# BRITISH CHEMICAL ABSTRACTS

# **B.**—APPLIED CHEMISTRY

SEPT. 16 and 23, 1932.\*

# I.-GENERAL; PLANT; MACHINERY.

Heat transmission to liquids flowing in pipes. T. K. SHERWOOD and J. M. PETRIE (Ind. Eng. Chem., 1932, 24, 736—745).—Heat transfers from a hot-water jacket to a Cu pipe through which kerosene,  $C_6H_6$ , and  $COMe_2$  were flowing, and from steam to  $H_2O$  and  $Bu^{\alpha}OH$ , were determined, using both viscous and turbulent flow. Results with turbulent flow agreed well with the Dittus and Boelter equation founded on previous tests with the heating of air and oils. A comparison of results with the vals. calc. from Prandtl's equation showed irregularities in the vals. of his velocity ratio rand the difficulty of obtaining this figure detracts from the practical val. of this equation. C. I.

Relation between heat transfer and fluid friction. E. V. MURPHREE (Ind. Eng. Chem., 1932, 24, 726-735). -A method of calculating heat transfers for fluids flowing turbulently in pipes is based on the assumption that the eddy currents increase from 0 at the wall to a const. val. over the main body of the pipe, the region over which the eddy currents vary being termed the film. An equation for turbulent flow is derived from the equations of Reynolds and others, and it is assumed that the "eddy viscosity" varies with the cube of the distance from the wall in the film. This connects the ratio of the film thickness with the pipe diam. and the ratio of eddy viscosity to true viscosity with Fanning's fluid friction factor and the velocity ratios. It is assumed that the temp. gradient across the pipe is not such as to cause any appreciable variation in the physical properties of the fluid. Under conditions of practice heat-transfer coeffs. are almost independent of pipe length, from which it is shown that the main resistance to heat transfer is in the film, the temp. gradients in the main body of fluid being negligible. Experimental data on gases, H<sub>2</sub>O, and oils are compared C. I. with the results of the equations.

Boiler accidents due to incomplete study of [alkaline] feed water. A. STEOPOE (Bul. Chim. Soc. Române Stiin., 1929, 32, