takes place at 800—1000°. The hardness and crushing point of talcs fired at 1300—1430° are sharply reduced. Breaks in the *d* curve occur at 800— 1000° and 1300—1380°. CH. ABS.

Gas permeability as a testing method for grog bodies. Y. KVASHENINIKOV and K. KUMANIN (Keram. i Steklo, 1932, 8, No. 1, 24—26).—Gas permeability is affected much more by factors of working than are  $H_2O$ absorption and shrinkage. Firing temp., followed by size of grain and proportion of clay to grog, have the greatest influence on gas permeability, which increases as the firing temp. is raised. With increased clay content the gas permeability and  $H_2O$  absorption decrease, whilst the shrinkage increases. CH. ABS.

Hydraulic losses in pumping clay slurries. A. TSCHITSCHENKO and B. BAKLANOV (Azerbaid. Neft. Choz., 1932, No. 4, 69—73). CH. Abs.

Talcs.—See VII.

See also A., Oct., 997. System FeO-SiO<sub>2</sub>. Ceramics of ZrO<sub>2</sub> refractories.

#### PATENTS.

(A) Frits for production of enamels. (B) Production of white opaque enamels and glazes. I. KREIDL (B.P. 375,670 and Addn. B.P. 375,672, [A] 28.10.31, [B] 29.10.31. Austr., [A] 10.12.30, [B] 12.3.31).—(A) Enamel frits containing a high content of Al<sub>2</sub>O<sub>3</sub>, preferably added in the form of a clay, and of F, preferably added as Na<sub>2</sub>SiF<sub>6</sub>, and of such a composition as to be colloidal after fine-grinding in  $H_2O$  are claimed. (B) The  $(Na,K)_2O$ :  $B_2O_3$  ratio in the frit is > 2:1, preferably 3-5:1. A. R. P.

Production of white opaque enamels and glazes, especially sheet-iron enamels. I. KREIDL (B.P. 375,673 and Addn. B.P. 375,674, [A] 2.11.31, [B] 4.11.31. Austr., [A] 10.12.30, [B] 24.12.30).—Enamel frits of the type claimed in B.P. 375,670 and B.P. 375,672 (preceding abstract) are opacified with (A) org. compounds which decompose at the firing temp. with the production of minute gas bubbles, *e.g.*, methylene-blue, or (B) mixtures or compounds of such org. substances with solid opacifying agents, *e.g.*, ZrO<sub>2</sub>, Sb<sub>2</sub>O<sub>4</sub>, or SnO<sub>2</sub>. A. R. P.

Making glass, enamels, glazes, and the like containing lead. P. BEYERSDORFER (B.P. 378,799, 18.2.32).—Finely-divided metallic Pb (conveniently dispersed by stirring molten Pb with the sand and afterwards roasting at 400—500° for several hr.) is introduced into the batch with sufficient KNO<sub>3</sub> or  $K_2CO_3$ , BaCO<sub>3</sub>, etc. to oxidise the remaining Pb to PbO. Other metals (Zn, Sb, Sn, Cu, Ni, Au, etc.) are conveniently introduced by alloying them with the Pb.

J. A. S.

Production of lead glasses, glaze frits, and the like. T. GOLDSCHMIDT A.-G. (B.P. 378,844, 21.6.32. Ger., 14.9.31).—PbO which is rendered more chemically reactive by being fused and then solidified (by fusion of powdered PbO or by blowing air through metallic Pb at 1000—1100°, removing the molten PbO from the surface, cooling, and grinding) is introduced into the batch, with or without previous reaction with  $SiO_2$ .

J. A. S.

Glass treatment. H. R. MOULTON and E. D. TILLYER, ASSTS. to AMER. OPTICAL CO. (U.S.P. 1,838,781, 29.12.31. Appl., 17.3.30).—Glass is strengthened by subjecting it to a compressive strain introduced by embedding in the soft glass a material (e.g., a metal ring) having a high coeff. of expansion. J. A. S.

Fusing of silica and the like. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of P. K. DEVERS (B.P. 375,500, 19.5.31. U.S., 19.5.30).—SiO<sub>2</sub> sand is fused by electric-resistance heating in an evacuated furnace completely filled with sand, and means are provided for tapping the fused sand without affecting the unfused surrounding material. The product is completely free from gas cavities. A. R. P.

Ceramic composition. T. S. CURTIS and M. J. WALSH, ASSTS. to KELCO CO. (U.S.P. 1,838,809, 29.12.31. Appl., 26.6.28).—Dry compositions needing only the addition of  $H_2O$  for the development of plasticity are made by mixing such non-plastic materials as cyanite, SiO<sub>2</sub> sand, etc. with 1—2% of dry, powdered NH<sub>4</sub> alginate (derived from kelp). J. A. S.

Plasticising of ceramic compositions of feeble plasticity. T. S. CURTIS, ASST. to KELCO Co. (U.S.P. 1,838,810, 29.12.31. Appl., 30.7.28).—The addition of 5% of NH<sub>4</sub> alginate paste (or other gel-producing materials derived from algæ such as kelp) to moistened non-plastic materials such as cyanite or SiO<sub>2</sub> sand renders them as plastic as a "fat" clay. J. A. S.

Manufacture of bricks and other ceramic moulded articles. F. BANDINI (B.P. 378,577, 9.6.31).—In

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order to accelerate and combine the drying and firing operations, the plastic clay is heated to approx.  $95^{\circ}$ , pressed or extruded, packed in sand (mixed with a powdered or liquid fuel and heated to  $95^{\circ}$ ) in a refractory container, and the whole passed through an externally heated tunnel kiln, the firing being completed by the fuel mixed with the sand. The combined operations occupy 36 hr. and the wt. of fuel required is only (approx.)  $6^{\circ}_{0}$  on the wt. of the brick. J. A. S.

Ceramic electric-heating bodies. PORZELLANFABR. KAHLA (B.P. 375,692, 2.12.31. Ger., 13.3.31).—The upper surface of electric hot plates is made of a plate (A) of a dense, sintered, ceramic mass and the lower surface of a plate (B) of porous sintered material, the resistance wires being arranged in channels in B; a spring device clamps A and B together. A. R. P.

Refractory insulator support for electron-discharge devices. C. V. IREDELL, Assr. to WESTING-HOUSE LAMP CO. (U.S.P. 1,838,766, 29.12.31. Appl., 24.9.28).—The body for the extrusion of the refractory former supporting the cathode of the "indirectly heated" thermionic valve consists of a mixture of pure  $Al_2O_3$  (sintered at 1500—1600°) with 2% of talc etc. plasticised with an org. binder (e.g., flour paste + aq. NH<sub>3</sub>) and fired at 1500—1600° for 3 hr. J. A. S.

(A, B) Moulding for manufacture of refractory articles. (c) Moulding of plastic masses. E. LUX, W. H. DANIELS, and E. KUHN, Assees. of H. KOPPERS A.-G. (B.P. 380,432-4, [A] 15.4.31, [B, C] 1.5.31. Ger., [A] 16.4.30, [B] 3.5.30. Addns. [B, C] to [A]).

(A) Deposition of granular material [sand] on an adhesive-coated carrier [paper]. (B, C) Manufacture of abrasive-coated materials [e.g., sandpaper]. (A, C) W. J. TENNANT. From BEHR-MANNING CORP. (B) BEHR-MANNING CORP., Assees. of E. C. SCHACHT (B.P. 378,014, 378,057, and 379,379, [A] 28.3.31, [B, C] 28.3.31. U.S., [B] 14.7.30).

Metal-porcelain joints.—See X. Ceramic pigments.—See XIII.

# IX.—BUILDING MATERIALS.

**Trass-Portland cement.** A. GUTTMANN and F. GILLE (Tonind.-Ztg., 1932, 56, 75-77, 90-92; Chem. Zentr., 1932, i, 1567).—Shrinkage of trass-Portland cement mortar is > that of trass-free cement. The standard strength with up to 30% of trass is sometimes > with normal cements. The compressive strength of the concrete is lower. Trass-Portland cements have a smaller modulus of elasticity. A. A. E.

Dissolution of wood in wood tars and phenols. H. BERGSTRÖM and K. N. CEDERQUIST (Iva, 1931, No. 1, 12—14).—The products resulting from heating wood with tar phenols under pressure are described.

#### PATENTS.

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CH. ABS.

**Cement.** CEMENT MARKETING Co., H. KEEBLE, and H. N. WRIGLEY (B.P. 377,956, 29.1. and 19.9.31).— In the prep. of a surface-coating material, cement or a mixture of cement and a finely-ground aggregate is mixed with size and a solution of a silicate or fluosilicate, e.g.,  $\leq 50\%$  of size,  $\leq 5\%$  of Na silicate, and  $\leq 25\%$  of Na fluosilicate on the cement wt. C. A. K.

Forming cementitious bodies at elevated temperatures. H. H. STEINOUR, ASST. to RIVERSIDE CEMENT Co. (U.S.P. 1,839,612, 5.1.32. Appl., 28.1.30). —For use in hot positions  $(55-95^{\circ})$ , cement with similar total  $R_2O_3$  content to, but containing less  $3CaO,Al_2O_3$  than, Portland cement is claimed. The mol. ratio of  $Fe_2O_3$  to  $Al_2O_3$  in the cement should be between 1:1.4 and 1:0.9 and the gypsum content equiv. to  $1-2.5SO_3$ . C. A. K.

Plasters and the like [ready for use]. G. O. CASE, E. M. ELLIS, and L. H. MONTIGUE (B.P. 378,209, 18.8.31).-70-85% of dry limestone or dolomite passing 50-mesh is mixed with 15-30% of dry Ca(OH)<sub>2</sub> or Mg(OH)<sub>2</sub>. The mixture may also contain 5-15% of plaster of Paris to accelerate setting. C. A. K.

Plastic-forming composition. [Cement substitute.] A. L. V. NLSSON (U.S.P. 1,841,547, 19.1.32. Appl., 26.3.30).—The composition comprises 36% of flint rock, 36% of calcined quicklime rock, and 28% of gypsum calcined at about  $170-200^\circ$ , all crushed to 200-mesh; about 10% of sawmill offal may be added when the material is to be used, *e.g.*, for plastering.

L. Ă. C.

Manufacture of light-weight aggregates and structural blocks formed thereof. E. V. POSTON (U.S.P. 1,842,048, 19.1.32. Appl., 28.8.29).—Argillaceous material (clay and/or shale), preferably worked up with  $H_2O$  to a plastic mass, is heated in a combustion chamber containing gases relatively free from  $O_2$  for a time sufficient to bring the material to a cellular condition; the product is subsequently shaped. L. A. C.

Production of light-weight building materials. R. K. MOBERTY (U.S.P. 1,842,186, 19.1.32. Appl., 3.5.29).—A mixture of clay and a hydrated salt capable of leaving an insol. residue on heating, e.g.,  $Ca(NO_3)_2.4H_2O$  or  $Al_2(SO_4)_3.18H_2O$ , is heated at 100— 300° in a mould permitting expansion in one direction, so that, by the escape of steam, a vesicular structure is obtained; the product is then fired at a higher temp. L. A. C.

Manufacture of light porous [building] materials. E. RODGERS, JUN., and G. D. DUNCAN, JUN. (U.S.P. 1,842,801, 26.1.32. Appl., 24.5.29.—Argillaceous material (shale), preferably moist and mixed with about 5% of coke, is passed over a furnace grate, provided with means for drawing the combustion gases through the material, so that it expands by generation of gases. The material is stirred at first to prevent coalescence, but before leaving the chamber it is allowed to coalesce into a slab which, after removal, is broken up for use as concrete aggregate. L. A. C.

Slag treatment and apparatus therefor. E. P. DUNN (B.P. 374,008, 23.2.31. Austral., 8.3.30).—Fe blast-furnace slag is run into a conical settler to allow the heavy impurities to settle and the lighter to rise to the surface, then tapped into a reverberatory furnace in which CaS and other reducing compounds are oxidised by hot air, and finally passed through a rotary furnace in which sand or other siliceous material is added to

bring the SiO<sub>2</sub> content to about 70% and render the slag resistant to weathering. A. R. P.

Insulating and sound-proofing materials. N. M. ELIAS (U.S.P. 1,842,083, 19.1.32. Appl., 12.8.29).-Na<sub>2</sub>SiO<sub>3</sub> of composition such as to yield a product containing 76-80% of SiO2 is heated below 200° until it contains  $\ge 27\%$  of H<sub>2</sub>O, and the product is heated in moulds above its fusion point (550-700°) for such a time that only the surface layer fuses, after which it is heated at  $400-500^{\circ}$  to complete expulsion of  $H_2O$ . L. A. C.

Production of a hydraulic binding medium in powdered form. H. PLAUSON and M. IMER (B.P. 378,107, 11.5.31).-Bitumen, tar, or pitch is ground with CaO, either cold or heated, so as to give a pulverulent mixture which may be mixed with cement or other constituents. C. A. K.

Emulsification of bituminous and like substances, especially for production of road surfaces. A. L. MOND. From "EMULGIA" VERTRIEBSGES. F. ASPHALT-, TEER-, U. STRASSENBAU-PRODUKTE CLAUSER & Co. Komm.-ges. (B.P. 379,710, 3.3.32).-Asphalt is heated at 130-160° with tar or with a tar-petrol-pitch mixture and the product, with or without the addition of an emulsifying agent (soap) and a coagulant (NaCl), is run into about an equal vol. of H<sub>2</sub>O. L. A. C.

Bitumen suspensions and preparation of road surfaces therefrom. N. V. MIJNBOUW- EN CULTUUR-MAATS. "BOETEN" (B.P. 379,915, 13.11.31. Ger., 17.12.30) .- "Meagered" bitumen (a mixture of 20-60% of bitumen with sand, limestone, etc.) is finely ground with H<sub>2</sub>O, the suspension is mixed with 20 vol.-% of an aq. mineral flux oil emulsion, and the mixture is heated for a short time at 100°. The product is diluted with H<sub>2</sub>O or mixed with sand, crushed stone, or meagered Portland cement before use. L. A. C.

Production of road-paving material. G. W. Alsdorf (U.S.P. 1,842,139, 19.1.32. Appl., 12.12.28).-Bitumen at about 150° is stirred into a mixture of moist aggregate with a kerosene-H<sub>2</sub>O emulsion. L. A. C.

Heat- and sound-insulating coverings. A. G. R. Виск (В.Р. 379,389, 1.6.31).

Adding fluid to pulverulent solid.-See I.

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

Effect of small additions of aluminium to grey cast iron on pouring. S. SIROVICH (Met. Ital., 1932, 24, 83-93; Chem. Zentr., 1932, i, 2226).-The viscosity is not diminished, but is increased by the addition of Al (1-5 in 10<sup>4</sup>), and zones of white cast Fe are formed. The quantity of Al added must not exceed 1 in 105, and a suitable time must elapse between addition and A. A. E. pouring.

Change in composition of basic-hearth steel in the foundry ladle. P. BARDENHEUER and A. RANFFT (Mitt. Kaiser Wilh.-Inst. Eisenforsch., 1931, 13, 291-305; Chem. Zentr., 1932, i, 2226).-Changes are due to diminution in basicity of the slag by absorption of SiO2 and Al2O3 from the lining. Reduction of oxides in the slag is effected by Mn or Si. A. A. E.

Separation of graphite in the crystallisation of cast iron. S. S. STEINBERG and K. A. MALYSHEV (Metallurg. Russia, 1931, 6, 123-129).-Graphite separates as (1) a eutectic consisting of small laminæ (0.01 mm.), (2) larger laminæ (0.1 mm.), (3) large aggregations and nests. Superheating the Fe at 1600° and keeping it liquid for a long time decreases the amount of graphite which separates and favours the formation of the fine graphite eutectic. Slow cooling during crystallisation increases the amount of graphite and the size of the particles. CH. ABS.

Age-hardening in carbon steels quenched below the A1 point. R. YAMADA and K. YOKOYAMA (Kinzoku Kenk., 1931, 480-495).-The age-hardening is closely related to the change in the solubility of C in *α*-Fe below A1; the increase is the greater in low-C steels. The electrical resistance increases by ageing but decreases by tempering; d increases only slightly. In agehardening at room temp., the C atoms in steel quenched below A1 form assemblages causing distortion of the Fe lattice. On tempering, cementite mols. separate in the boundary layers; above 250° the formation of its space lattice takes place. CH. ABS.

Polishing metallographic specimens of cast iron. J. R. VILELLA (Met. & Alloys, 1932, 3, 205-206).-Specimens are ground to 000 emery, then this paper is glazed by rubbing with graphite or soapstone, and the specimen is reground lightly on the glazed surface. Final polishing is carried out on a silk lap with MgO. Use of this method avoids the dragging out of graphite E. H. B. from the specimen.

Corrosive action of potassium (and other alkali) salt solutions on metals. I. Cast iron and lead. V. K. PERSHKE and G. I. CHUFAROV (Ann. Inst. Polytech. Oural, 1929-1930, 7, 81-90).-Cast Fe was most strongly attacked by NaCl, less by KCl, and least by MgCl<sub>2</sub>; sylvinite is more corrosive than carnallite, and dil. solutions are more corrosive than conc. solutions. Light (1.7 times) and air (3 times) increase the action. Pure Fe is less corroded than impure ; Pb is "9 times more resistant than Fe." CH. ABS.

Strength of materials [metals] as affected by discontinuities and surface conditions. F. C. LEA (J. Soc. Glass Tech., 1932, 16, 182-209 T).—The effects of surface flaws and corrosion on the fatigue behaviour of metals is described in detail. Surface flaws undoubtedly decrease the strength by concn. of stress. Although decarburisation of the surface layer may account in part for some failures, it is more probable that oxidation along the crystal boundaries acts as a discontinuity, and thus causes concn. of stress. J. A. S.

Action of sulphur on non-ferrous alloys. I. P. PODOLSKI and N. M. ZARUBIN (Vestn. Metalloprom., 1931, 11, No. 5, 57-62).-Metal rods were heated in sealed tubes with S at 600°; Cu (2-3 mm. diam.) was completely converted into Cu<sub>2</sub>S, whilst Ni becomes coated with Ni<sub>2</sub>S below 600° and Ni<sub>3</sub>S<sub>2</sub> above 600°. Ni-Cu alloys containing solid solutions become covered with an outer layer of Cu<sub>2</sub>S and an inner layer of Ni sulphide. With a W-Ni alloy, forming definite compounds, no zonal separation of the sulphides was observed. CH. ABS.

Rapid development of patina on copper. J. R. FREEMAN, JUN., and P. H. KIRBY (Met. & Alloys, 1932, 3, 190—194).—Two methods of producing patina are described. In the first the Cu is made the anode in an electrolyte containing Na<sub>2</sub>SO<sub>4</sub>, NaOH, and NaCN, and the resulting oxide is converted into basic sulphate by heating at 95° in a 5% solution of CuSO<sub>4</sub>. A more successful method consists in dipping the articles into a 10% solution of  $(NH_4)_2SO_4$  intermittently during 24 hr., followed by immersion for  $\frac{1}{2}$  hr. in hot H<sub>2</sub>O. The resulting patina [mainly (98%) CuSO<sub>4</sub>, 3Cu(OH)<sub>2</sub>] is very resistant in industrial and marine atm.

E. H. B.

Influence of lead and antimony on the working of certain grades of brass. N. S. SEVERGIN (Tzvet. Met., 1930, 5, 1695—1701).—The behaviour during hotrolling depends on the Cu and Pb contents; 62% Cu and 0.03% Pb is satisfactory. Sb has no influence. Traces of Sb, however, affect cold-rolling and diestamping; for ordinary purposes brass should not contain > 0.005% Sb. With Muntz metal the limiting safe concn. of Sb is probably 0.02%. CH. ABS.

Determination of zinc in brass and other alloys by the oxalate-permanganate method. E. KNOP-PICK (Chem.-Ztg., 1932, 56, 663).—The determination of Zn by pptn. with  $Na_2C_2O_4$  in presence of AcOH and titration with KMnO<sub>4</sub> gives very exact results. Sn, Pb, Cu, Fe, Al, and Ni must be separated, but Mn need not be completely removed. If present it is likewise pptd. as oxalate and determined by Volhard's method. AcOH can be replaced in part by the cheaper denatured alcohol. C. I.

Nickel and nickel alloys other than the nickelchromium-iron group. C. A. CRAWFORD and R. WORTHINGTON (Amer. Soc. Mech. Eng. and Amer. Soc. Testing Mat. Symp., 1931, 403–431).—A discussion of Ni and Ni-Cu, Ni-Cu-Zn, and Ni-Mn alloys.

#### CH. ABS.

Electric-resistance alloys for high-temperature service. Anon. (Metallurgia, 1931, 6, 125-127).-The electrical and mechanical properties of commercial Ni-Cr alloys (Nichrome IV, Brightray, and Pyromic) and Ni-Cr-Fe alloys (Nichrome, Glowray, and Calomic), based on makers' claims, are reviewed and the manufacture of 80-20 Ni-Cr wire and tape is briefly described.

E. H. B.

Theory of the tinning process. I. FESZCZENKO-CZOPIWSKI and M. STRZALKO (Prz. Górn.-Hutn., 1930, 22, 379—382).—The fact that the solubility of C in  $\alpha$ -Fe increases with increasing amounts of Sn in solid solution in the Fe is significant when Fe is tinned at too high temp.; optimal temp. are 260—300°, an uneven layer of Sn being produced at higher temp. owing to corrosion, probably because Fe diffuses into Sn more rapidly than the reverse action takes place. The hardness of the Fe near the Sn layer increased from 125 to 162 kg. per sq. mm., whilst that of the Sn near the Fe increased from 6 to 86 kg. per sq. mm.

CH. ABS.

Silumin and its structure. B. OTANI (Kinzoku no Kenkyu, 1930, 7, 666–686).—Addition of 0.5% Bi shifts the eutectic point of the Al-Si system by 2% to

the Si side, and no appreciable lowering or undercooling of the eutectic temp. occurs. The structure of the alloys is finer than that of unmodified alloys, but is coarser than in the case of silumin. Cd gives a similar result. On adding Ni and Mg to Al-Si alloys, ternary eutectics occur at Al 83.7, Si 11.8, Ni 4.5% (567°), and Al 79.7, Si 14.8, Mg 5.5% (551°). Ternary eutectic alloys show a very fine structure, as in the case of silumin. Addition of Cr, Fe, and Ca was also studied.

CH. Abs. Metallic magnesium from dolomite and magnesite. A. TIBY (L'Ind. Chimica, 1931, 6, 1380–1383; Chem. Zentr., 1932, i, 2230).—Thermal reduction with Al is preferred. A. A. E.

Electrochemical reproduction of the macrostructure of metallurgical specimens. E. JIMENO, J. BERNAL, and J. IBARZ (Anal. Fis. Quím., 1932, 30, 655—663).—A thin card moistened with  $K_4$ Fe(CN)<sub>6</sub> solution is placed between the specimen and a sheet of Cu which forms the cathode; the card used must be sized and should not be immersed in the solution for more than 5 min. A suitable wt. is laid on the specimen and current (2—4 volts, 0.02 amp. per sq. dm.) is passed for a few min. Illustrations are given of the results obtained, particularly with alloy steels, to which the method is especially applicable. H. F. G.

Chromium deposits, free from cracks and pores, prepared in the "Kr" chromium-plating bath. P. HENTSCHEL (Metallwaren-Ind. Galv.-Tech., 1932, 30, 106—108; Chem. Zentr., 1932, i, 2233).—In various Krämer and Kleinschroth (patented) electrolytes,  $1 \cdot 5$ ,  $1 \cdot 7$ , and  $2 \cdot 5\%$  H<sub>2</sub>SO<sub>4</sub> gave good results, and the current yield was 20—50% > usual; the electrolyte is not very sensitive to changes in H<sub>2</sub>SO<sub>4</sub> content. The H<sub>2</sub> absorbed by the deposits is less in the "Kr" than in normal baths. A. A. E.

Cadmium plating. C. M. HOFF (Monthly Rev. Amer. Electroplaters' Soc., 1930, 17, No. 6, 19–35).— For general purposes and for very bright deposits, respectively, the bath should contain Cd oxide 3.5, 6; NaCN 10, 16; Na<sub>2</sub>SO<sub>4</sub> 4.2, 6.6; NiSO<sub>4</sub> 0.08, 0.13— 0.21; ligninsulphonate 1, 1.6%. The baths are operated at 15–50 amp. per sq. ft., and at 25 amp. per sq. ft. have a cathode current efficiency of 96%. Accumulations of Na<sub>2</sub>CO<sub>3</sub> are preferably removed with CaO. Costs are discussed. CH. ABS.

Discoloration of cadmium plate and its prevention. G. SODERBERG (Trans. Electrochem. Soc., 1932, 62, 315–322).—Discoloration of Cd plate (deposited from cyanide bath) when stored in still, moist air is greatly accelerated if a HNO<sub>3</sub> bright dip has been used, and is attributed to action of moist NH<sub>3</sub> formed from traces of cyanide or NH<sub>4</sub> salt not washed away from the surface of the plate. Dipping in an oxidising solution before final rinsing and drying prevents this tarnishing. The use of a bright dip containing CrO<sub>3</sub> and H<sub>2</sub>SO<sub>4</sub> in suitable proportions which also prevents tarnishing has been developed. H. J. T. E.

Anodes for zinc plating. G. B. HOGABOOM and A. K. GRAHAM (Trans. Electrochem. Soc., 1923, 62, 283-294).—Addition of small proportions of Al to Zn

decreases the grain size of the cast metal, whereas addition of Hg has little effect on the grain size of Zn on Zn-Al alloys: the Hg accumulates at the grain boundaries. In plating Zn from the acid bath anodes of Zn alloyed with 0.5% Al suffer much less chemical corrosion than ordinary Zn and produce much less sludge. Zn-Hg anodes behave similarly but dissolve somewhat less during simple immersion in the electrolyte. Anodes of Zn alloyed with 0.5% Al and 0.3% Hg are the most satisfactory : chemical corrosion and sludge formation are eliminated, and anodic and cathodic current efficiencies become practically equal so that the Zn content and acidity of the solution are well maintained. The presence of Al and Hg in the solution has a beneficial effect on plating. In cyanide baths Zn-Al and Zn-Al-Hg anodes give much lower polarisation than ordinary Zn. The Zn-Al-Hg alloy gives less sludge than Zn-Al. H. J. T. E.

Deposition of bright zinc-cadmium alloys from cyanide baths. C. J. WERNLUND (Monthly Rev. Amer. Electroplaters' Soc., 1930, 17, No. 11, 4-9).—Permissible and optimal proportions are : Zn(CN)<sub>2</sub> 8—16 (10), NaCN 4—8 (5), NaOH 6—16 (12), Cd oxide 0.13— 0.4 (0.27) oz. per gal. The Zn anodes contain 8—15% Cd; the anode and cathode c.d. are, respectively, 6—10 and 20—40 amp. per sq. ft. with e.m.f. 1—3 volts; electrolysis is conducted at 20—40°.

CH. ABS.

Determination of selenium and tellurium in materials from electrolytic copper plants. A. I. GAIEV (Ann. Inst. Polytech. Oural, 1929—1930, 7, 95—115).—Pb(NO<sub>3</sub>)<sub>2</sub>, followed by excess of NH<sub>3</sub>, is added to a solution of the Cu in HNO<sub>3</sub>; Se and Te are extracted from the ppt. with cold HCl. CH. ABS.

Ni-plating baths .-- See XI.

See also A., Oct., 989, Systems Cu-Au, Cu-Ag, Cd-Hg, Mg-Al-Cu. Electrodeposited Ag-Cd. Hardenable Al-Cu. Time-potential curves on Fe and steel. 990, Systems Fe-C-Si and Cu-Zn. 997, Systems FeO-SiO<sub>2</sub>, MnSiO<sub>3</sub>-Fe<sub>2</sub>SiO<sub>4</sub>, and FeS-Fe<sub>2</sub>SiO<sub>4</sub>. 999, Cl<sub>2</sub>-resistant Ag-Pb electrodes. 1003, Film reactions and corrosion. 1005, Codeposition of Pb and Bi. 1009, Roasting etc. of spathic Fe ore. 1010, Analysis of gases in metals. 1011, Electrolytic separation of Cu and Pb.

#### PATENTS

Concentration of metals [sponge iron]. TRENT PROCESS CORP. (B.P. 377,260, 15.4.31. U.S., 17.4.30).— Sponge Fe, obtained by reduction of Fe ores and preferably ground to 100-mesh, is passed over concentrating tables using a relatively non-volatile hydrocarbon oil instead of  $H_2O$  as the separating medium. After removal of oil from the Fe concentrate it may be further purified by magnetic separation; the tailing in any case is treated magnetically to recover any fine Fe. A. R. P.

Sintering of [siderite] ores. VEREIN. STAHLWERKE A.-G. (B.P. 376,743, 8.4.31. Ger., 12.4.30).—Roasted siderite or similar finely-divided Fe ore is fed on to a layer of red-hot coke in a furnace with a travelling hearth, and a current of hot combustible gases is passed from the sides of the furnace laterally across the charge while air for the combustion of coke and gas is passed upwards from boxes disposed beneath the hearth. A. R. P.

Electrothermic reduction of iron ores. S. T. WILES, ASST. to BUFFALO ELECTRIC FURNACE CORP. (U.S.P. 1,837,696, 22.12.31. Appl., 7.12.28).—The charge of ore, coke, and fluxes is fed into an arc furnace with horizontal electrodes through tubes arranged along the longitudinal axes of the electrodes, so that the ore is reduced before it reaches the arc; means are provided for exhausting the gases from the furnace to prevent back-pressure in the feed tubes. A. R. P.

Smelting process [for making ferrochromium]. F. M. BECKET, Assr. to ELECTRO METALLURG. Co. (U.S.P. 1,835,925, 8.12.31. Appl., 3.5.29).—Chromite with 34% Cr (400), ferrochromium-silicon with 34% Cr, 51% Si, 14% Fe (246), NaNO<sub>3</sub> (117), and CaO (13 pts.), all ground through 100-mesh and intimately mixed, are charged on to a bed of molten slag from a previous operation. A vigorous exothermic reaction occurs and an Fe-Cr-Si alloy with 6% Si is produced ; after tapping off the slag, the alloy is refined by addition of chromite (160) and CaO (16 pts.) followed by electric heating for 1—1½ hr. The resulting alloy contains > 71% Cr and < 0.1% Si + C. A. R. P.

Apparatus for refining steel, cast iron, and other metals obtained by pouring. Soc. ANON. D'OUGRÉE-MARIHAYE, Assees. of J. M. CORNET (A. CORNET) and J. KALTE (B.P. 375,623, 18.9.31. LUXEMD., 22.9.30).—The apparatus comprises a receiver, between the taphole of the furnace and the mould, provided with tuyères for blowing refining gases, e.g., H<sub>2</sub>, around the walls on which the stream of molten metal is spread.

A. R. P.

Production of wrought iron. HIGHLAND IRON & STEEL Co., Assees. of L. E. FRAZIER (B.P. 378,891, 16.4.31. U.S., 8.1.31).—Steel and cast-Fe scrap are melted in a cupola to produce a molten alloy containing about 3.5% C, 0.35% Mn, 0.4% Si, 0.5% P, and 0.1% S, and the alloy is tapped into a rotary mechanical puddling furnace containing sufficient roll scale to oxidise the C, Mn, Si, P, and S. After cessation of boiling and graining, the plastic mass is ejected, squeezed, and rolled into muck bar in the usual way; it contains about 0.02% C, < 0.03% Mn, < 0.1% Si, 0.06% P, and < 0.04% S. A. R. P.

Nitrogenisation of iron, steel, and alloys thereof. ELECTRO METALLURG. Co., Assees. of J. J. EGAN (B.P. 374,096, 26.2.31. U.S., 26.2.30).—Nitrogenisation is effected in an atm. of NH<sub>3</sub> containing a cyclic N compound, e.g., PhNO<sub>2</sub>. A. R. P.

Hardening of [non-rusting] alloy steels. W. H. HATFIELD and H. GREEN (B.P. 377,244, 25.2.31).—The steel, e.g., 18:8 Cr-Ni steel, is pickled in 1:1 HCl, washed, dried, and case-hardened in NH<sub>3</sub> at 500°. A. R. P.

[Protection of] zinc or zinc-coated metal sheets [galvanised iron] and other articles [from "white rust."]. K. D. MACKENZIE and A. C. BARCLAY (B.P. 377,746, 4.5.31).—The articles are dipped into 0.5% aq. CrO<sub>3</sub> for about 5 sec., washed, and dried. A. R. P.

Manufacture of chromium steel by the Thomas process. VEREIN. STAHLWERKE A.-G. (B.P. 379,590 and Addn. B.P. 379,598, [A] 18.12.31, [B] 2.1.32. Ger., [A] 18.12.30, [B] 2.1.31).—(A) The steel charge is blown in the Thomas converter until it contains rather > 0.08% P, the Cr is then added, and the bath blown for a very short period to mix the charge. (B) The Fe charged into the converter contains about 0.6% Cr and is blown until the P is reduced to 0.1%, deoxidised with ferromanganese, and treated with the required quantity of ferrochromium. A. R. P.

Manufacture of iron-chromium-nickel alloys. ELECTRO METALLURG. Co., Assees. of F. M. BECKET (B.P. 377,519, 2.11.31. U.S., 18.11.30).—Non-rusting Cr-Ni steels are prepared by bessemerising a mixture of Ni matte, high-C ferrochromium, CaO, and CaF<sub>2</sub> until the S is completely removed and the product contains 1-2% C, adding the requisite amount of Fe, and continuing blowing with O<sub>2</sub> until the C is reduced to the desired amount. A. R. P.

[Nickel-chromium grey] cast iron. BRIT. CAST IRON RES. ASSOC., A. L. NORBURY, and E. MORGAN (B.P. 378,508, 14.5.31).—The alloy contains 4-8% Si, 4-20%Ni, and  $\gg 10\%$  Cr. Readily machinable alloys contain 18-20% Ni, 0.2% Cr, and 4-7% Si, and heat-resisting but not readily machinable alloys 4-18% Ni, 2-10%Cr, and 4-8% Si (cf. B.P. 323,076; B., 1930, 197). A. R. P.

Manufacture of articles [capable of withstanding high pressures] from steel alloys. F. KRUPP A.-G. (B.P. 377,011, 18.9.31. Ger., 20.9.30).—The alloy used consists of Fe with 0.08-0.3% C, 2-4.5% Cr, and 0.15-1% Mo or 0.3-2% W, with or without > 1.9% Ni. A. R. P.

Manufacture of strain-hardened manganese steel [castings]. F. A. FAHRENWALD, Assr. to AMER. MANGANESE STEEL CO. (U.S.P. 1,838,815, 29.12.31. Appl., 13.12.26).—The steel is cast with a no. of equispaced knob-like projections, heat-treated to convert it into the austenitic state, and then hammered or pressed until the projections are flattened out or compressed into the surface of the casting; surface layers having a hardness of > 700 Brinell can be obtained by suitable treatment in this way. A. R. P.

Magnetic [nickel-iron] alloys. INTERNAT. GEN. ELECTRIC CO. INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 377,969, 30.4.31. Ger., 30.4.30).—In the final rolling stage, a reduction of 40-50% is effected either (a) at a temp. at which recrystallisation is not complete, e.g., at  $600-900^\circ$  for the 50% Ni alloy, or (b) at room temp. followed by annealing for 1 hr. at  $600-800^\circ$ .

[Magnetic, non-rusting] alloy steels. A. E. WHITE. From FIRTH-STERLING STEEL Co. (B.P. 378,478, 13.3.31). —Steel containing < 1% C, > 6% Cr, 0.5—20% Ni, and 2.5—8% Al is rendered martensitic by heat-treatment at 950—1230°; 8:18 Ni-Cr steel with 3—5% Al is specifically claimed. A. R. P.

Stable-surface alloy steel. R. P. DE VRIES, ASST. to LUDLUM STEEL CO. (U.S.P. 1,839,089, 29.12.31. Appl., 20.1.26).—An alloy of Fe with 2—7% Si, 0.5—6% Cu, and >1.5% Mn, with or without 0.1-1% C, 0.1-5% Ti, and 0.5-5% Mo or W, is claimed. A. R. P.

Iron-titanium alloys. Stainless iron and steel alloys. F. KRUPP A.-G. (B.P. 375,776 and Addn. B.P. 375,777, 19.3.31. Ger., [A] 26.6.30, [B] 22.1.31).—(A) Mild steel containing  $\geq 0.5\%$  C, or (B) non-staining steel containing 6—40% Cr, 25—0.5% Ni, and one or more of the following: Mn, W, Mo, V, or Co, is alloyed with 0.1—8% Si and 0.1—10% Ti, annealed at 700—1300°, and either cooled slowly or cooled rapidly and tempered at 200—1000° to cause pptn.-hardening.

A. R. P. Iron-boron alloys. Stainless iron and steel alloys. F. KRUPP A.-G. (B.P. 375,792 and Addn. B.P. 375,793, 19.3.31. Ger., [A] 26.6.30, [B] 21.1.31).—(A) Steel containing  $\geq 0.5\%$  C and 0.01-4% B, with or without Ni, Cr, Mn, or Si, is annealed at 700—1300° until the B is in solid solution, then either cooled very slowly or cooled quickly and tempered at 200—1000° to produce pptn.-hardening. (B) The process is applied to nonstaining steels containing 6—40% Cr and 25—0.5% Ni, with or without Si, Mn, W, Mo, V, or Co; about 0.5%B is required to produce a good hardening effect.

A. R. P. Casting of [metal] ingots. VEREIN. ALUMINIUM-WERKE A.-G., and E. ROTH (B.P. 377,611, 7.3.32. Ger., 15.12.31. Addn. to B.P. 367,615; B., 1932, 389).— The mould is moved horizontally during solidification to prevent formation of large stalk-like crystals. A. R. P.

Copper-refining methods, apparatus, and products. AMER. METAL Co., LTD., Assees. of R. P. HEUER (B.P. 378,008, 1.5.31. U.S., 5.6.30).—Cu cathodes are heated above 800° in an atm. containing  $H_2$  to remove all S, then melted in a slightly oxidising atm. to remove  $H_2$ , and finally tapped into an electric furnace heated by resistance, the current being supplied by C electrodes dipping into the metal. In this furnace Cu<sub>2</sub>O in the Cu is reduced by forcing granular C below the metal surface, and the refined metal is cast into moulds in an atm. containing CO. A. R. P.

Manufacture of copper alloys. FURUKAWA DENKI KOGYO KABUSHIKI KAISHA (B.P. 378,388, 4.5.31. Jap., 10.5.30).—Cu alloys containing small quantities of Ni, Si, and Cd are hot-rolled, quenched, and then alternately drawn at 20° and tempered at 600—250°, these last two processes being repeated at least twice.

A. R. P.

Manufacture of bearing brass. G. BÜHLER (B.P. 379,155, 23,11.31).—A bronze tube containing 0.2—0.4% P is cut into sections which are subjected to alternate annealing and drawing operations until complete homogeneity of the crystals is obtained.

A. R. P.

Coalescing of metals [brass or bronze scrap]. COPPER DEOXIDATION CORP. (B.P. 377,640, 10.6.32. U.S., 10.6.31).—The scrap is pickled in dil.  $H_2SO_4$ containing CuSO<sub>4</sub> to deposit a film of Cu on the surface and, after washing and drying, heated in a non-oxidising atm. to a temp. between the recrystallisation temp. and the m.p.; pressure is then applied to produce coalescence of the fragments by grain growth. A. R. P.

A. R. P.

Reduction of zinciferous materials. E. H. BUNCE, Assr. to NEW JERSEY ZINC CO. (U.S.P. 1,838,296, 29.12.31. Appl., 8.8.28).—Briquettes of Zn ore (free from S) and carbonaceous reducing agent are fed direct from the screen of the briquetting machine on to a sloping channel through which they pass continuously to a vertical drying, preheating, and coking shaft (A). At the bottom of A they pass over a screen in a second sloping channel by which they are passed into the top of the reducing shaft in which they are heated to produce Zn vapour and CO. A. R. P.

Zinc-base die-casting alloy. New JERSEY ZINC Co., Assees of W. MCG. PEIRCE and E. A. ANDERSON (B.P. 376,534, 27.8.31. U.S., 29.8.30).—The alloy comprises Zn with < 0.02% of impurity, 2—10 (4)% Al, and 2—6 (3)% Cu. A. R. P.

Zinc-base die-casting alloy. NEW JERSEY ZINC Co. (B.P. 378,645, 17.8.31. U.S., 19.8.30).—The alloy comprises Zn (containing  $\geq 0.003\%$  Pb,  $\geq 0.003\%$ Cd, and  $\geq 0.001\%$  Sn) with 2—10 (4)% Al, 0.01— 0.3 (0.1)% Mg, and 0.05—2 (1)% Cu. [Stat. ref.] A. R. P.

**Treatment of nickel-bearing materials [mattes].** INTERNAT. NICKEL Co., INC., Assees. of R. L. PEEK and C. A. KNITTEL (B.P. 379,322, 24.4.31. U.S., 15.8.30). —Ni matte with 74—76% Ni,  $1\cdot5$ — $2\cdot5\%$  Cu, and  $0\cdot2$ — $0\cdot5\%$  Fe is desulphurised in a down-draught sintering apparatus until it contains < 0.6% S. About 5% of the roasted product is finely ground and reduced to metal sponge with water-gas at  $< 500^{\circ}$  and the remainder is smelted with coke and cast into crude Ni anodes. The latter are refined by electrolysis in a NiSO<sub>4</sub> solution, which is purified from time to time by treatment with the sponge; this ppts. the Cu and reduces the  $p_{\rm H}$  to  $5\cdot6$ , at which point the Fe is pptd. by blowing air through the solution, which is then returned to the tanks. [Stat. ref.] A. R. P.

**Recovering or refining lead and its alloys.** P. W. DAVIS (B.P. 377,298, 25.4.31).—Scrap battery plates are melted in a reverberatory furnace to give relatively pure Pb, and a rich antimonial PbO which is reduced separately to antimonial Pb. Similarly, scrap Pb-bearing metal may be intimately mixed with PbO and the mixture melted to obtain relatively pure Pb, and a dross which can be reduced to an alloy with a relatively low Pb content. A. R. P.

Production of homogeneous metalliform carbonitrides and superficial nitrification of tungsten. R. R. WALTER (U.S.P. 1,803,276, 28.4.31. Appl., 6.3.28. Ger., 13.5.27).—W powder is heated in a mixture of NH<sub>3</sub> and CH<sub>4</sub> or a volatile hydrocarbon vapour.

A. R. P.

Sintered hard metallic [tungsten carbide] alloys. C. E. EVERY-CLAYTON. From F. KRUPP A.-G. (B.P. 376,912, 5.6.31).—Sintered mixtures of Co and WC with 0.01—30% of V, Ta, or Nb carbides are claimed, e.g., 94% WC, 4% Co, and 2% V<sub>4</sub>C<sub>3</sub>. A. R. P.

Reduction of metal sulphides [e.g., molybdenite]. A. A. JOHNSON (A. JOHNSON & CO.), B. M. S. KALLING, and C. VON DELWIG (B.P. 379,331, 22.5.31).—A mixture of MoS<sub>2</sub> with CaO, MgO, or BaO and sufficient C to combine with the O is passed through a rotating furnace at about 900°, whereby a mixture of Mo and CaS, MgS, or BaS is obtained from which the metal is isolated by treatment with dil. HCl. A. R. P.

Manufacture of eyelets of metals of the tungstenmolybdenum class. W. F. DESTER, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,838,749, 29.12.31. Appl., 23.9.29).—Mo or W is rolled into sheet of such a thickness that it has a tensile strength of 100,000 lb. per sq. in. when determined on a strip  $12 \times 1$  in., and is then pressed into shape with suitable dies after annealing at 200° before every pressing operation. A. R. P.

Manufacture of hard [tantalum-nickel] alloys. FANSTEEL PRODUCTS Co., INC. (B.P. 375,854, 31.3.31. U.S., 11.10.30).—A mixture of 95— $93 \cdot 5\%$  Ta powder with 5— $6 \cdot 2\%$  of lampblack preheated at 1500° is heated slowly to 2000° until reaction ceases. The product is ground finely in a ball mill and again heated to 2000° in a Ta vessel. The resulting carbide is ground in naphtha in a Ni ball mill until the powder contains 3—15% Ni powder ; it is then dried, compressed into shape, and heated at 1300—1400° in H<sub>2</sub>. The lampblack may be replaced by B to obtain a Ta boride alloy. A. R. P.

[Hard] metallic alloys. K. HONDA and T. KASE (B.P. 378,055, 3.3.31).—Claim is made for alloys containing 35-70% Ta,  $44\cdot5-5\%$  Mo, and  $20\cdot5-50\%$ Cr; part or all of the Ta may be replaced by W (which, however, must not exceed 50%) and part of the Mo and/or Cr by 2-10% V. In addition,  $0\cdot1-2\%$  C,  $0\cdot1-5\%$  Ni,  $0\cdot1-15\%$  Fe, and  $0\cdot1-4\%$  Mn may also be present. [Stat. ref.] A. R. P

Hard metallic [boron] alloys for implements and tools. F KRUPP A.-G. (B.P. 376,836, 17.4.31. Ger., 8.7.30).—Claim is made for sintered compressed mixtures of B with a smaller proportion of a metal or alloy having m.p.  $> 1000^{\circ}$ , e.g., a mixture of 85% B and 15% Zr sintered at 2100°. A. R. P

Manufacture of sintered hard metallic alloys [containing boron carbide]. F. KRUPP A.-G. (B.P. 378,484, 4.5.31. Ger., 31.7.30)—B<sub>6</sub>C made by heating amorphous B with C in H<sub>2</sub> at 1700° is mixed with 5% Si and  $2 \cdot 5\%$  Fe powder and, after degassing at 1350° in vac., the mixture is pressed into shapes which are sintered in H<sub>2</sub> at 2000°, worked to the desired final shape, and again heated in H<sub>2</sub> at 2200—2300°. A. R. P.

Production of non-porous [aluminium] metal castings. E. STRASSER (B.P. 377,679, 21.4.31).—The molten metal or alloy is treated with  $H_2$  and a small quantity of SiH<sub>4</sub>,  $B_4H_{10}$ , ZrH<sub>4</sub>, or SbH<sub>3</sub>, preferably under a MgF<sub>2</sub> flux. A. R. P.

**Rendering [aluminium] vessels oilproof.** BRIT. THOMSON-HOUSTON CO., LTD., Assees. of J. R. BROSSMAN (B.P. 375,012, 17.4.31. U.S., 17.4.30).—The vessel is thoroughly scoured, filled with a resin of the "alkyd" type, heated at 90—110° for 10—24 hr. under 90 lb. per sq. in., drained, and baked at 90—110° for 10—12 hr. to harden the resin in the pores. A. R. P.

Aluminium alloy. H. C. HALL and T. F. BRADBUBY (B.P. 379,004, 4.6.31. Addn. to B.P. 323,353; B., 1930, 198).—The alloy consists of Al with 3.5—5% Cu,

Production of aluminium-magnesium alloys having a high resistance to corrosion. I. G. FARB-ENIND. A.-G. (B.P. 374,766, 13.11.31. Ger., 23.5.31).— Alloys of Al with 3—15% Mg, with or without other metals, are prepared by melting the Al under the saline fluxes claimed in B.P. 182,948, 219,287, and 287,360 (B., 1922, 715A; 1925, 75; 1928, 373) and then adding the alloying constituents. A. R. P.

Treatment of surfaces of aluminium or its alloys. VEREIN. ALUMINIUM-WERKE A.-G. (B.P. 377,385, 18.6.31. Ger., 2.2.31).—The articles are anodically oxidised in any of the known baths, using a.c. with a frequency > 50 periods per sec., and then painted or coloured in known manner. A. R. P.

Removal of oxides and gases from molten aluminium and its alloys. K. SCHMIDT GES.M.B.H. (B.P. 377,079, 14.12.31. Ger., 17.1.31).—The molten metal is treated with a mixture of an alkali bifluoride, preferably  $NH_4HF_2$ , and  $CCl_4$ . The latter can be wholly or partly replaced by  $SbCl_5$  and/or  $MnCl_2$ , and, when Si and Zn are not deleterious to the properties of the alloy,  $SiCl_4$  and/or  $ZnCl_2$  may be added to the mixture. The liquid purifying agents are preferably used adsorbed on charcoal powder. A. R. P.

Protection of magnesium and its alloys against corrosion. G. D. BENGOUGH and L. WHITBY (B.P. 378,916, 19.5.31).—The metal is dipped for a short time in aq.  $H_2SeO_3$ , with or without 0.1-0.5% NaCl, or in a mixture of Na<sub>2</sub>SeO<sub>3</sub> and  $H_3PO_4$ . A. R. P.

Obtaining or concentrating germanium. V. M. GOLDSCHMIDT (B.P. 378,017, 22.4.31. Ger., 27.3.31).— The material, e.g., ores or by-products containing small quantities of Ge, is heated with S,  $FeS_2$ ,  $H_2S$ , or other S-bearing material at 400—1200° (900—1000°) in a reducing atm., whereby GeS with some GeO and Ge distil and a sublimate containing up to 60% Ge is obtained. A. R. P.

[Cæsium] metal alloys. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of D. H. BROPHY (B.P. 377,469, 25.8.31. U.S., 26.8.30).—Cs is heated at 50—80° (60°) under a layer of "Nujol" or other hydrocarbon oil and small pieces of a metal, *e.g.*, Pb, Sn, Zn, Cd, Ag, Sr, Ba, or Mg, which alloy with the Cs but give it up again in vac., are added with stirring until a uniform, powdered alloy is obtained. The powder is washed with light petroleum and stored under CCl<sub>4</sub>. A. R. P.

Apparatus for casting alkaline-earth metals. H. S. COOPER, Assr. to KEMET LABS. Co., INC. (U.S.P. 1,834,049, 1.12.31. Appl., 10.12.28).—The apparatus comprises an Fe or Ni crucible (A) above which is clamped a cover carrying a slightly conical mould (B)closed at its lower end with a removable screw plug. A and B together form a melting chamber from which the air may be exhausted and replaced by A, He, or other inert gas, and are mounted on trunnions so that by inverting A the molten metal is cast gently into B while the impurities remain on the surface of A.

A. R. P.

Separation of minerals. A. NAGELVOORT (U.S.P. 1,839,117, 29.12.31. Appl., 26.9.28).—The crushed ore is elutriated to separate fines and the coarser material, graded if necessary, is passed wet through a tank con taining a layer of a heavy org. liquid  $(e.g., CBr_4, C_2H_2Br_4)$  below a layer of  $H_2O$ ; the heavier minerals sink into the heavy layer, whilst the lighter minerals are carried away by a current of  $H_2O$ . A. R. P.

Concentration of ores by flotation. H. A. LUBS and A. L. Fox, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,839,155, 29.12.31. Appl., 13.12.27).— The use of compounds of the type R·CS·SH as selective flotation agents for Cu sulphides is claimed. Dithiofuroic and dithiobenzoic acids and their salts are specifically mentioned. A. R. P.

Concentration of [oxidised copper] minerals by flotation. MINERALS SEPARATION, LTD., and S. TUCKER (B.P. 378,063, 27.4.31).—In the flotation of oxidised ores, using Na<sub>2</sub>S as a conditioning agent and Na<sub>2</sub>SiO<sub>3</sub> as a deflocculating agent for the gangue, the amount of the latter used is just sufficient to effect deflocculation, *e.g.*, about 0.1 lb. per ton instead of the usual > 1 lb. per ton. A. R. P.

Cleaning and pickling of metals. W. L. SEMON, Assr. to B. F. GOODRICH CO. (U.S.P. 1,830,566, 3.11.31. Appl., 5.12.28)—The *N*-alkylthiocarbamides and 2-iminothiazolidines obtained by the action of alkyl halides and alkylene dihalides, respectively, on thiocarbamides, are effective inhibitors. Examples are 2-phenylimino-3phenyl- and 2-o-tolylimino-3-o-tolyl-thiazolidines.

C. H.

Hydrogenation catalysts. E. B. MAXTED (B.P. 378,943, 20.5.31).—The catalysts are prepared by the surface oxidation of Cu-Cr or Cr-Zn alloys. They may be used for hydrogenating vegetable and animal fats,  $C_6H_6$ , etc. by means of  $H_2$  at 150°/200 atm. and for the conversion of COMe<sub>2</sub> into Pr<sup>3</sup>OH. A. R. P.

Extending the life of grates in agglomerating plants. VEREIN. STAHLWERKE A.-G. (B.P. 379,057, 20.7.31. Ger., 18.9.30).—The grate is covered with a thin paste of clay and Ca(OH)<sub>2</sub> to prevent action of S on the metal. A. R. P.

Coating or plating metallic surfaces [with silver] without the aid of electrical current or heat. F. W. V. FITZGERALD (B.P. 378,874, 17.3.31).-A plating liquid (A) is prepared by mixing 1000 g. of fuller's earth, 150 g. of Na2B407,10H20, 3 g. of AgNO3, and 300 c.c. of aq. NH<sub>3</sub> with sufficient H<sub>2</sub>O to form a thin cream. A coating paste (B) is obtained by dissolving 350 g. of AgCl in a solution of 5 kg. of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>,5H<sub>2</sub>O and 150-450 g. of K H tartrate in 5 litres of H<sub>2</sub>O, and adding 7 kg. of Na<sub>2</sub>CO<sub>3</sub> and 20 c.c. of pine or sweetalmond oil with sufficient fuller's earth, talc, or CaCO<sub>3</sub> to make a thick paste; preferably 35 g. of  $CrO_3$  and sufficient H<sub>2</sub>O<sub>2</sub> to form a blue colour are also added to harden the Ag coating obtained. To plate a metal, a cloth is moistened with A, dipped into  $\overline{B}$ , and rubbed on the cleaned metal surface until the desired Ag coating is obtained. A. R. P.

Securing metal to porcelain. BRIT. THOMSON-HOUSTON CO., LTD., and R. ADKINS (B.P. 378,974, 22.5.31).—The porcelain is painted with a paste of 75% of pptd. Ag and 25% of Bi subnitrate in turpentine, and fired at  $800^{\circ}$  to produce a Ag film, which is Cu-plated and tinned for soldering the metal to the porcelain. A. R. P.

**Production of coated filler [welding rods].** J. B. GREEN, ASST. to F. M. WARDEN (U.S.P. 1,839,961, 5.1.32. Appl., 14.5.28).—Filler materials (welding rods) for arc welding and cutting are dipped in a mixture of dextrin, Na<sub>2</sub>CO<sub>3</sub>, CaCO<sub>3</sub>, and H<sub>2</sub>O, and after draining are dried quickly by warm air. The treated rods give extreme penetration and sound welds at high c.d. S. S. W.

Apparatus for electrolytic deposition of metals. Soc. ANON. WALDBERG (B.P. 375,897, 8.4.31. Fr., 8.4.30. Addn. to B.P. 362,191; B., 1932, 351).—The apparatus claimed previously is modified by the provision below the vat of several electromagnets having unsymmetrically distributed polarities or pole surfaces of different sizes, and by the introduction of means for automatically switching the current on and off as the anode enters and leaves the vat. A. R. P.

[Chromium-]electroplating apparatus. C. G. FINK and C. H. ELDRIDGE (U.S.P. 1,838,666, 29.12.31. Appl., 12.11.27).—Apparatus for obtaining Cr-plate free from occluded  $H_2$  comprises an electrolytic tank with a hood mounted above the position of the cathodes therein, and means for intermittently raising the cathodes into the hood, closing it, and reducing the pressure in it to cause the  $H_2$  to be evolved from the deposited metal. A. R. P.

Electrolytic deposition of iron. E. KELSEN and E. AUSNIT (B.P. 378,962, 21.5.31).—The electrolyte contains Fe<sup>..</sup> 60, Na<sup>.</sup> 49, Ba<sup>..</sup> 13, and Cl' 202 g. per litre ( $p_{\rm H}$  3) and is caused to circulate through the cell at 90°, while the c.d. is gradually raised from 2 to 5 amp. per sq. dm. over a period of 30 min. In this way curling of the deposit by excessive adsorption of H<sub>2</sub> is avoided. A. R. P.

Electrically coating aluminium. W. J. TENNANT. From ALUMINIUM COLORS, INC. (B.P. 378,521, 15.5.31). —The metal is treated anodically in 60-77% H<sub>2</sub>SO<sub>4</sub> containing glycerin or a sulphonated phenol, and the resulting oxide film is coloured by the pptn. of a pigment therein by chemical reaction. A. R. P.

Electrolytic manufacture of beryllium. BERYL-LIUM DEVELOPMENT CORP. (B.P. 377,858, 2.11.31. U.S., 3.1.31).—A fused mixture of alkali chlorides and BeCl<sub>2</sub> is electrolysed to obtain Be flakes at the cathode ; the Cl<sub>2</sub> generated at the anode is passed over a heated mixture of BeO and C to obtain BeCl<sub>2</sub> vapour, which is condensed in the electrolyte, thereby regenerating it. A, R. P.

Moulds for casting ingots [for railway wheels etc.]. B. MATUSCHKA (B.P. 380,515, 19.6.31).

Production of rustless and stainless knives [built up of layers of different hardness]. W. E. EVANS. From DEUTS. EDELSTAHLWERKE A.-G. (B.P. 380,336, 19.1.32).

Production of imitation matt-etching on metal. VEREIN. GRABA- U. SCHREGERWERKE (A.-G. F. CARTON-NAGENIND.) (B.P. 378,744, 10.12.31. Ger., 11.12.30). Surface-treatment [hammering and rolling] of metal for the removal of scale, rust, and the like, J. H. SOUTHERN (B.P. 376,261, 30.3. and 12.5.31).

Xanthates.—See III. Catalyst for  $NH_3$  oxidation. Welding flux.—See VII. Sheet-Fe enamels.—See VIII. Slag treatment.—See IX. Sheet-Fe shells for furnaces. Lead-in wires.—See XI. Securing rubber to metal.—See XIV.

# XI.—ELECTROTECHNICS.

Electric furnaces for the chemical industry. E. F. RUSS (Chem. Fabr., 1932, 5, 353-356).—A no. of types of electric resistance furnaces, including rotary furnaces, for heating solids, liquids, and gases are described and illustrated. C. I.

Electrolytic manganese dioxide. G. W. NICHOLS (Trans. Electrochem. Soc., 1932, 62, 295-304).-Powdered rhodochrosite (MnCO<sub>3</sub>) is leached with hot dil. H.SO, (spent electrolyte) in a countercurrent system. and the neutral liquor after separation from insol. residue is aërated in the presence of CaCO<sub>2</sub> to remove Fe. The purified MnSO<sub>4</sub> solution, mixed with an equal vol. of spent electrolyte, is electrolysed at 90° and 6-10 amp. per sq. ft. between antimonial Pb anodes and graphite cathodes in a Pb-lined cell, the MnSO4 concn. falling from 150 to 50 g. per litre in passing through the cell. The dense, coherent coating which forms on the anode is stripped, and after drying at 110° is 85-90% MnO2. In experimental plant operation the current efficiency was normally 70-80% and the energy consumption < 1.92 kw.-hr. per kg. H. J. T. E.

Air-depolarised primary cell with caustic alkali electrolyte. G. W. HEISE and E. A. SCHUMACHER (Trans. Electrochem. Soc., 1932, 62, 337–344).— Factors affecting the operation of cells of the type Zn|NaOH solution|C (air) are discussed. The vol. of electrolyte for a cell of given amp.-hr. capacity can be reduced by addition of CaO, which regenerates alkali from NaHZnO<sub>2</sub> with formation of Ca(HZnO<sub>2</sub>)<sub>2</sub>,4H<sub>2</sub>O, which crystallises out. The construction and operating characteristics of a 600 amp.-hr. cell with a normal discharge rate of 0.5—0.65 amp. are described. The cell is kept dry and hermetically sealed until required for use. H. J. T. E.

Properties of low- $p_{\rm H}$  nickel-plating baths. I. Throwing power, cathode current efficiency, and conductivity. L. C. FLOWERS and J. C. WARNER (Trans. Electrochem. Soc., 1932, 62, 323-335).-The throwing power of low- $p_{\rm H}$  baths increases markedly with c.d., whereas that of high- $p_{\rm H}$  baths decreases somewhat, the val. for the two types approaching a common limit. Rise of temp. or increase of Ni content of low- $p_{\rm H}$  baths improves the throwing power, whilst that of high- $p_{\rm H}$ baths is scarcely affected. With high c.d., temp., and Ni content the throwing power of low- $p_{\rm H}$  baths is not much below that of high- $p_{\rm H}$  solutions. Other conditions being const., cathodic current efficiency increases with concn. of solution, especially for low- $p_{\rm H}$  baths. Solutions for which the efficiency decreases greatly at low c.d. exhibit poor throwing power. The conductivity of Ni-plating solutions increases linearly with temp.

between 25° and 70°, but is very little affected by a change of  $p_{\rm H}$  from 5.6 to 1.9. H. J. T. E.

Theory and practical applications of the highfrequency [electric] furnace. W. ESMARCH (Z. Elektrochem., 1932, 38, 812-825).

Prep. of  $MnO_2$ .—See VII. Patina development on Cu. Resistance alloys. Reproducing macrostructure of metals. Cr-, Cd-, Zn-, and Zn-Cdplate. Se and Te in electrolytic Cu.—See X. Pasteurisation of milk.—See XIX.

See also A., Oct., 989, Electrodeposited Ag-Cd. 999,  $Cl_2$ -resistant Ag-Pb electrodes. 1005, Codeposition of Pb and Bi. Prep. of formates from  $CO_2$ . Electro-org. reductions and oxidations. 1011, Separation of Cu and Pb.

# PATENTS.

Laminated sheet-iron shells for electric-induction furnaces not having a closed iron circuit. ALLMÄNNA SVENSKA ELEKTRISKA AKTIEB. (B.P. 376,660, 25.1.32. Swed., 2.1.32).—In shells formed of laminated sheet-Fe bundles surrounding cylindrical induction furnaces, the sides of the bundles are screened along the places of interruption by bundles of insulated, enamelcoated, Fe wire the longitudinal direction of which coincides with that of the plates. A. R. P.

Electric furnace resistor. J. KELLEHER, ASST. to HARPER ELECTRIC FURNACE CORP. (U.S.P. 1,841,537, 19.1.32. Appl., 27.11.25).—A zigzag graphite resistor and the inner parts of its conducting terminals are coated with readily fusible material without filling the slots between sections of the resistor; a mixture containing coarse and fine[SiC and borax is moulded around the coated resistor and terminals, and the whole is heated electrically to a white heat by current flowing in the resistor. J. S. G. T.

[Electric] heaters for fluids or solids. E. WHITBY, I. A. D. PEDLER, E. J. SELSTON, L. R. LESTER, and A. J. NEWMAN (B.P. 378,886, 18.3.31).—An electrical H<sub>2</sub>O heater controlled thermostatically is described.

B. M. V.

Lead-in wires for incandescence lamps, radio tubes, and similar devices. AMER. BRASS Co., Assees. of H. C. JENNISON (B.P. 379,034, 23.6.31. U.S., 6.3.31).—A deoxidised Cu wire containing 0.001—0.2 (0.075)% Mn is claimed. A. R. P.

Highly-emissive thermionic cathode. RADIO-A.-G. D. S. LOEWE, H. BENJAMIN, and E. EHRIG (B.P. 379,702, 30.5.31. Ger., 31.5.30).—Highly-emissive material, e.g., Ba, is deposited from vapour upon a core, e.g., of W, Pt, or Ni-Cr, coated with a definite thickness of conducting material, e.g., NaOH or KOH, by being drawn at a definite speed through a melt of the material. J. S. G. T.

Cathode for thermionic devices. A. McL. NICOL-SON, ASST. to COMMUNICATION PATENTS, INC. (U.S.P. 1,840,789, 12.1.32. Appl., 10.12.26).—A core, e.g., of Pt or Pt-Ir, coated with a mixture of an alkaline-earth oxide and Mn oxide is claimed. J. S. G. T.

Electrode for gaseous-discharge devices. C. H. THOMAS, ASST. to WESTINGHOUSE LAMP CO. (U.S.P. 1,842,215, 19.1.32. Appl., 28.6.30).—The recessed surface of a hollow refractory electrode containing Th is coated with activated alkaline-earth metal compounds, *e.g.*, BaO. J. S. G. T.

Electric-discharge devices [neon lamps]. GEN. ELECTRIC CO., LTD., H. G. JENKINS, and J. W. RYDE (B.P. 378,178, 15.7.31).—A small, hollow, Fe or Ni cylinder closed at one end is coated with a suspension of  $Ba(NO_3)_2$  in EtOH, then heated at 750° in vac. for 30 min. to cause a reaction to occur between the metal and salt. The electrode is then mounted in the lamp bulb and degassed by heating in vac. at 1000° by means of eddy currents; the bulb is finally filled with Ne at 5 mm. and run with a c.d. of 30—100 milliamp. per sq. cm. of cathode glow. A. R. P.

Electric-discharge devices. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of H. W. JACKSON (B.P. 378,076, 6.5.31. U.S., 29.5.30).—One of the electrodes comprises a hollow metal cap containing a readily decomposable compound of an alkali or alkaline-earth metal, e.g.,  $CsN_3$ . After evacuating the tube, the electrode is heated to decompose the compound, and any gas liberated is removed. The tube is further heated in vac. at 325° until any Cs deposited on the walls is removed; it can then be filled with Ne and He. [Stat. ref.] A. R. P.

Electron-emitting device and its preparation. M. N. RICH and E. A. LEDERER, ASSTS. to WESTINGHOUSE LAMP Co. (U.S.P. 1,842,203, 19.1.32. Appl., 19.5.26).— Activated thoriated W cathodes are heated at 1500— 2000° in  $H_2$  to remove C and WC and then at 2850° *in vacuo* to decompose Th compounds. J. S. G. T.

Photoelectric devices. N. V. PHILIPS' GLOEILAMP ENFABR. (B.P. 379,788 and Addn. B.P. 379,893, [A] 18.6.31. [B] 14.10.31. Ger., [A] 18.9.30, [B] 6.12.30).-(A) In devices of the types described in B.P. 378,451 and 378,457 (B., 1932, 945), the oxidised surface of a conducting electrode, e.g., of Zr, and the photoelectric electrode, e.g., of Cs, are arranged in contact with the opposite faces of an insulating or semi-conducting material, e.g., metal gauze, extending over the whole surface of the photoelectric electrode [Stat. ref.] (B) A device, as described under (A) and in which fluorescent material may be added to the insulating or semi-conducting separator, is arranged with electron-emitting material, more especially radioactive material emitting  $\beta$ -rays, in a container so that the electrons emitted travel to the photoelectric electrode. J. S. G. T.

**Electrolytic cell.** N. STATHAM and T. G. LEEK, Assrs. to WEST VIRGINIA PULP & PAPER Co. (U.S.P. 1,842,296, 19.1.32. Appl., 30.10.29).—A perforated, cylindrical, steel cathode with conical closures at its top and bottom respectively provided with a gas- and a liquid-discharge outlet, is covered with a porous asbestos diaphragm and surrounded by a no. of graphitebar anodes arranged within a container of vitreous material resistant to  $Cl_2$  J. S. G. T.

Electrolytic cell [for sodium chloride]. La F. D. VOYCE, ASST. to WESTVACO CHLORINE PRODUCTS INC. (U.S.P. 1,842,703, 26.1.32. Appl., 14.9.29).-- An orificed, cylindrical, metal shell (A), serving as cathode, rests upon the bottom of a surrounding, opentopped, cylindrical casing (B) forming a cathode chamber and is laterally spaced from the walls of B. Anodes are arranged in an interior chamber defined by a diaphragm within A and are supported by a 2-part circular cover fitting gastight inside the top edge of B and attached to A. J. S. G. T.

Manufacture of depolarisers for galvanic cells. N. AHLMANN, Assr. to F. L. SMIDTH & Co. (U.S.P. 1,840,296, 12.1.32. Appl., 30.10.30. Denm., 4.12.29).— A mixture of graphite (A) and MnO<sub>2</sub> (B) is ground and air-sifted so that the grain size of A is > that of B.

J. S. G. T.

[Electrolytic] apparatus for generation of fuel [oxygen-hydrogen mixtures] for internal-combustion engines. J. A. G. BAKER (B.P. 378,005, 30.4.31).—The apparatus comprises a cell containing a no. of positive and negative plates arranged alternately and insulated from one another, a space in the cell casing above the liquid for storing gas, and a valve-restricted tube communicating with a chamber in which air may be mixed with the gas before it passes to the cylinders. [Stat. ref.] A. R. P.

Insulating material for electric conductors. INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS GES. (B.P. 379,918, 17.11.31. Ger., 17.11.30).—Wire wrapped with paper or cotton is drawn through synthetic resin, other than a citric acid product, together with, if desired, varnish and fillers, and heated at 400° for 5—15 sec. J. S. G. T.

Resistance device and insulator. C. O. TERWIL-LIGER, Assr. to WARD LEONARD ELECTRIC CO. (U.S.P. 1,842,433, 26.1.32. Appl., 28.12.28).—A support containing TiO<sub>2</sub>, apatite, and flint as principal ingredients, and a resistor are coated with a fused insulator.

J. S. G. T.

Production of magnetic bodies [cores for loading coils etc.]. G. W. ELMEN, ASST. to BELL TELEPHONE LABS., INC. (U.S.P. 1,840,352, 12.1.32. Appl., 22.1.31). —A mixture of magnetic material, e.g., Ni-Fe, and insulating material, e.g., chromic acid, Na silicate, and talc, is compressed and heat-treated at 500° in H<sub>2</sub>.

J. S. G. T.

Galvanic batteries. OLDHAM & SON, LTD., and H. HOLT (B.P. 380,561, 5.8.31).

Mercury-vapour rectifiers. BRIT. THOMSON-HOUSTON CO., LTD., and F. P. WHITAKER (B.P. 380,097 and 380,142, 11.3.31). A.-G. BROWN, BOVERI & CO. (B.P. 380,261, 24.9.31. Switz., 30.9.30).

Electric incandescence [red] lamps [for photographic dark-rooms]. Egyesült Izzòlàmpa ès VILLAMOSSÀGI RÉSZVENYTARSASÀG (B.P. 379,854, 27.8.31. Hung., 28.8.30).

Electric plugs. J. A. BOND, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 380,128, 11.6.31).

Separation of materials.—See I. Heating bodies. Insulator support.—See VIII. Concentrating sponge Fe. Reduction of Fe ores. Magnetic alloy steels. Cu refining. Ni mattes. Recovering Pb [from battery plates]. Treating Al surfaces. Electro-deposition. Cr- and Fe-plate. Coating Al, Be. Phonograph records.—See X. Treating Soil, See XVI.

# XII.—FATS; OILS; WAXES.

Extraction by means of benzene of fat treated with a catalyst. A. SLASHCHEV (Masloboino Zhir. Delo, 1931, No. 11, 9—14).—Extraction of fats and sludges of hydrogenation plants with 96% EtOH +  $C_6H_6$  (7:5), in preference to acid or alkali which causes hydrolysis and loss of Ni, is satisfactory, but the cost is high and the presence of H<sub>2</sub>O increases the loss of solvent. Cold extraction 4 times, each with 4 vols. of gasoline for 1 hr. followed by filtration, is satisfactory on the laboratory scale ; the residue contained only 0.5—1% of fat.

CH. ABS.

Conditions of formation of *iso*-acids in the hydrogenation of fats. R. GUTMANN (Masloboino Zhir. Delo, 1931, No. 8—9, 32—36).—Sunflower-seed oil and oleomargarine were hydrogenated in presence of Ni; the amount of unsaturated solid *iso*-acids increases with the temp. and time of hydrogenation. Apparently dehydrogenation takes place at high temp. (about 300°), whilst experiments with Et oleate show that it does not occur at lower temp. At lower temp. hydrogenation takes place progressively; in this case the presence of linoleic and linolenic acids favours the formation of *iso*acids. CH. ABS.

David's method for separation of solid and liquid fatty acids. R. JUNGKUNZ (Chem. Umschau, 1932, 39, 171—173; cf. B., 1910, 1395).—The separation of mixtures of saturated and unsaturated acids (by pptn. by, and in, aq.  $NH_3$ ) is unreliable for quant. work; oleic acid alone does not give a ppt. E. L.

Determination of fatty acids in soaps loaded with clay. G. TURBIN (Masloboino Zhir. Delo, 1931, No. 10, 20–21).—The soap (5 g.) is dissolved in hot H<sub>2</sub>O, decomposed with H<sub>2</sub>SO<sub>4</sub>, and treated with saturated aq. NaCl (100—150 c.c.); the fatty acid layer is washed with saturated NaCl to neutrality (Me-orange) and the acids are dissolved in EtOH. The fatty acids retained by the clay (washed, neutral) are dissolved on the filter in EtOH and the combined alcoholic solutions titrated with 0.5N-alkali. CH. ABS.

Extraction of soap stock with benzine. A. SLASH-CHEV (Masloboino Zhir. Delo, 1932, No. 1, 46-47).--Na soaps, except that of castor oil, which is insol., form colloidal solutions in benzine. By increasing the oil concn. of the benzine solution of the soap stock to 69-71.5% (according to the room temp.) the neutral oil is completely separated from the soap and colouring matter. CH. Abs.

Determination of free [caustic] alkali in [potash] soap. G. KNIGGE (Chem. Umschau, 1932, 39, 173-174). —The accuracy of the Davidsohn modified alcohol method for K soaps (B., 1927, 18) is confirmed; access of  $CO_2$  should be avoided, and the solution should be filtered before titration, in order to get a sharp endpoint.  $K_2CO_3$  is partly sol. in a mixture of glycol and  $Pr^{\circ}OH$ , which cannot therefore be used for K soaps (cf. Poethke, B., 1932, 777). E. L.

Oxidation of drying oils. G. PETROV and T. KAS-TERINA (Masloboino Zhir. Delo, 1931, No. 10, 30-37) .--10% K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> (2-30% of oil) in HCl (1:2) was stirred with linseed oil and the oil, after washing with hot H<sub>2</sub>O, filtered through floridin. With 30% K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> the I val. changed little; the OH-acids increased from 1 to 9.5%, the acid val. from 2.0 to 3.75, sap. val. from 186 to 194.3, and the Ac val. from 7.31 to 21.1. The fatty acids, respectively treated and untreated, had I val. 152, 177.3; acid val. 183.4, 193.1; sap. val. 202.95, 200.6; Ac val. 39.7, 5.0. Oxidation with PbCrO<sub>4</sub> or BaCrO<sub>4</sub> affected the co. sts. of linseed and sunflowerseed oil only slightly. Oxidation with O2 or air (50-170°, 30-40 hr., 170-175 litres per hr.) considerably lowered the I val. (e.g., O2 at 135-140°, 175.4-100.6); the amount of OH-acids was higher, and lactone formation was indicated. Oils treated with air at 135-140° for 17 hr. dried more, and at 165-170° less, rapidly than those oxidised with K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>. Soaps from the oxidised oils were harder, but were hygroscopic and darkened on ageing. CH. ABS.

Effect of age of cottonseed oil on the Halphen reaction. T. STATHOPOULOS (Praktika, 1930, 6, 173—177; Chem. Zentr., 1932, i, 1591).—After 10 years, but not after 14 years, rancid, colourless cottonseed oil gave the Halphen reaction. Vals. for fresh and 14 year-old oil, respectively, are:  $d^{25} 0.9270, 0.9815; n 1.4742, 1.4715;$ I val. (Hübl) 102.0, 52.40; sap. val. 190.0, 225.70; Hehner val. 93.50, 94.70; acidity (oleic acid) 0.40, 17.20%; Tortelli val. 78, —. A. A. E.

Hydrogenation of soya-bean oil. A. LAPTEV (Masloboino Zhir. Delo, 1932, No. 1, 31–33).—Lecithin in the crude oil poisons the Ni catalyst. It is removed by treating the oil with 1% H<sub>2</sub>SO<sub>4</sub> (d 1.84) at room temp. and, after removal of the ppt., with NaOH (d 1.16) at 60–70°. After being washed to neutrality with H<sub>2</sub>O the oil is dried by a current of air at 100°.

CH. ABS.

Determination of m.p. from the refractive index in the hydrogenation of soya-bean oil. F. PINSKER (Masloboino Zhir. Delo, 1931, No. 1, 21-22).—A diagram is constructed from the Zeiss butyrometer reading, m.p. of refined oil, and I val. CH. Abs.

Purification of sunflower-seed oil with dilute caustic [soda]. R. HEUBLYUM and M. KLYACHKO (Masloboino Zhir. Delo, 1931, No. 11, 5—9).—Dil. NaOH  $(d \ 1\cdot 03 - 1\cdot 06)$  at a low temp. is subsequently heated at 55—60°; emulsification aids purification. CH. ABS.

Utilisation of [sunflower-seed] hulls. M. TZONEV and N. YAVNEL (Masloboino Zhir. Delo, 1931, No. 11, 22-23).—Heating at  $> 100^{\circ}$  with PhOH and mineral acid affords resinous compounds sol. in org. solvents. CH. ABS.

Vegetable oils. X. Oil of *Dipsacus fullonum*, L. S. IVANOV (Chem. Umschau, 1932, **39**, 173; cf. B., 1932, 390).—Seeds of the fullers' teazle from Suchum yielded a semi-drying oil  $(20 \cdot 9\%)$  having  $n_{D}^{cs} 1 \cdot 474$ , acid. val.  $8 \cdot 9$ , sap. val. 189, I val. (Hübl) 119—119 \cdot 7, CNS val.  $82 \cdot 1$ —  $82 \cdot 5$ , saturated acids (Bertram)  $4 \cdot 05\%$ , hexabromide test negative; linoleic acid was identified as tetrabromide. The presence of  $52 \cdot 5\%$  of monounsaturated acids (as glycerides) and  $42 \cdot 8\%$  of linoleic acid (as glycerides) is deduced from the above figures. E. L.

Peanut oil from nuts of different degrees of maturity. N. DUBLIANSKAYA (Masloboino Zhir. Delo, 1931, No. 11, 55-57).—The acid, I, sap., and ester vals. changed little, but the CNS val. (Kaufmann) was lower for oil from green nuts; hence the saturated acids decrease and the oleic acid increases with ripening. CH. ABS.

Commercial arachis oil. G. SCHUSTER (J. Pharm. Chim., 1932, [viii], 16, 236–239).—Commercial arachis oil is considered to contain saturated glycerides < 1%, triethylenic glycerides 82.6%, and monosaturated diethylenic glycerides 8.6%. These results are based on the amounts of products of the oxidation of the oil with KMnO<sub>4</sub> in COMe<sub>2</sub>, and the separation into saturated, diazeleic, and triazeleic glycerides, the two last-named by the difference in solubility of the Li salts. A. L.

Seeds and oil of the Chinese radish. N. BELIAEV (Masloboino Zhir. Delo, 1930, No. 11-12, 51-53).— The seeds of *Raphanus sativus oleiferus chinensis*, L., contain H<sub>2</sub>O 11.5, crude oil 39.1, crude protein 23.87, albumin (Barenstein) 11.9, ash 4.40, crude fibre 8.30, N-free extract 12.83%. The oil (edible, semi-drying) has d 0.9165,  $n_{20}^{20}$  70.26 (Zeiss), acid val. 2.3, sap. val. 184.9, I val. (Hübl) 109.18, flash point 248°.

CH. ABS.

Tobacco-seed oil. I. MOROZOV and A. GRASHIN (Masloboino Zhir. Delo, 1930, No. 11—12, 53—54).— The seeds contain  $H_2O$  7·36, oil 36·0%. The oil (edible, semi-drying) has d 0·9259, acid val. 2·57, sap. val. 194·6, I val. 135·46,  $n_D^{20}$  1·4756, unsaponifiable matter 1·53%. CH. ABS.

Utilisation of proteins of oil-bearing seeds as a detergent material. G. PETROV and S. DIMAKOV (Masloboino Zhir. Delo, 1931, No. 12, 36—40).—Proteins  $(51\cdot48\%)$  of sunflower-seed cake  $(6\cdot36\%)$  oil) were hydrolysed by blowing steam for 6 hr. through a mixture of finely-ground cake (20), H<sub>2</sub>O (200), 28% HCl (4), and naphthenesulphonic acids (6 pts.). The NH<sub>2</sub>-acids (yield 85%) were then washed and dried. Hydrolysis with 10% NaOH and pptn. with 25% H<sub>2</sub>SO<sub>4</sub> gave only  $45\cdot35\%$ . Solutions of the hydrolytic products with an equal quantity of Na<sub>2</sub>CO<sub>3</sub> had good detergent effects on cotton goods. CH. ABS.

Effect of the production process on the constants of certain oils. N. DUBLIANSKAYA (Masloboino Zhir. Delo, 1932, No. 1, 65—68).—Perilla, linseed, soya-bean, sunflower-seed, rape-seed, arachis, and castor oils were prepared by cold-pressing and by extraction with  $Et_2O$  or benzine. The consts. of the semi-drying oils show only slight variations; the drying oils extracted have lower I vals. and higher acidity. Castor oil extracted by benzine has a lower sap. val. owing to the incomplete solubility of ricinoleic glycerides. The extracted oils were darker than the cold-pressed oils.

CH. ABS.

Fish oils. M. BELOPOLSKI and I. MAXIMOV (Masloboino Zhir. Delo, 1931, No. 10, 43-46).—The fatty acids are brominated in Et<sub>2</sub>O or light petroleum. The yield of Br-derivatives insol. in light petroleum (those of unsaturated aliphatic acids) is const. Cod- and herring-liver oils contain at least two classes of unsaturated acids, but linoleic and linolenic acids were not found. Bromination of the fatty acids in  $\text{Et}_2O$  and in light petroleum, together with the examination of all the fractions of Br-derivatives, afford sufficiently accurate identification of fats. CH. Abs.

Combating nickel losses in the hardening of oil. L. TURBIN (Masloboino Zhir. Delo, 1930, No. 9–10, 17–18).—The pptd. NiCO<sub>3</sub> should be washed with hot  $H_2O$  free from CO<sub>2</sub> and the spent catalyst with high Ni content should be regenerated. CH. ABS.

Sulphonated oils. VIII. Composition and properties of the two components of sulphonated oils on the market. IX. Changes of ricinoleic acid and its sulphuric ester. K. WINOKUTI (Tech. Rep. Tôhoku, 1932, 10, 387–415, 416–432; cf. B., 1931, 307, 894).—VIII. [With S. IGARASI and Y. YAGI.] A detailed description is given (in English) of the method of analysis of sulphated oils, and of the work outlined in Pts. X and XI of the Japanese series (B., 1932, 648).

IX. A full account is given (in English) of the work sketched in Pt. XII of the Japanese series (B., 1932, 648). Polyricinoleic acids are chiefly formed, with small amounts only (mainly in the second case) of neutral substances (lactones, lactides, etc.) when ricinoleic acid is stored or subjected to prolonged treatment with mineral acids at various temp. and concns. The ricinoleic acid product obtained by acid hydrolysis of the sulphuric ester is considerably condensed (estolides) particularly when conc. HCl is used; only negligible amounts of neutral fatty compounds are formed. E. L.

Oxidation of higher hydrocarbons.-See III.

See also A., Oct., 988, Ricinoleic acid. 1017, Prep. of alcohols by reduction of fats. 1018. Catalytic hydrogenation of fatty oils.

#### PATENTS.

Manufacture of alkaline or ammoniacal sulphites or hyposulphites of fatty acids. R. VIDAL (B.P. 379,438, 20.6.31).—Mixed fatty acids are treated with alkaline or NH<sub>4</sub> sulphites or hyposulphites, with or without addition of caustic or carbonated alkali or  $(NH_4)_2CO_3$ ; the solid (cryst.) products are sol. in H<sub>2</sub>O and suitable for detergents. E. L.

Manufacture of homogeneous intermixtures of hydrocarbons with soap-stocks, creams, or pastes. M. HUBMAJER (B.P. 378,400, 2.4.31).—A mixture of a 5—10% glue solution and a 3% starch solution, which has been opened up by alkali, is used in the prep. of soaps containing solvent (petrol), polishing creams, etc.; the amount incorporated should correspond to  $\frac{1}{2}$ — $1\frac{1}{2}$ % of dry substance (glue + starch) on the final emulsion. E. L.

Production of hard soaps. F. W. LEFFER (B.P. 379,760, 5.6.31).—Hard, stable soaps, containing 60—70% of fatty matter, are produced by saponifying fatty acids, fats, or oils with the equiv. amount of caustic alkali at about  $125^{\circ}/2-10$  atm. for  $\frac{1}{2}-\frac{3}{4}$  hr. 43-49% of fat may be used in the charge and the excess  $H_2O$ 

allowed to evaporate during the cooling of the product, or the full amount of fat may be charged and the solidifying point of the soap mass lowered by the addition of hydrated salts ( $Na_2CO_3$ ,  $Na_2SO_4$ , etc.). E. L.

Soap and like preparations. H. T. BÖHME A.-G. (B.P. 379,534, 25.9.31. Ger., 6.10.30).—The foaming and detergent properties, and stability to hard and acid waters, are increased by incorporating the alkali salts of acid sulphuric esters of higher fatty ( $> C_9$ , e.g., lauryl, cetyl, stearyl) alcohols in soap compositions containing sulphonated oils, solvents, etc. E. L.

**Production of soft soaps.** F. W. LEFFER (B.P. 378,913—4, 16.5.31).—(A) The solidification temp. of soft soaps is reduced and their consistency improved by saponifying the oils under pressure with a mixed lye in which the ratio of potash to soda is  $\geq 9$  to 1 and which also contains 2—6% of alkali salts. The electrolyte is best pumped in aq. solution into the autoclave at 110—120° after the saponification. (B) When saponification is effected in the presence of a solution of an alkali salt under pressure, and above 100°, the soap produced is soft even with soda lyes; potash lyes under these conditions yield transparent soft soaps, whilst potash-soda mixtures yield transparent soft soaps. M.

Manufacture of soap powders. LEVER BROS., LTD., C. W. MOORE, and H. BALLANTYNE (B.P. 378,973, 22.5.31)—Addition of 0.6—1.8% of MgSO<sub>4</sub>,7H<sub>2</sub>O or of equiv. proportions of other Mg salts to alkali perborate soap powders retards loss of their active O and produces uniformity of their bleaching action with waters of varying hardness. S. M

Preparation of rinsing, cleansing, and fatremoving agents CHEM. FABR. BUDENHEIM A.-G. (B.P. 379,152, 20.11.31. Ger., 1.12.30)—Insol., finelydivided SiO<sub>2</sub> is dissolved in a melt or solution of Na<sub>3</sub>PO<sub>4</sub> and the product atomised. S. M.

Production of cleaning paste for dissolving and absorbing dirt, fat, and the like. H. WINTER, M. HORBERT, and G. SCHWARZE (B.P. 378,335, 10.2.32. Ger., 11.2.31)—A paste for cleaning paint, fabrics, etc. is prepared by mixing a solution (A), containing rice starch, deodorant, and H<sub>2</sub>O, with an emulsion (B), prepared by mixing CCl<sub>4</sub>, decahydronaphthalene cyclohexanol, olive oil, H<sub>2</sub>O, and finally adding aq NaOH to form a paste. E. L

Manufacture of washing and cleansing agents. A. SCHREIBER and F. MARTENS (B.P. 379,318, 24.2.31).— Dairy-waste products rich in hydroxy-acids (curdled milks, rotten cheese, etc.) are treated with caustic or carbonated alkali or alkaline earth, together with a small amount of a weak hydroxy-acid (e.g., lemon or apple juice); the mash is then incorporated with soap etc. to form household or toilet detergents. E. L.

Production of emulsions, especially of such from milk and fat or oil. A. HOFFMANN (B.P. 378,066, 30.4.31).—In continuous-acting apparatus, the oil (disperse phase) is injected under pressure into the mixing chamber containing the milk (continuous phase) through an atomising nozzle (situated in the base), which is in the form of an adjustable valve with an inverted conical seating; this causes the injected fat to spread out into a flat or cone-shaped surface. Other modified details are described. E. L.

Reagent for volumetric determination of fat [in milk etc.]. W. E. PETERSEN (U.S.P. 1,841,672, 19.1.32. Appl., 29.11.29).—The milk (etc.) is treated with an equal amount of a reagent consisting of a mixture of a salt of an aromatic acid (e.g., Na salicylate or KOBz 400 g.), a carbonate (e.g., Na<sub>2</sub>CO<sub>3</sub>, 210 g. made up to 1000 c.c. with  $H_2O$ ), a hydroxide (e.g., 40 c.c. of 50% aq. NaOH), and two alcohols (e.g., 80 c.c. each of pure or commercial BuOH and MeOH) before use. After warming (e.g., to 120°) and centrifuging, the fat, free from phospholipins, separates as a well-defined layer. E. L.

Extraction of oils [from seeds, sewage, etc.]. C. J. BLEIL (U.S.P. 1,840,149, 5.1.32. Appl., 12.4.28) .---The oil-bearing materials are agitated with a solvent, e.g.,  $C_6H_6$ , and filtered through a filter consisting of an endless chain of compartments moving over two sprocket wheels. Below the upper horizontal travel of the compartments is a vac. chamber into which the filtrate is sucked, whilst above the lower horizontal travel is a pressure chamber from which vapours and some of the recovered solvent force the residue from the compartments, the solvent containing the finer particles being pumped to the top travel. The residue is dried in a steam-heated chamber and the solvent removed from the filtrate by distillation. All vapours are vented to oil scrubbers. D. K. M.

Fish oil extraction. S. HILLER, Assr. to S. HILLER, INC. (U.S.P. 1,840,715, 12.1.32. Appl., 24.2.30). —The undried raw material is pulped and disintegrated by passage through a perforated screen, and the resultant emulsion (with or without heat-treatment, steaming, etc.) is separated into oil, H<sub>2</sub>O, and solids by centrifugal means, conveniently in two stages. E. L.

Preparation of ergosterol. C. E. BILLS, Assr. to MEAD JOHNSON & Co. (U.S.P. 1,842,929, 26.1.32. Appl., 9.4.28).—Fungus (yeast) fat (e.g., 100 g.) is dissolved in COMe<sub>2</sub> (500 c.c.) and saponified with alcoholic KOH; on diluting the mixture with the requisite amount of H<sub>2</sub>O (1000 c.c.) and chilling to  $-20^{\circ}$  for 10–12 hr. the ergosterol is pptd. and separated by filtration or centrifuging. E. L.

Wetting etc. agents.—See III. Hydrogenation catalysts.—See X. Drying oil compositions.— See XIII. Edible emulsions.—See XIX.

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

Polymerisation of turpentine oils. O. ROUTALA and O. KUULA (Suomen Kem., 1932, 5, 52-67).— Impure or partly purified turpentine is transformed on heating into a semi-solid pitch. Prolonged heating of pure turpentine at 250° in a sealed tube causes considerable polymerisation. In the presence of ZnCl<sub>2</sub>, AlBr<sub>3</sub>, or AlCl<sub>3</sub>, alkyl halides and a dark fluorescent oil are formed. The latter contains paraffin, olefine, naphthene, and benzene hydrocarbons. The fraction b.p. 172-177° is approx. 44% p-cymene. Fractionation of the distillation residue yields a brittle hydrocarbon resembling colophony and suitable for varnish making. A. G. P. Phenols in crude oil of turpentine. H. BERG-STRÖM and K. N. CEDERQUIST (Iva, 1931, No. 1, 17).— The turpentine fraction contained 15.2 g. and the tar oil fraction 33.5 g. of phenols per litre. 62 g. of phenols were recovered per 100 kg. of dry wood. CH. ABS.

Water-soluble fatty acids in crude oil of turpentine. H. BERGSTRÖM and K. N. CEDERQUIST (Iva, 1931, No. 1, 18–19).—The oil contains 20 g. of fatty acid, calc. as AcOH, per 100 g. of dry wood.

CH. ABS.

Theory of lithographic printing. F. J. TRITTON (J.S.C.I., 1932, 51, 299-313T).-All the major steps in the prep. of lithographic images on metal have been considered from the chemical point of view and compared with the earlier published ideas. Proof is given that lithography is based essentially on the adsorption of fatty acid by the metal, while the non-image areas are kept free from ink by gum arabic, which is also adsorbed. Sensitisers have been shown to produce basic ppts. on the surface which are capable of absorbing fatty acid; desensitisers produce neutral non-polar deposits not capable of adsorbing an acid. Both contact angle and lithographic experiments have been used to demonstrate that, in the absence of adsorption, H<sub>2</sub>O is capable of displacing grease from a metal surface; thus it becomes possible to explain the real difference between grease and H<sub>2</sub>O-receptive lithographic surfaces. Etches have two distinct functions which can be measured separately and the relative efficiencies of different formulæ are compared. The acidity of lithographic inks has been investigated and suggestions are made as to the acidity of the ideal inks.

Water-solubility of lead colours. M. VITTLICH (Tehn. Ajakiri, 1930, 9, 165).—Max. solubility of Pb from Pb paint in spring water coincided with max. Cl and  $N_2O_5$ ; vals. were 0.2-0.5 mg. per litre. Distilled water, when shaken with white lead and minium, dissolves 7.5 mg. Pb per litre. Ch. Abs.

Substituting asphalt obtained from acid sludge for imported gilsonite [in varnish]. L. M. BUKREEVA (Neft. Choz., 1932, 22, 293—295).—Acid sludge from Emba spindle oil was broken up by live steam, PhOH (0.5%), or Na naphthenate, and the first and second (of three) layers were oxidised in presence of MnO<sub>2</sub>. Varnishes of higher dielectric strength than that from gilsonite were prepared from the asphalts. CH. Abs.

Stability of varnish films towards mineral acids. L. RINOVA (Masloboino Zhir. Delo, 1931, No. 10, 46-50). --Varnishes made from gilsonite and linseed oil were most resistant. CH. Abs.

Analysis of varnishes. S. V. ALEXEEV (J. Appl. Chem., Russia, 1931, 4, 1096–1099).—A summary of methods. CH. Abs.

Raising of the m.p. of rosin. P. RUFIMSKI (Masloboino Zhir. Delo, 1931, No. 1, 31-34; No. 10, 17-19).-Blowing air through the molten rosin at  $130-150^{\circ}$  for 10 hr. raised the m.p. from  $60^{\circ}$  to  $72 \cdot 2^{\circ}$ ; after addition of 1% of CaO and blowing for 6 hr. at  $200-212^{\circ}$  the material had m.p. 107°. Soaps prepared from the oxidised product were hard, but rapidly darkened in air. Changes in the rosin were : acid val.  $158 \cdot 56$  to  $145 \cdot 26$ ;

sap. val. 170.00 to 161.29, ester val. 10.44 to 16.03, portion insol. in light petroleum 17.89 to 46.07%. CH. ABS.

Glyceryl esters of rosin. A. KOGAN (Masloboino Zhir. Delo, 1930, No. 9-10, 32-39).-Glucose and sucrose do not form esters when heated with rosin in a current of air at 275-280°; mannitol in presence of 1% of Zn decreases the acid val. from  $164 \cdot 6$  to  $108 \cdot 1$ . Catalysts for esterification with glycerol are, in order of decreasing activity, Zn, NaOAc, Mg, Al, and Cd. In presence of Zn the lowest acid val. observed, using glycerol, was 4.5. ZnCl<sub>2</sub>, gaseous HCl, and FeCl<sub>3</sub> are efficient esterification catalysts, but they also cause decomp. with formation of unsaponifiable products. In the esterification products the fall in acid val. is accompanied by a rise in ester val. and fall in sap. val., owing to the difficult cleavage of abietic esters with 0.5Nalcoholic KOH. CH. ABS.

Modification of the Storch-Morawski reaction for resins. Z. LEPPERT and Z. MAJEWSKA (Przemysł Chem., 1932, 16, 130–132).—The material is wetted with 2—3 drops of a freshly-prepared mixture of 15—20 vols. of  $Ac_2O$  and 1 vol. of conc.  $H_2SO_4$ , when a violet changing to blue coloration is obtained in the presence of colophony, and a red changing through violet to beetrootred and brown in the presence of albertol. R. T.

Sedimentation apparatus.—See I. [Resin from] sunflower-seed hulls.—See XII.

#### PATENTS.

Paints, colouring materials, and the like. T. F. KING (B.P. 378,051, 6.2. and 10.4.31).—Mixtures of creosote or other hydrocarbon residues, rosin, boiled linseed oil, and moderately fresh seaweed (e.g., in the proportions of 10 gals., 70 lb., 1 gal., and 4 lb., respectively) are heated at 60° and fillers and/or pigments, solvents, e.g., solvent naphtha, are incorporated if desired. S. S. W.

Manufacture of bituminous and like paints or coating materials. P. LECHLER (B.P. 379,632, 27.2.32).—Tar oil (b.p.  $\geq 200^{\circ}$ ), free from pitch and containing one or more unsaturated hydrocarbons, e.g., heavy benzol, solvent naphtha, is treated with about 1 wt.-% of conc. H<sub>2</sub>SO<sub>4</sub> at 20°, the product being subjected, if desired, to further treatment with, e.g., FeCl<sub>3</sub>, anhyd. AlCl<sub>3</sub>, PCl<sub>5</sub>, after elimination of the H<sub>2</sub>SO<sub>4</sub>, and 40-60% of bitumen, e.g., cracking-still asphalt of softening point 80°, is introduced. S. S. W.

Drying oil compositions. BAKELITE CORP., Assees. of V. H. TURKINGTON and W. H. BUTLER (B.P. 378,094, 8.5.31. U.S., 9.5.30).—Mixtures of drying oils, e.g., linseed and tung oils, are heated to 200° with < 50 (and preferably < 20) wt.-% of an oil-sol., polynuclear, phenolic resinoid, e.g., a *p*-hydroxydiphenyl resinoid, driers etc. being incorporated if desired. The resinoid (1 pt.) is preferably first combined with the tung oil (1 pt.), and the linseed oil (> 1 pt.) incorporated subsequently. S. S. W.

Coating compositions. E. I. DU PONT DE NEMOURS & Co. (B.P. 379,292, 21.5.31. U.S., 3.6.30).—Compositions of PhMe-sol. polymerides of vinyl chloride, softeners, e.g., Bu phthalate, tolyl phosphate, solvents, pigments, and, if desired, further film-forming ingredients, e.g., natural and/or synthetic resins, drying oils, are claimed. S. S. W.

Adhesives, glues, distempers, coating compositions, and aqueous paints and products resulting therefrom. C. LE B. D'ESPINOY (B.P. 375,032, 12.5.31. Fr., 3.11.30).—The addition of raw or calcined kieselguhr as an anticoagulant permitting the use of larger proportions of adhesives in  $H_2O$ -paints and distempers is claimed. A. R. P.

Production of a transparent flexible coating impervious to water vapour on pellucid [cellulose etc.] films. WOLFF & CO. KOMM.-GES. AUF AKT., R. WEINGAND, and J. SEIBERLICH (B.P. 375,194, 23.11.31. Ger., 7.3.31).— Films of gelatin, artificial resin, or cellulose derivatives are coated on one or both sides with a thin film (< 0.001 mm.) of H<sub>2</sub>O-repellent mixtures containing one or more waxes, *e.g.*, a mixture of carnauba wax (20), ceresin (5), vaseline (10), and colophony (5 pp.). A. R. P.

Manufacture of [ceramic] pigments. DEUTS. GOLD- U. SILBER-SCHEIDEANSTALT VORM. ROESSLER (B.P. 379,572, 27.11.31. Ger., 14.1.31).—Refractory white oxides serving as colour carriers and suitable as opacifiers, e.g.,  $SnO_2$ ,  $ZrO_2$ , together with small quantities of other white oxides, e.g.,  $SiO_2$ ,  $TiO_2$ ,  $Al_2O_3$ , and 1-2% of colouring oxides, e.g., of W, U, Fe, Mn, if desired, are calcined at 1100—1500° with small quantities of oxides of V and/or Mo, or suitable compounds thereof convertible into oxides on heating. Cheap, refractory, non-poisonous, yellow pigments are obtained which will withstand firing at 1300°. S. S. W.

**Printing inks.** Soc. ANON. CHLOROSODA, Assees. of L. MAGNIEN (B.P. 379,094, 25.8.31. Fr., 2.9.30).— As a fixative for the colouring material a resin is used which has been saturated by treatment with  $HNO_3$ ( $d \ 1.332$ ) at 160° and then dissolved in di- or tri-chloroethylene. The usual solvents are added as thinners. S. M.

Manufacture and use of chemical compounds which can be used as plasticisers. COURTAULDS, LTD., W. H. GLOVER, and W. BERRY (B.P. 378,911, 16.5.31).—When castor oil is heated in approx. mol. proportions with phthalic anhydride at 100° for 120 hr., a pale yellow to brown oil is obtained possessing increased  $\eta$ . It plasticises nitrocellulose lacquers, and a lacquer which also contains paraffin wax can be used to waterproof cellulose ester films.

Production of caoutchouc varnishes or lacquers. Röнм & HAAS A.-G. (B.P. 378,900, 13.2.31. Ger., 15.5.30).—Odourless, light-coloured varnishes are obtained by heating caoutchouc in hydrocarbons (b.p. 120—230°) with S and "chloride of lime" at 1—15 atm. The process is accelerated by chlorides of metals, *e.g.*, Mg, Al, Fe, Zn, or by CCl<sub>3</sub>·CO<sub>2</sub>H. Addition of vinyl compounds yields adhesives for plastics. S. M.

Removal of solvents from plastic colloids. MEIGS, BASSETT, & SLAUGHTER, INC. (B.P. 378,052, 2.3.31. U.S., 4.3.30).—The plastic, e.g., cellulose sheet, is immersed in a bath comprising non-solvents therefor, e.g., H<sub>2</sub>O or aq. NaCl or sugar, and an anti-blushing

agent, e.g., 20—40 vol.-% of EtOH, having osmotic pressure  $\prec$  that within the plastic, and the osmotic pressure of the bath is gradually reduced, e.g., by addition of H<sub>2</sub>O. S. S. W.

Manufacture of lacquers, films, coating preparations, adhesives, impregnating agents, and the like. I. G. FARBENIND. A.-G. (B.P. 378,608, 29.6.31. Ger., 7.7.30).—Condensation products of polyvinyl alcohols and aldebydes, and solutions thereof in org. solvents, are claimed. S. S. W.

Lacquer or like coatings. KODAK, LTD., Assees. of N. S. KOCHER (B.P. 378,927, 16.4.31. U.S., 16.4.30). —Cellulose acetate finishing coats are made to adhere to nitrocellulose undercoats by the use of an intermediate coat comprising, *e.g.*, nitrocellulose with or without plasticiser, *e.g.*, tolyl phosphate, this buffer coat being free from resin, gum, and oils. [Stat. ref.] S. S. W.

Lacquer composition. J. G. DAVIDSON, ASST. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,838,368, 29,12.31. Appl., 10.2.27).—Lacquers comprising a cellulose ester, a solvent therefor, and a polymerisation product of a mixture of vinyl acetate and chloride (sol. in PhMe,  $C_6H_{e_0}$  and BuOAc) are claimed.

S. S. W.

Quick-drying lacquer. L. N. BENT, Assr. to HERCULES POWDER Co. (U.S.P. 1,839,529, 5.1.32. Appl., 22.3.27).—An org. abietate, preferably one prepared from wood rosin and a monohydric aliphatic alcohol, *e.g.*, Et abietate, is added to nitrocellulose or nitrostarch lacquers. S. S. W.

Wax-conditioned finish remover. C. ELLIS (U.S.P. 1,838,908, 29.12.31. Appl., 6.1.26).—Mixtures of wax, e.g., hard paraffin wax, with monocyclic hydrocarbon solvents therefor, e.g., benzol, together with 10-20% of an alkyl ester of b.p.  $\gg 100\%$ , e.g., EtOAc (to inhibit crystallisation of the wax), and other finishloosening solvents, e.g., COMe<sub>2</sub>, EtOH, the whole substantially free from corrosive phenolic substances, are claimed. A small amount of nitrocellulose may be added as gelatiniser for the wax. S. S. W.

Production of synthetic resins from alcoholamines and organic acids. CHEM. FABR. DR. K. ALBERT G.M.B.H. (B.P. 378,596, 23.6.31. Ger., 17.4.31. Addn. to B.P. 376,929; B., 1932, 948).—The alcoholamines are caused to react with the polybasic org. acids or their anhydrides together with natural or synthetic resin acids or with higher unsaturated fatty acids or their esters. S. S. W.

Manufacture of condensation products of urea or its derivatives and formaldehyde. O. NEUSS (B.P. 379,154, 21.11.31).—Urea or a derivative thereof is fused, with or without the addition of condensing agents, e.g., conc.  $H_2SO_4$ , fillers, e.g., fibrous materials, fluxes, e.g., PhOH, etc., and aq.  $CH_2O$  is added to the melt. The pptd. product is washed, dried, and hotpressed. S. S. W.

Manufacture of condensation products [resins from aldehydes and arylsulphonamides]. I. KREIDL, C. ROSEN, and K. RUTTER (VEREIN CHEM. FABR. KREIDL, HELLER & Co.), Assees. of G. WALTER (B.P. 375,843, 27.3.31. Austr., 27.3.30).—An aldehyde, preferably  $CH_2O$ , is condensed with an arylsulphonamide carrying "further  $NH_2$ - or substituted  $NH_2$ -groups," especially  $SO_2NH_2$  groups, *e.g.*, benzene-*m*-disulphonamide, mixed xylenedisulphonamides, aniline-2:4:6trisulphonamide (product, m.p. 305°), *p*-toluidine-2:5disulphonamide (m.p. 237°). C. H.

Production of chlorinated diaryl resins. FEDERAL PHOSPHORUS Co., Assees. of R. L. JENKINS and J. A. SIKARSKI (B.P. 378,849, 12.3.31. U.S., 23.4.30).— Resins containing > 60% Cl, prepared as in B.P. 358,703—4 (B., 1931, 174, 1147), e.g., by chlorinating mixtures of technical Ph<sub>2</sub> with molten, distilled, highboiling compounds and still bottoms produced during thermal synthesis of Ph<sub>2</sub>, are fractionally distilled, e.g., until 45—95% is distilled over, giving pale resins.

S. S. W.

Plastic composition. M. E. GRIFFITHS, Assr. to A. S. BOYLE Co. (U.S.P. 1,838,618, 29.12.31. Appl., 17.11.23).—15—30 pts. by wt. of a filler, e.g., wood flour, are incorporated with 85—70 pts. by wt. of the following solution: 15-20% of nitrocellulose, e.g., celluloid scrap, 5-9% of resin, e.g., ester gum, 1-5% of non-drying vegetable oil, e.g., castor oil, 79-66% of a nitrocellulose solvent liquor, e.g., methylated spirit 5—15,  $C_6H_6$  20—30, low-b.p. ketone 54—21, to give a doughy putty-like composition. S. S. W.

Moulded compositions. IMPERIAL CHEM. INDUS-TRIES, LTD., and R. GREENHALGH (B.P. 377,979, 1.5.31).--Dried and ground mixtures of an artificial, thermohardening, resinous binder, *e.g.*, an acid-condensed PhOH-CH<sub>2</sub>O resole, with a filler of chopped cordage fibre, *e.g.*, sisal or hemp  $(\frac{1}{2}-\frac{1}{8}$  in. long), are claimed. S. S. W.

Manufacture of moulded article. L. FREUDER, Assr. to FIBERPLASTIC CORP. (U.S.P. 1,842,544, 26.1.32. Appl., 8.5.28).—An aq. solution of gelatin (hide glue) and  $Na_2Cr_2O_7$  is dried below 100° and the product, after pulverising and admixture, if desired, with fillers, is moulded at 150—160° under pressure. Alternatively, sheets of paper or cloth are impregnated with the solution, and, after drying (<100°), the sheets are united by heat and pressure. L. A. C.

Manufacture of [moulded] glued materials. A. WINOGRADOW (U.S.P. 1,841,564, 19.1.32. Appl., 24.9.29).—A mixture of gum or glue, a hydrated salt (e.g., Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>,5H<sub>2</sub>O, Na<sub>2</sub>HPO<sub>4</sub>,12H<sub>2</sub>O, MgHPO<sub>4</sub>,7H<sub>2</sub>O, alum), and a filler, e.g., sawdust, is moulded under heat and pressure until the H<sub>2</sub>O has been absorbed by the glue. The products are water- and fire-proof. L.A.C.

Coverings for floors, walls, and the like [from tesseræ of plastic materials]. LINOLEUM MANUFG. Co., LTD., and A. A. GODFREY (B.P. 380,456, 14.4.31).

Oilproofed Al vessels.—See X. Insulating material.—See XI. Cleaning paste.—See XII.

# XIV.—INDIA-RUBBER; GUTTA-PERCHA.

See also A., Oct., 1036, Thermal decomp. of caoutchouc. Gutta-percha and rubber hydrocarbons.

#### PATENTS.

Treatment of rubber latex or similar colloidal substances. ELECTRICAL RESEARCH PRODUCTS, INC., Assees. of A. R. KEMP (B.P. 378,952, 20.5.31. U.S., 21.5.30).—The colloidal suspension is heated in an autoclave at a temp. above the normal b.p. of the liquid, the space above the level of the liquid being maintained at a pressure above the v.p. of the liquid, *e.g.*, by introduction of steam, air, or inert gas. The temp. of the upper portion of the autoclave is maintained above the temp. of the liquid, *e.g.*, by a special heating coil. This procedure prevents the undesirable formation of films of rubber on the inner surface of the autoclave. D. F. T.

Manufacture of vulcanisable rubber compounds. COLVULC RUBBER CO., Assees. of P. N. SYLVESTER (B.P. 379,819, 21.7.31. U.S., 5.8.30).—Rubber latex is mixed thoroughly with a solution of rubber in a solvent, a small quantity of an accelerator capable of vulcanising at ordinary temp. is added, and powdered scrap vulcanised rubber is then incorporated. The product can be more effectively bonded to metal or similar surfaces than the usual compounds. D. F. T.

Rubber mixes. A. H. STEVENS. From PURE CALCIUM PRODUCTS CO. (B.P. 379,942, 15.12.31).—A compounding ingredient for rubber is prepared by grinding an inorg. earthy material, e.g.,  $CaCO_3$ , with 0.1—10% of an org. acid, e.g., stearic acid or BzOH, or a salt or ester thereof, in a ball mill under specified conditions. Vulcanised rubber containing such a product has improved tensile, wearing, and flexing qualities. D. F. T.

Composition of matter [for rubber compounding] and its manufacture. H. A. ENDRES, ASST. to CELITE CORP. (U.S.P. 1,842,394, 26.1.32. Appl., 7.2.28. Cf. U.S.P. 1,574,380; B., 1926, 439).—The CaO-reacted and disruptively dried siliceous product is exposed to SO<sub>2</sub> until the wt. is considerably increased. D. F. T.

Vulcanisation of rubber. C. O. NORTH, Assr. to RUBBER SERVICE LABS. Co. (U.S.P. 1,838,862, 29.12.31. Appl., 13.4.26).—The condensation products of tetrahydroquinaldine with an aliphatic aldehyde below  $C_8$ , e.g., the ethylidene derivative, are applied as accelerators of vulcanisation and show little tendency to lead to overvulcanisation with prolongation of heating. D. F. T.

Vulcanisation of rubber. W. Scott, Assr. to RUBBER SERVICE LABS. Co. (U.S.P. 1,838,159, 29.12.31. Appl., 1.11.26).—The product of interaction of CNCI and the Na salt of mercaptobenzthiazole is used in admixture with a basic org. accelerator, *e.g.*, diphenylguanidine, to expedite vulcanisation. D. F. T.

Treatment of rubber. S. M. CADWELL, Assr. to NAUGATUCK CHEM. Co. (U.S.P. 1,840,932, 12.1.32. Appl., 2.7.29).—A methylated amine resulting from the reaction of  $CH_2O$  and polyethylene polyamine, preferably in an inorg. acid medium, is incorporated in rubber to accelerate vulcanisation. D. F. T.

Incorporation of plasticisers and activators into rubber. GOODYEAR TIRE & RUBBER CO. (B.P. 378,518, 15.5.31. U.S., 19.6.30).—Crude rubber, preferably in a crêped condition as produced on washing machines, is dipped into molten stearic acid, lauric acid, or oxidised paraffin wax for a few min. After withdrawal, the rubber mass, which will have become impregnated with half of its wt. of the softening agent, may be kept at approx.  $51^{\circ}$  to increase its homogeneity. The presence of 1-2% of  $H_2O$  is necessary in the rubber for satisfactory absorption of stearic acid. The products, preferably after further homogenisation by short mechanical working, are suitable for use as "master batches" for the compounding of additional rubber. D. F. T.

Impregnating compound [containing rubber]. W. N. TRAYLOR, ASST. to HERCULES POWDER CO. (U.S.P. 1,840,989, 12.1.32. Appl., 24.8.29).—A mixture for impregnating felt, canvas, etc., e.g., for shoe manufacture, is made by fusing together rubber and rosin and a plasticiser such as pine oil foots. D. F. T.

Manufacture of rubber-fibre articles. F. O. WOODRUFF, Assr. to H. H. BECKWITH (U.S.P. 1,842,706, 26.1.32. Appl., 13.2.30).—A loose mass of unwoven fibres, preferably carrying on their surfaces a latex coagulant, e.g., AcOH, is mixed with an aq. dispersion of rubber. After removal of excess dispersion, the mass is dried and beaten in  $H_2O$  to individualise the fibres; the suspension is then formed into the desired article, e.g., by sheeting. D. F. T.

Manufacture of rubber-fibre products. G. P. F. SMITH and T. G. RICHARDS, ASSTS. to DISPERSIONS PROCESS, INC. (U.S.P. 1,841,067, 12.1.32. Appl., 29.10.28).—The fibrous portion, roughly separated from rubber scrap such as pneumatic tyres, is disintegrated, beaten with  $H_2O$ , and mixed with an aq. dispersion of rubber. The dispersed rubber in the mixture is then coagulated on to the fibre and the mixture shaped, *e.g.*, by sheeting on a paper machine, and dried. D. F. T.

Reclaiming of vulcanised rubber-fibre products. RUBBER REGENERATING CO., Assees. of A. W. BULL (B.P. 378,643, 11.8.31. U.S., 22.8.30).—The dry material is disintegrated and any loose rubber is removed by screening. The remaining mass is beaten with H<sub>2</sub>O and then screened; these processes are then repeated, and finally the freed rubber is removed, *e.g.*, by centrifuging. D. F. T.

Manufacture of rubber articles. W. F. ZIMMERLI and W. L. SEMON, Assrs. to B. F. GOODRICH CO. (U.S.P. 1,841,076, 12.1.32. Appl., 4.10.28).—A coating of rubber is applied to an article by spraying thereon simultaneously a compounded rubber latex and a solution of AcOH so that a smooth dense layer is obtained substantially without flow. The coating is then dried and vulcanised. If the coating is to remain permanently attached, a preliminary layer of an adhesive composition may be applied. D. F. T.

Manufacture of composite articles [containing rubber]. GOODYEAR'S INDIA RUBBER GLOVE MANUFG. Co., Assees. of R. B. MARR (B.P. 379,985, 19.2.32. U.S., 8.1.32).—Vulcanised rubber is attached to fibrous material such as leather by disposing a layer of semivulcanised rubber between the two, preferably roughened, surfaces, and uniting it to them by a rubber-like cement. D. F. T.

Method of securing rubber to metal. C. M. CAR-SON, ASST. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,841,322, 12.1.32. Appl., 23.2.29).—A surface of one

of the materials to be attached is first coated with a cement comprising latex, hemoglobin, S, a tanning agent, e.g.,  $CH_2O$ , and, if desired, also an accelerator of vulcanisation. After drying the coating and baking it, e.g., at 120° for  $1\frac{1}{2}$  hr., the two surfaces to be secured are brought into contact and the whole is subjected to vulcanisation. D. F. T.

Improving the [physical] properties of rubber, J. McGAVACK, Assr. to GEN. RUBBER Co. (U.S.P. 1,840,243, 5.1.32. Appl., 13.12.29).—Its resistance to abrasion and flexing especially are improved by incorporating in the rubber latex a soap comprising a higher fatty acid (e.g.,  $C_{10}$ — $C_{30}$ ) and a volatile base and a watersol. substance (e.g.,  $NH_4Cl$ ,  $NH_4NO_3$ ,  $NH_4$  lactate) hydrolysable to products capable of decomposing  $NH_4$ soaps and proteinates; the mixture is then dried.

D. F. T.

Manufacture of rubberised articles. J. McGAVACK, Assr. to NAUGATUCK CHEM. Co. (U.S.P. 1,839,983, 5.1.32. Appl., 19.2.27).—The tendency of goods with a coating of latex rubber to absorb  $H_2O$  is decreased by treatment, while wet, with a  $H_2O$ -sol. soap and a precipitant bath (e.g., of alum) successively. The solutions may be warm, and a washing operation may follow the application of each solution. D. F. T.

Rubber and articles manufactured therefrom. F. A. and L. E. FISHER (B.P. 379,746, 4.6.31).—The wearresisting qualities of rubber are improved by incorporation of a proportion, e.g., 25%, of a micaceous hæmatite. D. F. T.

Preservation of oxidisable organic compounds [e.g., rubber]. IMPERIAL CHEM. INDUSTRIES, LTD., H. M. BUNBURY, K. W. PALMER, and W. J. S. NAUNTON (B.P. 379,740, 4.6.31).—Rubber and rubber-like materials are preserved by incorporating up to approx. 5% of a mercaptophenol, e.g., monothiopyrocatechol, monothioquinol, or 1-thiol-β-naphthol. D. F. T.

Treatment of rubber [for retardation of deterioration]. S. M. CADWELL and L. MEUSER, ASSTS. to NAUGATUCK CHEM. Co. (U.S.P. 1,839,950, 5.1.32. Appl., 15.2.27).—Compounds containing the group  $\cdot X \cdot C:N$ , X being an element of the S group linked to a metal or org. group, and free from any metallic element capable of forming 2 or more stable chlorides, are incorporated in rubber or rubber latex to improve the ageing qualities of the manufactured product. Examples are  $NH_4SCN$ and dicyanodiamide. D. F. T.

Manufacture of rubber derivatives. G. OENSLAGER. Assr. to B. F. GOODRICH CO. (U.S.P. 1,841,295, 12.1.32, Appl., 9.2.29).—Rubber is converted into a hydrohalide by treatment with a solution of H halide in a solvent (preferably volatile) capable of dissolving substantial proportions of the latter and of being readily absorbed by rubber, but substantially incapable of dissolving rubber. A solution of HCl in EtOAc is convenient, and previously shaped rubber articles may be so treated. The treatment converts thin rubber sheet into a hard, tough, translucent material. D. F. T.

Xanthates. Vulcanisation accelerators.—See III. Caoutchouc varnishes.—See XIII. Chewing gum. —See XIX.

## XV.—LEATHER; GLUE.

Action of pancreatin on raw hide. M. BERGMANN and G. POJARLIEV (Collegium, 1932, 608—612).—Küntzel and Dietsche's results (cf. A., 1931, 654; B., 1931, 732) are explained. When raw hide was soaked and mechanically worked, some of the fibres were abraded, the pieces floated in the soak liquor, and owing to their large surface area were dissolved by the pancreatin, so producing a high N val. When the soak liquor was thrown away or the mechanical treatment omitted, the N val. of the liquors after treatment of the pelt with pancreatin was normal. The action of pancreatin on raw hide was not increased by presoaking the hide in  $H_2O$ . D. W.

Transmission of water vapour through leather. R. S. EDWARDS (J. Soc. Leather Trades' Chem., 1932, 16, 439—449).—Apparatus is described for the determination of the amount of  $H_2O$  vapour passing through a sample of leather across the faces of which flow currents of air maintained at certain R.H. The  $H_2O$  vapour transmitted through sole leather was const. and independent of R.H. and the velocity and temp. of the air flow. The relative water vapour permeability const. of leather is given by  $k_{\theta}/D$ , *i.e.*, the ratio of the conductivity of the leather at  $\theta^\circ$  to that of air per unit surface area.

Molecular absorptive power of sole leather for water. F. BĚLAVSKY and G. WANEK (Gerber, 1931, 57, 135—138; Chem. Zentr., 1932, i, 1748).—A distinction is drawn between capillary and mol. absorption of  $H_2O$ by leather; the latter process is associated with swelling. Characteristic  $H_2O$ -absorption curves have been constructed for various leathers of known tanning history. Capillary absorption can be determined in 3, and mol. absorption in 24, hr. A. A. E.

Determination of the apparent specific gravity of leather. W. ACKERMANN (Collegium, 1932, 613-618) .-- Some of the Hg enters the leather in the determination of the apparent vol. of the leather by means of Thuau and Goldberger's apparatus (cf. B., 1931, 986). This error caused by evacuation is greatest with the most porous leathers, e.g., chrome-tanned. Only 1% of the apparent vol. change in the leather caused by the evacuation of the apparatus is due to adhering air. By allowing for this correction, and determining the Hg forced into the leather, vals. can be obtained which show the total vol. change in the leather caused by the evacuation; the vol. change caused by the closing up of leather pores can then be calc. Such measurements have been derived for box sides and chrome-tanned calf. D. W.

Gambier catechins. C. FLURY (J. Soc. Leather Trades' Chem., 1932, 16, 449—451).—Of the original catechins in the gambier leaf (Uncaria gambier) only about 20% is left unchanged in the finished extract, whereas the remainder (about  $37\cdot4\%$ ) is condensed to sol. tannins. Small quantities are converted into insol. condensation products forming dyes of the anthocyanidin, flavone, and phloroglucinol series, hence the red colour of the insol. matter. No tanning or dyeing properties are shown by the fresh juice of the gambier leaf. D. W. [Tannery] leach operation and control. L. M. WHITMORE (J. Amer. Leather Chem. Assoc., 1932, 27, 334—342).—Methods of recording operating results are given, and a brief outline of the effect of such factors as the prep. of material, temp. gradients, flow of liquor, time of extraction, quality of  $H_2O$  used, size of extraction unit, etc. is given. Methods of tanning analysis are examined. D. W.

Effect of volume of the precipitate on the accuracy of the determination of insoluble matter in tannin analysis. J. P. BERKMANN (Collegium, 1932, 618— 622).—Inaccuracy arises owing to the fact that the vol. of the ppt. is greater the more conc. is the solution. The correction factor for the insol. matter is d/(d-a), where d is the sp. gr. of the insol. matter and a the dry residue from 1 c.c. of the clear filtered solution.

D. W.

Fermentation in tanning liquors. D. MICHLIN, P. KOPELIOVICH, and A. SIMSKAYA (Vestn. Kozhev. Prom. Torgov., 1929, 125-127).-CH2O (0.007%) stopped the fermentation of willow-bark liquor  $(d \ 1.02)$ ; the acid content did not change, and the skins were well tanned. 0.1% NaF stopped the formation of volatile acids, and 0.01% N<sub>2</sub>H<sub>4</sub> prevented the formation of non-volatile acids; these reagents should be used together. The liquor  $(p_{\rm H} 2.9)$  was brought to  $p_{\rm H} 5.5$  with Na<sub>2</sub>CO<sub>3</sub> and NaHCO<sub>3</sub>; in the absence of skins the  $p_{\rm H}$  decreased to 4.4 after 5 days, then remaining almost const. for 4 weeks. By addition of 0.5% NaHSO<sub>3</sub>  $p_{\rm H}$  5.5 was reached ; after 5 days it was 5.2, const. for 2 weeks, and increased to  $5 \cdot 8$  after 4 weeks. If the  $p_{\rm H}$  is brought to 4.9 with  $SO_3'' + HSO_3'$  it remains const. for 4 weeks. 0.1% NaF + 0.1% CaCl<sub>2</sub> buffers the liquor to  $p_{\rm H}$  5.7-5.8, 0.007% CH<sub>2</sub>O + 0.1% NaF to 5.5, 0.25%HSO<sub>3</sub>' + 0.1% NaF to 5.2-5.7; these vals. are CH. ABS. unchanged for 2 weeks.

Influence of alkalinity on the tan content of tanning liquors. G. ARBUSOV and A. MICHAILOV (Vestn. Kozhev. Prom. Torgov., 1929, 127—129).—In alkalised liquors the  $p_{\rm H}$  decreases and frequent addition of alkali is necessary; alkalisation does not cause large changes or losses of tanning material. The amount of insol. matter decreases by keeping, even at the same  $p_{\rm H}$ . CH. ABS.

Oxidising enzymes of vegetable tanning materials. D. MICHLIN and N. KOPELIOVICH (Vestn. Kozhev. Prom. Torgov., 1929, 53—54).—Peroxidase was isolated from pine and willow barks; the optimum  $p_{\rm H}$  is  $3\cdot 8$ —  $5\cdot 6$ . Diminution of activity is caused by  $\rm H_2O_2$ , HCN, and  $\rm H_2S$ . CH. Abs.

Determination of lactic acid in vegetable tan liquors. J. H. HIGHBERGER and D. L. YOUEL (J. Amer. Leather Chem. Assoc., 1932, 27, 343—358).—The liquor is detannised with Pb(OAc)<sub>2</sub> and basic Pb carbonate and filtered, sugars are removed by boiling the filtrate with  $Ca(OH)_2$  and  $CuSO_4$  and filtering, and the lactic acid in the filtrate is oxidised to MeCHO by treatment with  $H_2SO_4$  and  $MnSO_4$ . The MeCHO is then distilled over into a known amount of standard NaHSO<sub>3</sub> and the excess NaHSO<sub>3</sub> is titrated with 0·1N-I. Tannins and tannin-like substances do not interfere. Results of the determinations on a series of tan liquors are given, and the differences from the official method of determining the lactic acid as total acidity are shown. A special apparatus has been devised for the determination.

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D. W.

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Microscopical determination of tanning power and distribution of tans in leather layers. G. WOLPERT (Vestn. Kozhev. Prom. Torgov., 1929, 687– 688).—The section (cut while frozen in  $CO_2$ ) is stained with hæmatoxylin, washed with  $H_2O$ , treated with 70% EtOH containing a little HCl, then with NH<sub>3</sub>, and finally washed with  $H_2O$ , and immersed in EtOH of increasing concn. and then in xylene and Canada balsam. Spots not thoroughly tanned appear violet; the % of tanning is determined micrometrically. For the determination of the distribution of tans in leather layers the sections are dyed with Fe NH<sub>4</sub> alum, washed, and treated with EtOH, xylene, and Canada balsam. If much tanning material is present the section is coloured black; less tan causes a cherry-red and too little a light grey coloration. CH. ABS.

Humidity and resistance properties of tanned leather. G. POVARNIN, P. POLUS, and V. VASILIEV (Vestn. Kozhev. Prom. Torgov., 1929, 55-56).—The firmness of tanned leather is < that of calf skin containing 40-50% H<sub>2</sub>O; at a higher H<sub>2</sub>O content it is larger. Wetting increases the elasticity of the leather < that of skin, whilst the resistance properties undergo little change. CH. ABS.

Analysis of glue and gelatin. L. SZIKORA (Requind, 1932, 1, 54-56).—Turbidity is compared with that of ignited light fuller's earth (1 g.), passed through a 200mesh sieve and suspended in  $H_2O$  (1000 c.c.); the turbidity of this is 1000 units. Various determinations are described and a specification for gelatin is given.

CH. ABS. New curve of thermal behaviour of gelatin. M. BRIEFER and J. H. COHEN (Ind. Eng. Chem., 1932, 24, 892—894).—For all gelatins irrespective of origin or chemical history the gelatin structure attains its max. consistency at an ageing temp. of 20° (approx.). Gelatin incorporated in an ice-cream mixture shows the same thermal behaviour as pure gelatin-H<sub>2</sub>O solutions. W. J. B.

# Freezing of H<sub>2</sub>O in gelatin.—See XIX.

See also A., Oct., 992, [H2O] absorption [by leather].

#### PATENTS.

Manufacture of leather. F. LOBER (B.P. 379,281, 22.5.31).—Defective hides or skins, or parts thereof, are treated with a sol. albuminous substance such as that obtained by hydrolysing leather scrap with acid until < 10% of its N content is titratable with CH<sub>2</sub>O.

D. W.

Treatment of leather [for boots and shoes]. H. S. POCHIN (B.P. 379,785, 16.6. and 5.10.31).—The cut parts are immersed in  $H_2O$  in a suitably constructed cabinet, which is subsequently partly or completely evacuated, and the leather is allowed to remain until tempered. D. W.

Tanning of hides or skins. UNITED SHOE MACH-INERY CORP., Assees. of M. M. MERRITT (B.P. 379,300,

25.4.31. U.S., 12.1.31).—The hides (etc.) are treated with a non-tanning agent, e.g.,  $K_2Cr_2O_7 + HCl$ , slicked out upon a smooth supporting surface, and in this condition treated with a second reagent, e.g.,  $Na_2S_2O_3$ , to produce a tanning reagent by interaction with the non-tan. D. W.

Oil tanning [of skins]. A. ROGERS (U.S.P. 1,841,633, 19.1.32. Appl., 16.3.29).—The skins are drummed with a paste of NH<sub>3</sub>, CH<sub>2</sub>O, and incompletely saponified marine-animal oils. D. W.

Moulded materials. Adhesives etc.—See XIII. Composite articles.—See XIV.

# XVI.—AGRICULTURE.

Fractionation, composition, and hypothetical constitution of certain colloids derived from the great soil groups. I. C. BROWN and H. G. BYERS (U.S. Dept. Agric. Tech. Bull., 1932, No. 319, 43 pp.) .---Fractionation of the fine silt and clay particles of various soil types into size ranges of 50-5µ, 5-1µ. 1-0.3  $\mu$ , 0.3-0.1  $\mu$ , and < 0.1  $\mu$  is described and chemical analyses of the fractions are recorded. Colloid fractions of chernozem and prairie soils showed general similarity of composition within a given size range, and the existence of a similar predominating type of colloidal acid in each soil is suggested. The considerable variation in the composition of podsol fractions is ascribed to the presence in the colloid of varying proportions of Fe oxide with the colloid acid characteristic of chernozems. In laterite soils the dominant complex differs from that in the above types. The adsorption of H<sub>2</sub>O-vapour by soils is not dependent on surface alone, but is influenced by chemical composition. The origin and nature of the characteristic acid complexes (montmorillonitic and halloysitic acids) are discussed. Natural soil colloids are partly neutralised salts of these acids and contain both basic and acid radicals together with org. colloids. A. G. P.

Adsorption of vapours and gases by soils and clays and their application to the determination of the surface of these substances. H. KURON (Kolloid-Beih., 1932, 36, 178-256).-The isotherms expressing the adsorption of  $CO_2$  by closely related soils are practically coincident. The amount of  $CO_2$ adsorbed depends largely on the H<sub>2</sub>O content of the soil or clay. The adsorption of H<sub>2</sub>O is also similar for related soils; it varies with their composition and dispersity, with the content of org. substances, and the nature of the adsorbed cations, which should be present in a readily exchangeable form. A method is devised for deriving a val. for the surface of the soil from the H<sub>2</sub>O-adsorption data. The results obtained thereby are in agreement with those deduced from measurements of particle size of the same soils. E. S. H.

**Phosphates in soil. I, II.** S. OSUGI, S. YOSHE, and N. NISHIGAKI (J. Agric. Chem. Soc. Japan, 1932, 8, 280–298, 361–368).—I. The solubility of the  $P_2O_5$ of  $Ca_3(PO_4)_2$  is min. at  $p_{\rm H}$  7.68, increasing rapidly on the acid side and slightly on the alkaline side; that of AlPO<sub>4</sub> is min. at  $p_{\rm H}$  4.07–6.93 and of FePO<sub>4</sub> at  $p_{\rm H}$ 3–6, with a remarkable increase on the alkaline side. II.  $P_2O_5$  solubilities at various  $p_H$  vals. were determined for various soils. CH. ABS.

Water-soluble magnesium in soils. J. E. CHAP-MAN (Science, 1932, 76, 83).—The presence of sol. Mg salts is suspected to be a factor in the apparent infertility of certain soils towards lucerne. Soils which develop a lake with Ruigh's test for  $H_2O$ -sol. Mg give increased yields of lucerne and sweet clover when treated with superphosphate. L. S. T.

Producing soda and potassium nitrate fertiliser from sylvinite. V. S. YATLOV (J. Chem. Ind., Russia, 1932, No. 3, 34—37).—Traces of Ca and Mg are removed by  $CO_2$  in the prep. of NaHCO<sub>3</sub>, but  $SO_4''$  from CaSO<sub>4</sub> accumulates and decreases [K'] by nearly 33%. During addition of  $CO_2$  K<sub>2</sub>SO<sub>4</sub> does not ppt., and the yield of NaHCO<sub>3</sub> is sufficiently high. A solution saturated at 0° with KCl + NaCl + NH<sub>4</sub>Cl dissolves more NaCl when the temp. is raised. CH. ABS.

Effect of manurial treatment on nitrogen fixation and nitrification in certain experimental plots. B. THOMAS and F. J. ELLIOTT (J.S.C.I., 1932, 51, 332—336T).—N fixation and nitrification in pasture land was found to depend primarily on reaction. On arable soils no significant relationship could be established. Org. manures proved more effective than inorg. fertilisers. The amounts of N fixed and of NO<sub>3</sub>-N produced on an arable soil were markedly greater than on grassland.

Influence of soil and climatic conditions and of fertiliser on the quality of sugar beet and sugar manufacture. V. DENISIEVSKI, F. UKRADIGA, S. SHATZ, L. B. MINTZ, P. GROSHEV, S. KLEIBS, and A. SHCHEPETILNIKOV (Nauk. Zapiski Tzuk. Prom., 1931, 14, 347–379).—N fertilisers increase the yield of sugar per acre, but slightly increase that of molasses.  $P_2O_5 + N$  gives good results and decreases losses. Variation in the harmful-N and ash content of beets also depends on the fertilisers used. CH. ABS.

Report on experiment with different forms of phosphatic fertilisers [for sugar cane] at Wilton Park Estates, Empangeni. H. H. DODDs and P. FowLIE (Proc. S. Afr. Sugar Tech. Assoc., 1932, 57-61). —Results obtained with plant cane, calculating the profit by deducting the cost of the fertiliser from that of the increased sucrose yield over and above the sucrose yield from the control plots without fertiliser, show the variation in profit from various phosphatic combinations to be not very great. Bone meal gave the greatest yield of cane but superphosphate the greatest profit per acre, owing to its lower cost. In the case of ratoon canes also phosphatic fertilisers were found to give a very useful profit. J. P. O.

Significance of potash fertilisation in viticulture. O. ENGELS (Wein u. Rebe, 1932, 13, 388—393; Chem. Zentr., 1932, i, 2223).—Generous K fertilisation increases carbohydrate formation, and therefore the sugar content of the grapes. A. A. E.

Nutrition of the cultivated mushroom, *Agaricus* campestris, and chemical changes brought about by this organism in the manure compost. S. A.

WAKSMAN and W. NISSEN (Amer. J. Bot., 1932, 19, 514-537; cf. B., 1932, 907).—During growth, mushrooms utilise lignin and org. N complexes and, to a smaller extent, cellulose and hemicellulose. Much of the org. N is converted from an insol. into a sol. form and into NH<sub>a</sub>. A. G. P.

Effect of storage on pyrethrum flowers. C. B. GNADINGER and C. S. CORL (Ind. Eng. Chem., 1932, 24, 901—903).—A loss of  $30-43\cdot6\%$  of the pyrethrin content occurred when freshly ground pyrethrum flowers were stored for 1 year. The loss was greatest for samples stored in open trays, but for other types of containers it was practically the same in all cases. Storage in partial vac. did not retard the decomp. Losses of  $43\cdot6\%$  and  $33\cdot3\%$  of the pyrethrin content were accompanied by losses of toxicity of  $22\cdot7\%$  and  $20\cdot4\%$ , respectively. W. J. B.

Fibre waste. Straw-board effluent.-See V.

See also A., Oct., 1067, Soya-bean culture media.

# PATENTS.

[Electrical] treatment of soil for plant-culture purposes. COUNTY OF LONDON ELECTRIC SUPPLY Co., LTD., and R. E. LLOYD-OWEN (B.P. 378,130, 29.5. and 22.8.31).—The soil is sterilised by resistance heating with a.c. in a closed container. A. R. P.

Manufacture of fertilisers. F. ROTHE and H. BRENEK, ASSTS. to RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G. (U.S.P. 1,842,843, 26.1.32. Appl., 19.7.27. Ger., 30.7.26).—A mixture of raw phosphate, SiO<sub>2</sub>, an alkali carbonate, and an alkali chloride and/or sulphate, containing sufficient SiO<sub>2</sub> to convert 1 mol. of CaO in the Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> into 2CaO,SiO<sub>2</sub>, and sufficient alkali salts to replace the CaO leaving the phosphate by alkali oxide, is calcined at 1200°, steam being introduced. W. J. W.

Production of a fertiliser mixture. KALI-FORSCH-UNGS-ANSTALT G.M.B.H. (B.P. 375,965, 29.5.31. Ger., 12.11.30).—K<sub>2</sub>H<sub>3</sub>(SO<sub>4</sub>)PO<sub>4</sub> produced by the action of conc. H<sub>3</sub>PO<sub>4</sub> on K<sub>2</sub>SO<sub>4</sub> is heated with (a)  $29 \cdot 5\%$  of NH<sub>4</sub>NO<sub>3</sub> and NH<sub>3</sub> to neutrality to obtain a mixed fertiliser containing 23% K<sub>2</sub>O, 17% P<sub>2</sub>O<sub>5</sub>, and 17% N, or (b) 48 · 5% of (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> and 37% of Florida phosphate to obtain a dry, non-hygroscopic product containing 18 · 6% K<sub>2</sub>O, 5 · 5% N, and 21% P<sub>2</sub>O<sub>5</sub>. A. R. P.

Treatment of seeds and the like. SCHERING-KAHLBAUM A.-G. (B.P. 379,956, 8.1.32. Ger., 22.1.31). —The development and flowering of plants and the like derived from seeds, root stocks, tubers, etc. is accelerated by impregnation, injection, or dry-process treatment of the seeds etc. with hormones or hormone derivatives or preps., with or without one of the customary immunising processes. E. H. S.

[Arsenical] dressing for seed grain. A. SCHMIDT, Assr. to WINTHROP CHEM. Co. (U.S.P. 1,831,721, 10.11.31. Appl., 7.1.27. Ger., 2.1.26).—A mixture of phenylarsineoxide (5), CuSO<sub>4</sub> (15), Na sulphonate of naphthol pitch (70), and the Na salt of a dibutylnaphthalenesulphonic acid (10%) is claimed. A. R. P.

Manufacture of insecticides. R. H. CARTER (U.S.P. 1,842,443, 26.1.32. Appl., 15.11.29).—An AI compound, insol. in  $H_2O$ , e.g.,  $Al_2O_3$ , is treated in  $H_2O$ suspension with an alkali hydroxide in presence of  $HF_3$ , the mixture being heated to and maintained at the b.p. The pptd. K Al fluoride is filtered, washed, dried, and ground. W. J. W.

Insecticide. K. MARX and H. WESCHE, Assrs. to WINTHROP CHEM. Co. (U.S.P. 1,842,993, 26.1.32. Appl., 5.7.28. Ger., 9.5.28).—The active constituent is an aliphatic ether of an *ar*-tetrahydronaphthol. W. J. W.

Xanthates.—See III. Ca(NO<sub>3</sub>)<sub>2</sub>. Fertilisers.— See VII.

# XVII.—SUGARS; STARCHES; GUMS.

Sugar-beet presses instead of diffusion batteries. I. B. MINTZ and B. E. KRASILSHCHIKOV (Nauk. Zapiski Tzuk. Prom., 1931, 13, 447—468).—Losses of sugar in pulps can be decreased to 0.5-0.6% of the wt. of the beets by : pressing (85 atm.), steaming, pressing, maceration with 35% of juice (Brix 7.0), pressing, maceration with 15% of H<sub>2</sub>O, pressing, and repeating the last maceration and pressing. CH. ABS.

Volume of marc of normal weight of beets. I. B. MINTZ, A. K. KARTASHOV, and N. S. TROFIMOVSKI (Nauk. Zapiski Tzuk. Prom., 1931, 14, 483–494).— When hot-digestion is employed for the determination of sugar in beets the pulp and ppt. may adsorb sugar; extraction with  $H_2O$  (suction) must be applied as a control method. Methods for the determination of the vol. of hydrated marc based on the difference between the amount of sugar determined by digestion and that extracted by some other method must be rejected. Indirect determination of the vol. of marc occupied in a normal wt. of pulp leads to vals. varying between 1 and 2 c.c. CH. ABS.

Extraction of the juice from dried cossettes by means of cold diffusion. V. E. MALINOVSKI and N. K. PETROV (Nauk. Zapiski Tzuk. Prom., 1931, 13, 433—440). —Diffusion for 195 min. with  $H_2O$  at 23° gave good results; the charge capacity of each cell is increased and the juice withdrawn at 27—28° Brix, resulting in a 12% increase in the efficiency of the battery. The cold diffusion juice is treated with 2% CaO; after a second defecation and carbonatation no further treatment with kieselguhr or SO<sub>2</sub> was required. CH. Abs.

**Preservation of cane juices by refrigeration.** C. H. KARLSON and W. H. FOSTER (Proc. S. Afr. Sugar Tech. Assoc., 1932, 37—39).—Juice samples for analysis were placed in a refrigerator at —10° and were found to keep indefinitely in the frozen state; the frozen juice should be analysed soon after melting, as deterioration then sets in very rapidly. J. P. O.

Influence of organic non-sugars on molasses formation. R. CLAASSEN (Z. ver. Deut. Zucker-Ind., 1932, 82, 742-743).—A discussion. A. A. L.

Testing of bagasse. J. BRUNIQUEL and E. P. HEDLEY (Proc. S. Afr. Sugar Tech. Assoc., 1932, 13—16). —In the official method of the Association, the same result is obtained by drawing off the sample for analysis as soon as the boiling has finished, or by allowing to cool before drawing off. J. P. O.

Standard alkalinity and removal of calcium salts [in sugar manufacture]. A. M. PSHENICHNI and B. P. SHUMKOV (Nauk. Zapiski Tzuk. Prom., 1931, 14, 407-422).—When the natural alkalinity is low, Na<sub>2</sub>CO<sub>3</sub> (2Na<sub>2</sub>CO<sub>3</sub>: 1CaO) is added to the juice at the second defecation. When manufacture has been prolonged the Ca salts increase; boiling of the massecuite is difficult and the yield of molasses increases. CH. Abs.

Keeping qualities [in storage] of Natal [raw] sugar. L. BLACKLOCK (Proc. S. Afr. Sugar Tech. Assoc., 1932, 17—24).—It is possible to make a raw or cargo sugar which will, over a period of 2—8 months under good conditions, maintain its polarisation and realise its original commercial val. at the end of this time. Experience shows the following points must be considered in ensuring this object : (1) efficient juice clarification with a min. of insol. matter in the syrups ; (2) production of a hard strong grain in the pans ; (3) thorough purging of the massecuite so as to give a sugar having a safety factor < 0.25; strict attention to sanitary conditions around the centrifugals, filling bins, and bagging rooms. J. P. O.

Fertilisers etc. for sugar beet and cane.—See XVI. See also A., Oct., 1004, Lignosulphonic acid [and sucrose inversion].

## XVIII.—FERMENTATION INDUSTRIES.

Calculation of [extract in] original wort in beer analysis. H. SIEGFRIED (Mitt. Lebensm. Hyg., 1931, 22, 354—364; Chem. Zentr., 1932, i, 1588).—Balling's, Dolmens', and Saar's formulæ are satisfactory; the Swiss official formula is inaccurate. Determination of EtOH by distillation is more accurate than that by the use of the refractometer. A. A. E.

Malt extract and malted sweets. H. JESSER (Chem.-Ztg., 1932, 56, 662--663).--Malt extract contains about 5% of albumin and 0.5% of  $P_2O_5$  if pure : it appears to be frequently adulterated with potato starch. The albumin and  $P_2O_5$  contents in samples of malted sweets were about 25% of the above. Albumin may, however, be derived from brown sugar or maize syrup in part, and  $P_2O_5$  may of course be added in other forms. H<sub>2</sub>O insuch preps. can be determined by means of the pyknometer (drying at 103° results in decomp.). C. I.

Theories of fermentation and their practical application. H. L. HIND (J. Inst. Brew., 1932, 38, 391 -402).—A review is given of the theories of Harden and Young and of Neuberg with their applications to the manufacture of glycerol, COMe<sub>2</sub>, BuOH, and various by-products. The intimate connexion of fermentation with respiration and their stimulation is dealt with, and examples are quoted showing how the results of fermentation research have been applied to the solution of problems of human physiology. C. R.

Determination of volatile acids in wines. H. ASTRUC and A. CASTEL (Ann. Chim. Analyt., 1932, [ii], 14, 145—152).—A comparison of various methods has proved that the direct distillation of the wine, with or without dilution (Duclaux and Gayon), followed by titration of the distillate with 0.1N-NaOH against phenolphthalein gives results which are equally as satisfactory as those given by the more complicated and tedious method of defecation with CaO, reacidification with tartaric acid before distillation, and titration with CaOH against litmus (Semichon). H. J. D.

Fibre waste.—See V. Enzymes in tanning materials.—See XIV. Viticulture.—See XVI.

See also A., Oct., 1062, Separation of malt amylases. 1063, Separation of sucrose-splitting enzymes. Pectolase. Enzymic hydrolysis and synthesis of aspartic acid.

#### PATENTS.

Conditioning of water [particularly brewing water]. CHEM. FABR. BUDENHEIM A.-G. (B.P. 379,911, 4.11.31. Ger., 4.11.30).—An aq. solution of primary phosphate or mixture of primary phosphates is added to the H<sub>2</sub>O so that the resulting liquor has  $p_{\rm H} \ge 7.1$  at 18°. CO<sub>2</sub> is removed by stirring, aëration, or heating, and the pptd. matter is separated. C. R.

Preserving the taste of bottled beer by treating the bottles. H. VAN DE SANDT (B.P. 379,472, 18.7.31). —The beer is protected from the action of light or heat rays by covering the bottles with a combination of foils or coatings of cellulose hydrate or the like. The coverings may be coated with metal powder or white lacquer. [Stat. ref.] C. R.

Extracting proteolytic enzymes from fig, mulberry, papaya, pineapple, or banana trees. C. WADA (B.P. 379,205, 11.1.32. Jap., 17.1.31).—The clarified juice expressed from the branches, stalks, leaves, and fruits is acidified with AcOH and conc. at low temp. and reduced pressure. COMe<sub>2</sub> is added to the syrup, and 24 hr. later the pptd. enzyme is separated, washed with MeOH, and dried. C. R.

Production of lactic acid, [alcohol,] and other products by fermentation. S. D. WELLS, Assr. to PAPER MILL LABS., INC. (U.S.P. 1,838,599, 29.12.31. Appl., 26.7.28).—The liquor resulting from the cooking of straw or other fibrous plant material with a solution of alkali carbonate or sulphite so that the fibrous material is softened but not pulped is hydrolysed by heating with acid under pressure. Excess of acid is neutralised by CaCO<sub>3</sub>, and the liquor is fermented in the presence of lactic acid bacteria. U. R.

Recovering enzymes etc. Acidifying agent for baking purposes.—See XIX. Skin powders.—See XX.

## XIX.—FOODS.

Sterilisation and bleaching of whole grain. H. SIMONS (Mühlenlab., 1932, 49–51).—Details of bleaching whole grain with commercial NaHSO<sub>3</sub> are given, and the chemical reactions which take place when using solutions of commercial NaHSO<sub>3</sub> and Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> are dealt with. E. B. H.

Detection of rye flour in wheat flour, and of barley flour in rye and wheat flours. H. KÜHL (Chem.-Ztg., 1932, 56, 561-563).—Destructive criticism of the methods of Rudolph and Barsch (B., 1932, 239).

H. J. D.

Pasting of starch [in bread]. R. I. KATZ (Mühlenlab., 1932, 53-55).—The staling of bread in relation to the swelling and retrogradation of starch is discussed. The use of X-ray spectra is described (cf. B., 1931, 1069). E. B. H.

Heat-coagulation of milk. I. Variations in the compositions, heat-stability, and other tests of milks during the course of a lactation period. G. E. HOLM, B. H. WEBB, and E. F. DEYSHER (J. Dairy Sci., 1932, 15, 331–343).—No relationship was observed between the heat-stability of milk from individual cows, and the salt balance, buffer intensity,  $p_{\rm H}$ , titratable a didity, chemical composition, or rate of coagulation with rennin. "Rapid" tests of heat-stability commonly used are not satisfactory. A. G. P.

Coagulation of milk-protein and depression of the f.p. M. SATTO (J. Agric. Chem. Soc. Japan, 1931, 7, 498—506).—For an acidity of 0.18-0.20% the f.p. falls from  $-0.555^{\circ}$  to  $-0.58\%^{\circ}$ . For milk in which protein was coagulated by putrefaction it was  $-0.820^{\circ}$ . The depression was approx. proportional to the amount of lactic acid produced. With milk coagulated by rennin or heated at 96—10.5° for 2 hr. the f.p. did not change.

CH. ABS.

Effect of season on the reliability of the percentage of solids-not-fat in milk as calculated by formulæ. S. BARTLETT, J. GOLDING, and A. WAG-STAFF (Agric. Progress, 1932, 9, 95—98).—Richmond's formula gives with winter milk vals. 0.4% too high; Fleischmann's formula is less accurate. The sp. gr. of milk increases during approx. 12 hr. after milking, the rate and extent of the change being influenced by the temp. at which the milk is kept. CH. ABS.

Cryoscopy and refractometry [of milk]. H. ALFONSUS (Milch. Zentr., 1932, 61, 201-205).—The most convenient means of testing for adulteration of milk by  $H_2O$  is the determination of the *n* of the CaCl<sub>2</sub> serum. Cases of doubt must be decided by determining the f.p. E. B. H.

Public health significance of the growth of thermophilic bacteria in pasteurised milk. P. A. HENSEN (New York State Agric. Exp. Sta., Tech. Bull., 1932, No. 196, 16 pp.).—11 cultures of thermophilic bacteria grown in sterile skim-milk were fed to guineapigs. The animals increased normally in wt. both during and after the feeding experiments. The presence of thermophilic bacteria in milk is unimportant from the public health viewpoint. E. B. H.

Pasteurisation of milk by an electric current. C. G. KING (Trans. Electrochem. Soc., 1932, 62, 245–248).—Milk preheated to 49° is circulated at an automatically controlled rate between  $H_2O$ -cooled C electrodes across which a.c. at 220 volts is passing, thus raising the temp. to 72° without danger of local overheating and with min. exposure to air or metals. By passage through a heat interchanger and over tubes cooled by brine, the temp. is lowered to  $4 \cdot 5^\circ$  for bottling. Al is used for all pipes except those carrying brine. Commercial units of proved bacteriological efficiency are in operation. H. J. T. E.

Detection of washed, abrased, and oiled eggs. P. F. SHARP (Ind. Eng. Chem., 1932, 24, 941-946).— A method of detecting washed eggs is based on the observation that the material on the surface of the shell contains K and Cl which are removed by washing. Details for making the Co nitrite reagent for K are given. When micro-pptn. tests of the shell extract were made, the unwashed eggs gave tests for K and Cl, whereas the washed eggs did not. The effect of the following factors on the tests was investigated : washing solutions, oil-treating, source of the eggs, season, colour of shell, storage, sweating, scraping, sanding. Oiltreated eggs can be recognised by dipping a portion of the shell into a drop of Et<sub>2</sub>O and observing the oily ring after evaporation of the Et<sub>2</sub>O. Sand-blasted and scraped eggs are detectable by staining tests. W. J. B.

Freezing of water in fish muscle and in gelatin. J. H. MENNIE (Canad. J. Res., 1932, 7, 178-186).-The  $H_2O$  content (%) of the muscle of various fish which is frozen on cooling to  $-5^{\circ}$  to  $-20^{\circ}$  has been calc. from published calorimetric data. The influence of dissolved salts on the apparent quantity of H<sub>2</sub>O bound by the colloidal material present cannot be ignored. The observed heat capacities of 1.7% NaCl solutions, referred to 20°, at temp. between 0° and  $-65^{\circ}$ agree well with the vals. calc. from the sp. heats of the components, the heat of dissolution, and the f.-p. curve. The heat capacities of dry gelatin and of 20, 40, and 66% gelatingels have been determined; the percentages of  $H_2O$  remaining unfrozen at  $-20^\circ$  have been calc. and the results compared with those obtained by other workers. Prolonged storage (1 month) and repeated freezing and thawing do not alter the amount of H<sub>2</sub>O frozen. No further separation of ice occurs on lowering the temp. below  $-20^{\circ}$ . H. F. G.

Detection of traces of hexamethylenetetramine in fish-preserve media. A. VAN DRUTEN (Chem. Weekblad, 1932, 29, 501-504).- A sample (10 c.c.) is filtered, and after addition of 1 c.c. of 85% H<sub>3</sub>PO<sub>4</sub> and 5 g. NaCl is steam-distilled until 25 c.c. of distillate are obtained; this is tested for CH<sub>2</sub>O by peptone and FeCl<sub>3</sub>. If the test is positive, hexamethylenetetramine is confirmed in another sample by microscopical observation of the compound with HgCl<sub>2</sub>, or, alternatively, a 75-c.c. sample is rendered alkaline with CaO, filtered, and extracted with CHCl<sub>3</sub>; the extract is evaporated and the HgCl<sub>2</sub> test is applied to a HCl solution of the residue. The limit of sensitivity of this method is about 10 mg. per litre. Hexamethylenetetramine gradually decomposes in such preserves and cannot be detected 50 days after its addition; CH<sub>2</sub>O, however, may be detected by the first test mentioned above. H. F. G.

Tenderness of meat. II. Determination of period of ageing grade-A beef required to produce a tender quick-frozen product. D. K. TRESSLER and W. T. MURRAY (Ind. Eng. Chem., 1932, 24, 890-892; cf. B., 1932, 815).—Sirloin steaks aged for 4 days at  $1-3^{\circ}$  and then cut, packaged, quick-frozen, and stored at  $-18^{\circ}$  for a month or longer, when thawed are as tender as and of better flavour than adjacent steaks aged for 6 or 7 days at  $1-3^{\circ}$  and then tested immediately without freezing. Further refinements in the technique of testing meat with the perforating or cutting gauge and the penetrometer are described. W. J. B.

Spoilage of dressed ducks by sliminess. W. L. MALLMANN (J. Agric. Res., 1932, 44, 913-918).-Bacterial sliminess of dressed ducklings is prevented by immersion in brine. Sauerkraut from pretreated cabbage. C. H. KEIPPER, W. H. PETERSON, E. B. FRED, and W. E. VAUGHN (Ind. Eng. Chem., 1932, 24, 884–889).—In factory experiments an increase of 17—50% in the output of high quality product was obtained by washing the cabbage. By this treatment the no. of undesirable organisms was reduced and the desirable types were freer to develop. The use of chlorinated H<sub>2</sub>O affected the flavour of the product adversely. Inoculation of the washed cabbage with pure cultures of lactic acid bacteria gave, in general, further improvement, but this was insufficient to warrant its adoption in commercial practice. W. J. B.

Regulation of the sulphurous acid content of sulphited plant products. K. P. KARDASHEV (Izv. Zentr. Nauch.-isledov. Inst. pish. vkus. Prom., 1930, No. 1, 1-25).—For the determination of SO<sub>2</sub>, dried apples (10-20 g.) are kept in H<sub>2</sub>O for 2 hr.; this is diluted to 250 c.c. and 50 c.c. of the filtrate are decomposed with N-KOH (25 c.c.). After 15 min. H<sub>2</sub>SO<sub>4</sub> (1:3, 10 c.c.) and starch solution (1 c.c.) are added and the solution is titrated with I. For the determination of SO<sub>2</sub> in tomato purée, 5 c.c. in 25 c.c. of KOH are filtered, washed, and treated as above. CH. ABS.

Manufacture of grape juice. J. TESTA (Requind, 1932, 1, 56–58).—The process is described. Argentine grape juice had  $d^{15} 1 \cdot 06$ —1 $\cdot 12$  and contained EtOH 0, dry extract (100—105°) 150—300, total sugar 133—290, ash  $1 \cdot 8$ —3 $\cdot 8$ , acid as tartaric  $3 \cdot 3$ —6 $\cdot 8$ , acid  $0 \cdot 03$ —0 $\cdot 3$ , free SO<sub>2</sub>  $0 \cdot 01$ —0 $\cdot 03$ , combined SO<sub>2</sub>  $0 \cdot 002$ —0 $\cdot 03$  pts. per 1000. CH. ABS.

Relation of deterioration of orange juice to its iodine reducing value. M. A. JOSLYN and G. L. MARSH (Science, 1932, 76, 82-83).—Direct titration of orange juice with 0.01N-I gives a good indication of the extent of browning and of general deterioration in quality. The titration may also be an indication of the probable vitamin-C potency of the preserved juice.

Gravimetric and titrimetric determination of pectins. W. STRUBCZEWSKI (Przemysł Chem., 1932, 16, 135—137).—Higher vals. are obtained for the pectin content of fruit juices by the gravimetric than by the titrimetric method; in the latter method pectins are pptd. by EtOH and the liquid is filtered through collodion. The residue is hydrolysed by a known vol. of standard NaOH, excess of which is then determined by electrotitration. R. T.

Factors which determine the nutritive value (stock-carrying and fattening capacity) of untreated natural pastures. E. J. SHEEHY (Sci. Proc. Roy. Dublin Soc., 1932, 20, 325–346).—No consistent differences occur in the composition of the org. portion of the dry matter of herbage from soils of varying fertility. Cases of higher protein content in better pastures were due to a higher proportion of grass to weeds in the herbage. The Ca content of herbage did not vary with the soil type. Herbage from poorer soils had a slightly lower  $PO_4^{\prime\prime\prime}$  content than that from richer areas. The digestibility of typical plants was not affected by soil conditions. The feeding capacity

of pastures is indicated by the total dry-matter yield. A. G. P.

Formula for evaluating feeds on the basis of digestible nutrients. W. E. PETERSEN (J. Dairy Sci., 1932, 15, 293-297).—A formula, graph, and data for calculation are given. A. G. P.

Fibre waste.—See V. Radish oil. Tobacco-seed oil.—See XII. Malted sweets.—See XVIII. Prep. of lecithin.—See XX.

See also A., Oct., 1055, Determination of [H'] of milk. 1069–70, Vitamins (various).

#### PATENTS.

[Preparation of a] baking composition. F. W. THIELE, ASST. to HANSEATISCHE MÜHLENWERKE A.-G. (U.S.P. 1,843,051, 26.1.32. Appl., 1.9.28. Ger., 14.6.28). —The incorporation of 1% of vegetable lecithin in a bread dough softens the gluten, promotes yeast action, and increases the yield of bread. E. B. H.

Production of bakery products. HANSEATISCHE MÜHLENWERKE A.-G., and B. A. REWALD (B. REWALD) (B.P. 378,989, 27.5.31).—The addition of 1—4% of lecithin to bread, cake, and pastry mixes improves the keeping quality of the products and enables the proportion of egg used to be reduced. E. B. H.

Acidifying agent for baking purposes. W. SCHROEDER (B.P. 379,678, 17.2.31. Ger., 12.8.30).— A nutrient liquid, consisting of skim milk or a wort prepared from cereals, is fermented by acid-producing bacteria in the presence of substances which will neutralise the acid as produced. The salts formed are decomposed by acids and the acid liquid is mixed with flour, dried, and then incorporated in the bread dough. [Stat. ref.] E. B. H.

Cereal food preparations. F. A. LINTON (B.P. 379,412, 9.6.31. Can., 13.6.30).—A mixture of grains is subjected to ultra-violet radiation, ground, and heated to 65° with the addition of steam and inorg. salts, the product being finally dried at 65°. E. B. H.

Manufacture of breakfast foods. E. HILL (B.P. 379,944, 18.12.31).—A mixture of 99% of rolled oats, 1% of bran, and 0.06% NaCl is boiled under pressure, dried, and packed. E. B. H.

Treatment of milk. A. H. JOHNSON and S. M. WEISBERG, ASSTS. to RESEARCH LABS. of NAT. DAIRY PRODUCTS CORP., INC. (U.S.P. 1,841,184, 12.1.32. Appl., 30.6.30).—To make milk more readily digestible, it is homogenised at pressures adjusted according to the predetermined curd tension of the milk. E. B. H.

Determining the physical curd character of milk. R. L. HILL, ASST. to UTAH STATE AGRIC. COLLEGE (U.S.P. 1,840,392, 12.1.32. Appl., 13.10.28).—A process for determining the curd tension of milk is described. A suitable coagulant is added and the resistance of the curd formed to the passage of a knife is determined.

E. B. H.

Food product. S. MUSHER (U.S.P. 1,841,842, 19.1.32. Appl., 7.12.27).—Rancidity of milk products is substantially inhibited by admixture with vegetable butter or cake, especially crushed sesamé seed. Deficiencies in

L. S. T.

vital food elements are made up, and the product has a pleasant flavour. The crushed seed is mixed with the milk before or after spray-drying. E. B. H.

(A) Dairy products. (B) Machines for pasteurising dairy or other plastic edible products. (A) J. H. BAUMGARTNER, (B) G. C. ALSTAD and J. H. BAUMGARTNER (B.P. 378,375—6, 29.1.31).—Plastic dairy products, such as cheese, are pasteurised and emulsified by apparatus designed to heat the material to pasteurising temp. by frictional heat, produced by directing the product through narrow constrictions between moving surfaces. E. B. H.

**Ripening or curing of cheese.** H. L. H. WATERS (B.P. 379,797, 25.6.31. Can., 27.6.30).—Cheese is matured by maintaining in an atm. the temp. and humidity of which are co-ordinated with the  $H_2O$ content of the cheese. The cheese is periodically turned and the rind treated with a solution of Na<sub>2</sub>HPO<sub>3</sub>. E. B. H.

Production of margarine and like edible fat emulsions. EMULSOL CORP., Assees. of (A) A. K. EPSTEIN and (A, B) B. R. HARRIS (B.P. 378,372–3, 29.1.31. U.S., [A] 31.10.30, [B] 27.2.30).—(A) A paste emulsion is prepared with milk (or fat) with a mixture of an anti-spattering compound, containing hydrophile and lipophile groups (cf. B.P. 366,909; B., 1932, 526), particularly Na monostearin sulphoacetate, and an "anti-weeping" compound [cf.(B)], and the paste is kneaded into the margarine. (B) 0.5—1% of an alkyl or acyl (R=C<sub>6</sub> or higher) derivative of a polyhydroxycompound, of the type  $H_w C_x(OH)_y(OR)_z$ , where y is  $\geq z (e.g., monoglycerides, diethylene glycol monostearate)$ is incorporated as an anti-weeping agent to check sweating of the margarine etc. [Stat. refs. to (A) and (B).] E. L.

Oil emulsions for use in production of food. J. SCHWARZKOPF (B.P. 378,273, 6.11.31).—An emulsion is prepared with  $H_2O$ , locust-bean flour, oil, and a little NaOH, and may be used in the prep. of food emulsions such as mayonnaise, artificial cream, etc. E. B. H.

Preventing discoloration of vegetables. B. R. HARRIS (U.S.P. 1,842,720, 26.1.32. Appl., 11.6.28).— An acid sulphite or other  $SO_2$ -liberating chemical or  $H_2O_2$  is used as an anti-catalyst to prevent the darkening which otherwise occurs in ground horseradish, potatoes, etc. E. B. H.

Preparation of [vegetable or fruit] extracts. D. K. TRESSLER, ASST. to FROSTED FOODS CO., INC. (U.S.P. 1,840,798, 12.1.32. Appl., 5.9.30).—An improved process for extracting flavours from fruit such as strawberries is claimed. The frozen comminuted fruit is extracted in vac. with a suitable solvent (such as EtOH). Apparatus is described. E. B. H.

Preparation of disinfected fruit. H. B. SLATER, ASSR. to FOOD MACHINERY CORP. (U.S.P. 1,842,696, 26.1.32. Appl., 12.9.30).—After cleaning and disinfecting the fruit in a 0.1-0.5% solution of NaOCl or other alkaline hypochlorite, at 35-40°, it is, if necessary, passed upwards through a layer of molten paraffin wax or the like to give a waxy coating. E. B. H.

Preservation of orange juice. D. M. SMALLE, Assr. to Dr. P. PHILLIPS CO. (U.S.P. 1,838,517, 29.12.31. Appl., 4.12.30).—Flavour, colour, and vitamins may be maintained indefinitely in sealed containers by heating the juice to 50—60° for 5—15 min. and then raising the temp. to 65—75° as the juice passes through a pipe to the filling device. E. B. H.

Jelly compound. H. T. LEO, Assr. to G. SMITH (U.S.P. 1,842,757, 26.1.32. Appl., 4.2.27).—A jellymaking product may be prepared by heating hydrated sugar (e.g., glucose) to a temp. at which the  $H_2O$  of crystallisation is driven off, mixing into the molten anhyd. sugar a solution containing an edible acid, cooling, pulverising the mass, and intimately mixing with powdered pectin. E. B. H.

Manufacture of chewing-gum base. W. W. TRIGGS. From SWEETS LABORATORIES, INC. (B.P. 378,073, 6.5.31).—Milled unvulcanised rubber is mixed with a hard fat, a resinous material, and  $H_2O$  in a steam-heated pan. The presence of  $H_2O$  keeps the temp. below 115° and prevents the development of a "burnt rubber" flavour. E. B. H.

Recovering products rich in mineral salts and enzymes and also containing vitamin-B. B. W. DEDRICK (U.S.P. 1,842,933, 26.1.32. Appl., 30.4.29).— The bran and fine middlings of the residue left after making flour from wheat are extracted with  $H_2O$  at approx. 38° with alternate agitation and rest periods. The mash is pressed and the separated fluid, carrying suspended flour, cerealin, and other food substances which had been attached to the inner sides of the bran, is allowed to settle. The supernatant liquid is siphoned off and the residue dried. C. R.

Drying fruit juices.—See I. Determining fat in milk. Emulsions. Washing etc. agents.—See XII.

# XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Reactions of new anæsthetics. H. SZANCER (J. Pharm. Chim., 1932, [viii], 16, 239–241).—The formation of MeOBz on heating with conc.  $H_2SO_4$ may be used to distinguish psicaine from novocaine and tutocaine. A. L.

[Preparation of] lecithin from soya-beans. G. EICHGORN, A. MILSKI, and E. KALASHNIKOV (Masloboino Zhir. Delo, 1931, 11, 26-38).-For the determination of lecithin the ground material (1.5-2 g.) is extracted (Soxhlet) with EtOH for 5-6 hr., and then with Et<sub>2</sub>O for 3-4 hr. After partial evaporation of the solvents the residue is treated with H<sub>2</sub>SO<sub>4</sub> and conc. H<sub>2</sub>O<sub>2</sub> in a Kjeldahl flask and the P content determined colorimetrically; lecithin contains 3.90% P. Lecithin can be completely extracted with  $EtOH + C_6H_6$  (9:1). On distilling off the solvents, first up to 54.85° (EtOH +  $H_2O$  +  $C_6H_6$ ) and then up to  $68 \cdot 25^{\circ}$ (EtOH +  $C_6H_6$ ), the alcoholic residue separates on cooling into two layers, the lower containing the oil and the upper consisting of the alcoholic solution of lecithin and the free fatty acids. The crude lecithin is purified by pptn. with COMe2. The carbohydrate accompanying the lecithin with the EtOH + C6H6 extract in Bollmann's process is sucrose. CH. ABS.

Separation of pure santonin. P. S. MASSAGETOV (Khim. Farm. Prom., 1932, No. 2-3, 68-71).—The flowers (5 g.) are triturated with CaO (1 g.), and boiled with  $H_2O$  (250 c.c.); the filtrate is acidified with HCl, cooled, extracted with CHCl<sub>3</sub>, rendered alkaline with 4% NaOH, and again extracted with CHCl<sub>3</sub>. The extract is filtered through animal C and evaporated; the residue is dissolved in a min. quantity of EtOH, treated with boiling  $H_2O$  (100 c.c.), evaporated (50%), allowed to crystallize and filtered. The solid is trans-

allowed to crystallise, and filtered. The solid is transferred with CHCl<sub>3</sub> to a tared flask, dried, and weighed. CH. ABS.

Microchemical identification of alkaloids. F. AMELINK (Pharm. Weekblad, 1932, 69, 1009–1011).— Acidified solutions of larocaine (A., 1931, 387) yield no ppt. with PtCl<sub>4</sub>, but if NaI is added a black ppt. which crystallises only slowly is formed. With AuCl<sub>3</sub> a yellow amorphous ppt. is formed, and on addition of NaBr (in acid solution) brown dichroic needles separate.  $K_4Fe(CN)_6$  yields a characteristic ppt. of flat rectangular crystals, and may be employed to identify the alkaloid in 0.1% solution. HgCl<sub>2</sub>, Dragendorff's reagent, and KOH yield only amorphous ppts. H. F. G.

Fixed oils [castoreum]. A. Yost (Requind, 1932, 1, 66–67).—Castoreum contains cholesterol, a volatile oil, castorin  $(2 \cdot 5\%)$ , resin, an extract sol. in H<sub>2</sub>O and Et<sub>2</sub>O, albumin, fat,  $(NH_4)_2CO_3$ , CaCO<sub>3</sub>, Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>, CaSO<sub>4</sub>, K<sub>2</sub>SO<sub>4</sub>, MgSO<sub>4</sub>, and H<sub>2</sub>O. CH. ABS.

Analysis and adulteration of citric essences. A. BRUNETTI (Requind, 1932, 1, 46–48, 72–73).—A discussion. Ch. Abs.

Detecting cyclic amines in medicaments.—See III. Tobacco-seed oil.—See XII. Pyrethrum flowers.—See XVI.

See also A., Oct., 1026, Halogen derivatives of monohydroxydiphenylmethane. 1046, Alkaloids of *Angostura* bark. 1047, Antimalarials from cotarnine. 1048—9, Alkaloids of *Sinomenium* and *Cocculus*. 1051, Determination of adrenaline. 1052, Reagent for purine derivatives. Determination of As in organo-As derivatives. 1058, Na iodobismuthite prep. 1069—70, Vitamins (various).

## PATENTS.

Skin powders. E. DonÁTH (B.P. 378,888, 11.4.31. Czechoslov., 14.4.30).—A mixture of one or more amylolytic or proteolytic enzymes with an aq. sol. substance such as NaOBz, NaCl,  $Na_2B_4O_7$ , or albumin is incorporated with a carrier substance such as starch, tale, or ZnO. C. R.

**Production of foams.** A. KARRETH (B.P. 379,892, 9.10.31).—Two salve-like materials, both of which may have tragacanth, agar, or similar material as a basis and also contain a disinfectant, medicinal, or other active substance, consist of (I) an acid or acid-reacting salt, e.g., tartaric acid,  $NH_2OH$ , HCl,  $NH_4Cl$ , and (II) a gasevolving salt such as  $NaHCO_3$ ,  $Na_2S$ ,  $NaNO_2$ , etc. Both components, which are kept separate until the time of their use, contain the necessary amount of  $H_2O$  to form a foam without any external liquid. E. H. S.

Therapeutic iodine solution. O. P. AMEND, ASST. to EIMER & AMEND (U.S.P. 1,841,694, 19.1.32. Appl., 16.5.30).—Stable, practically neutral, aq. solutions of I, incapable of pptg. albumin, are prepared by adding I in sufficient amount to produce a neutral reaction to an aq. solution of an alkali, *e.g.*, NaOH, and a non-nitrogenous protective colloid, *e.g.*, Irish moss. E. H. S.

Manufacture of sterilised surgical sewing materials. SÄCHSISCHES SERUMWERK A.-G., and M. KNORR (B.P. 379,421, 12.6.31).—Raw material, *i.e.*, catgut, silk, or silk-worm gut, is incubated under anaërobic conditions in a medium to which has been added sterile portions of the organs of man or animals, whereby [H<sup>\*</sup>] and O concest analogous to those in the human body are produced and responsible to those in the human body are produced and responsible to those in the human body are produced and responsible to the series of the retarded. The material is freed from  $H_2O$  and sterilised. *E.g.*, iodised catgut is immersed in baths of decreasing concest of EtOH, then incubated in the medium, and  $H_2O$  removed by treatment in baths with ascending EtOH content. Incubation and sterilisation may be repeated several times. E. H. S.

Manufacture of halogenated products of [2:6-]diaminopyridine. E. T. TISZA and B. JOOS, ASSTS. to PYRIDIUM CORP. (U.S.P. 1,830,301, 3.11.31. Appl., 7.5.29).—2:6-Diacetamidopyridine, m.p. 205°, is brominated in H<sub>2</sub>O at 85° in presence of FeCl<sub>3</sub> and the product is hydrolysed to 3-bromo-2:6-diaminopyridine, m.p. 174—175°. C. H.

Manufacture of hydroxyphenylazo- $\alpha\alpha$ - [benzeneazo-2 : 6-]diaminopyridines. E. T. TISZA and B. JOOS, ASSTS. to PYRIDIUM CORP. (U.S.P. 1,830,300, 3.11.31. Appl., 18.3.29).—An aminophenol is diazotised and coupled with 2 : 6-diaminopyridine to give therapeutically valuable azo compounds sol. both in alkaline and in acid liquids. Compounds from p- [m.p. 218—220° (decomp.)], m- (m.p. 212—213°), and o- [m.p. 220—222° (decomp.)] -aminophenols are described. C. H.

Manufacture of polymethylenediguanidine compounds. W. H. LEWERS. From ANTICOMAN GES.M.B.H. (B.P. 376,806, 7.4.31).—Deca- or other poly-methylenediamine is melted with guanidine thiocyanate at 130— 160° and the product, pptd. from EtOH by  $K_2CO_3$ , is converted into its tartrate, phosphate, or citrate. The salts are non-toxic and are stable in the stomach, being assimilated in the small intestine; they reduce the sugar in the blood without the ill-effects of guanidine salts. C. H.

Synergised hypnotic and sedative [containing magnesium]. M. L. CROSSLEY, ASST. to CALCO CHEM. Co. (U.S.P. 1,830,313, 3.11.31. Appl., 17.4.28).— Barbituric acid derivatives, *e.g.*, Na barbital, are more active when mixed with a Mg compound, *e.g.*, MgCl<sub>2</sub> or MgO. C. H.

Manufacture of neutral solutions of complex metallic compounds suitable for injection. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 376,346, 9.4.31).—Discoloration on sterilisation at  $p_{\rm H}$  6.5—7.5 is prevented or minimised by incorporation of sulphites, e.g., Na metabisulphite (0.1%), with the complex metal salts of aromatic polyhydroxy-compounds, e.g., Na antimonyl- (or bismuthyl- or manganese-)pyrocatecholdisulphonate, and Na antimonylprotocatechuate.

Obtaining and separating physiologically active substances from placenta. I. G. FARBENIND. A.-G. (B.P. 378,647, 19.8.31. Ger., 20.8.30).—The solvent-free residue remaining after treatment of placenta with org. fat solvents is subjected to hydrolysis and the active substances (inducing precocious sexual maturity) are isolated from the reaction mixture. E. H. S.

Pyridine double compounds.—See III. Ergosterol.—See XII.

# XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Determination of silver halides in photographic printing papers. S. FUKUSHIMA and C. HORIE (Waseda Appl. Chem. Soc. Bull., 1932, 16, 1--6).--The Ag halides are extracted twice with 10% aq. NH<sub>3</sub> with intermediate washing with H<sub>2</sub>O; the extract is partly evaporated and then boiled with HNO<sub>3</sub>. The ppt. is washed twice with EtOH by decantation and the residue, after treatment with H<sub>2</sub>O, boiled with HNO<sub>3</sub> and filtered (Treadwell tube). The wts. of AgBr and AgCl, respectively, are obtained indirectly. Since printing papers contain no AgI, this method can replace the cyanometric method. CH. ABS.

See also A., Oct., 1006, Latent image formation.

#### PATENTS.

Photographic material. I. G. FARBENIND. A.-G. (B.P. 379,934, 7.12.31. Ger., 6.12.30).—Photographic material comprising several emulsion layers, superimposed and dyed with dyes corresponding to the final colours in the picture, is modified by treating one or more layers with filter dyes, which are easily removed during development, the spectral absorption of which roughly corresponds to the permeability of the layer in question. These filter dyes are added in such quantities that the gradations of the layers are adapted to one another. Suitable dyes are, *e.g.*, Me-blue, acid fuchsin, and tartrazine, to modify the sensitivity to red, green, and blue light, respectively. J. L.

Sensitising photographic emulsions. KODAK, LTD., Assees. of L. G. S. BROOKER (B.P. 378,455, 12.3.31. U.S., 12.3.30).—Dyes sensitising Ag halide emulsions in the region 440—560 mµ are 3 : 4- or 5 : 6-benzthioisocyanine salts (e.g., iodide) and their 1': 2-Et<sub>2</sub> derivatives. J. L.

Photographic films. KODAK, LTD., Assees. of A. A. YOUNG (B.P. 379,984, 16.2.32. U.S., 16.2.31).—An antihalation layer consists of carnauba wax or  $C_{10}H_5Cl_3$ , with or without an intermediate sol. layer between the wax and the film. A suitable black dye is contained by either or both layers. J. L.

[Non-inflammable] films for photographic purposes. W. M. STILL & SONS, LTD., and A. G. ADAMSON (B.P. 378,394, 5.3.31).—A hardened (insol.) gelatin core layer is coated on both sides with a thin layer of rubber and an outer layer of celluloid or collodion varnish to take the sensitised emulsion. J. L.

Simultaneous development to different gradations of several silver halide emulsion layers applied on different supports. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 379,820, 21.7.31).— One of the exposed emulsions is pretreated with Na<sub>2</sub>CO<sub>3</sub> or K<sub>2</sub>CO<sub>3</sub> solution (0.5-1%), or with a hardening agent, e.g., 2% CH<sub>2</sub>O solution; the two films may then be developed together in a developer of flat action, e.g., p-methylaminophenol with sulphite. The pretreated film will develop with considerably steeper gradation. J. L.

Obtaining contrasts with regenerated cellulose by means of light. S. G. S. DICKER. From N. V. PHILIPS' GLOEHLAMPENFABR. (B.P. 379,715, 25.4.31).— Regenerated cellulose products, e.g., artificial silk, Cellophane, are soaked in  $K_2Cr_2O_7$  solution, dried, and exposed under a templet etc. The unexposed portions absorb dyes well, except mordant dyes, which are absorbed by the exposed portions (Cr<sup>--</sup> salts). Twocolour effects can thus be obtained. [Stat. ref.] J. L.

Indirect reproduction of colour photographs. B. GÁSPÁR (B.P. 379,679, 26.2.31. Ger., 1.3.30) .---Esters, ester salts, or ethers of leuco-compounds of vat dyes are incorporated in Ag salt emulsions ; these leucocompounds, e.g., K indoxyl sulphate, diacetylindigowhite, sulphate esters of 6:6'-dibromo-4:4'-dimethylbisthionaphthenindigo, are such as are unaffected by the production of the Ag image, and can be subsequently converted into the dye by oxidation or reduction processes. These processes are induced by converting the Ag into oxidising etc. compounds, e.g., PbCrO4, Ag<sub>4</sub>Fe(CN)<sub>6</sub>, and treating with HCl, alkali, etc. solutions. Single-solution treatment with K chlorochromate may be used in conjunction with the sulphate esters specified above. Various modifications of the processes are described. J.L.

Coatings for films.—See XIII.

# XXII.—EXPLOSIVES; MATCHES.

Evolution of gas during manufacture of trinitrotoluene. K. NEDERER (Z. ges. Schiess- u. Sprengstoffw., 1932, 27, 217—222).—The gases evolved in the nitration process were condensed by liquid air and separated by fractional distillation. In the manufacture of  $C_6H_3Me(NO_2)_2$  from  $o-C_6H_4Me\cdotNO_2$  there is loss of HNO<sub>3</sub> by reduction to N<sub>2</sub>O or N<sub>2</sub>. On further nitration of  $C_6H_3Me(NO_2)_2$  to  $C_6H_2Me(NO_2)_3$ , oxidising reactions become apparent by evolution of CO and CO<sub>2</sub>. This oxidation is of  $C_6H_3Me(NO_2)_2$  rather than of  $C_6H_2Me(NO_2)_3$  and its extent and the proportions of CO and CO<sub>2</sub> depend on temp. and time. Below 75° there is little development of gas. Notwithstanding the formation of CO<sub>2</sub>, there is little reduction of HNO<sub>3</sub> to N<sub>2</sub>O or N<sub>2</sub>, although some occurs in all nitrations. The presence of  $C(NO_2)_4$  is indicated. W. J. W.

Chemical stability of nitroglycerin powders. L. METZ (Z. ges. Schiess- u. Sprengstoffw., 1932, 27, 188—191).—The merits of various stability tests (B., 1932, 705) are summarised. Qual. tests, e.g., Abel and ZnI<sub>2</sub>-starch tests, give appreciably different results from others. The Me-violet test is influenced by volatilised nitroglycerin. Results obtained by storage tests at  $132^{\circ}$ ,  $100^{\circ}$ , and  $75^{\circ}$  agree. The high temp. of the first-mentioned is a disadvantage, however, and the test does not discriminate sufficiently. The last two give a good idea of the stability in a short time, the  $75^{\circ}$  test being specially suitable for distinguishing stable from unstable powders. Brunswig's method is unsuitable for very unstable powders, and for comparison by it,

CL. XXIII.-SANITATION; WATER PURIFICATION.

powders must have a similar physical condition. In Mayrhofer's test, the hydrolysing effect of  $H_2O$  vapour at 132° tends to imply instability. Hansen's test gives results in agreement with the 75° test. It may be used in conjunction with the 100° test, and in doubtful cases may be supplemented by the Mayrhofer test. The behaviour of powders on heating at 90° with  $H_2O$ gives valuable information. There appears to be a relation between stability and explosion temp., the latter decreasing with rising stability. W. J. W.

The Trauzl lead-block test [for explosives]. P. NAOUM (Z. ges. Schiess- u. Sprengstoffw., 1932, 27, 181-184, 229-232, 267-271).-If the test is carried out by Neubner's method, and provided there is no loss of sensitiveness through compression, expansion is proportional to the sp. pressure. The ratio of sp. pressure to expansion is only slightly influenced by the max. detonation velocity, but the latter does exert a certain effect in altering the ratio with explosives of very different velocities. Given sufficient sensitiveness, the density of an explosive bears no functional relation to the expansion, but compression of a cryst. explosive lowers its sensitiveness and capacity for initiation, and thereby reduces the expansion. With very sensitive and with cast explosives, the effect of compression is noticeable only when it is very high and thus reduces the sensitiveness extensively. Loss of sensitiveness may be overcome by increasing the tamping. The relation between expansion and heat of explosion, between compression and expansion for cryst. aromatic NO<sub>2</sub>-compounds and cryst. nitric esters and nitroamines, respectively, and between expansion and density, and the influence of compression on powder explosives are tabulated. W. J. W.

See also A., Oct., 1051, Determination of C and H in explosive liquids.

#### PATENTS.

Priming mixture. J. D. MCNUTT, Assr. to WIN-CHESTER REPEATING ARMS CO. (U.S.P. 1,842,556, 26.1.32. Appl., 5.10.27).—The mixture contains Hg fulminate, a metal chromate (with or without a metal peroxide) as the oxidising agent, and a nitrated org. compound. A suitable composition consists of Hg fulminate 25—40, BaCrO<sub>4</sub> 5—10, PbO<sub>2</sub> 5—10, and tetranitroaniline 3—15%, with 15—25% of ground glass if desired.

#### W. J. W.

Propellent powders. R. G. WOODBRIDGE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,838,346-7, 29.12.31. Appl., [A] 16.6.28, [B] 22.4.29).-(A) Stannous phthalate or a Sn salt of another org. acid, e.g., SnC<sub>2</sub>O<sub>4</sub>, is incorporated in the powder as a muzzle-flash preventive, the powder being subsequently coated with a deterrent material, e.g., diphenyldimethylurea. (B) A non-hygroscopic alkali salt, such as K<sub>2</sub>SO<sub>4</sub>, preferably incorporated in a colloid derived from a cellulose derivative, is added to propellent powders comprising nitrocellulose, a polynitrotoluene, and a dialkyl phthalate, the amount of the alkali salt being insufficient to eliminate muzzle flash in certain guns. Excessive evolution of smoke in daylight firing is therefore avoided, but for night firing a further quantity of the salt may be added so as to suppress muzzle flash. W. J. W.

Charge for high-explosive shells. N. C. BOYD, Assr. to HERCULES POWDER Co. (U.S.P. 1,840,431, 12.1.32. Appl., 19.1.28).—The m.p. of shell-filling compositions, *e.g.*, amatol, is reduced by adding a hydrated salt, such as  $Mg(NO_3)_2$ ,  $6H_2O$ . A mixture of 20% of trinitrotoluene and 80% of  $NH_4NO_3 +$  $Mg(NO_3)_2$ ,  $6H_2O$  (90:10) melts at 127°. W. J. W.

Detonating pyrotechnical composition. A. BLANC (B.P. 379,910, 4.11.31. Fr., 7.11.30).—A device for producing repeated detonations on impact comprises a hard substance coated with a mixture of  $P_4S_3$ , MgCO<sub>3</sub>, CaCO<sub>3</sub>, KClO<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, and a binding agent. W. J. W.

# XXIII.—SANITATION; WATER PURIFICATION.

Detection of ethylene oxide in residual gas. W. DECKERT (Angew. Chem., 1932, 45, 559-562).— The gas is led through conc. aq. NaCl;  $(CH_2)_2O$  reacts, liberating OH', which is titrated. Phenolphthalein as indicator permits the detection of 0.5 mg.  $(CH_2)_2O$ per litre of air; 0.1 mg. per litre can be determined with bromothymol-blue (cf. B., 1931, 180). E. S. H.

Behaviour of gas-mask charcoal towards phosgene and chlorine. J. B. NIELSEN (Z. ges. Schiessu. Sprengstoffw., 1932, 27, 208—211, 244—248, 280— 284).—Resistances of various wet and dry charcoals to the penetration of  $COCl_2$  and HCl are tabulated, the effects of variations in the amount and moisture of the charcoal, the humidity of the air, and the concn. of the  $COCl_2$  being indicated. W. J. W.

Field tests of quality of drinking water. V. A. SPASSKI (Voyenno-Med. Zhur., 1931, 2, 122–132).— A discussion. CH. Abs.

Straw-board effluent.—See V. Bacteria in milk.—See XIX.

## PATENTS.

Sewage treatment [sludge digestion]. (A) A. M. BUSWELL and S. L. NEAVE, (B) A. M. BUSWELL, ASSTS. to BOARD OF TRUSTEES OF UNIVERSITY OF ILLINOIS (U.S.P. 1,838,474—5, 29.12.31. Appl., 9.5.29).—(A) A twostage process is claimed in which the sludge after a primary, rapid, acid-digestion stage is passed into a much larger secondary tank to complete the alkalinedigestion stage. The primary tank is fitted for gas collection and means for scum removal etc. (B) A form of sludge-digestion tank is claimed which includes means for gas collection and for withdrawing a portion of the upper layer of liquid and distributing it over the surface as a means of preventing or removing accumulations of scum. C. J.

Incineration of refuse and similar waste materials. O. UHDE and V. KOLE (U.S.P. 1,838,596, 29.12.31. Appl., 22.1.29. Ger., 3.8.26).—The refuse charged at the periphery of a shaft furnace is ignited at the centre and subjected to a pulsating blast of air, whereby the fine refractory particles are carried away and later separated in a dust chamber. C. J.

Production of disinfectants. A. L. MOND. From KALI-CHEMIE A.-G. (B.P. 380,077, 4.6.31).—See U.S.P. 1,823,095; B., 1932, 578.

Oil from sewage.—See XII.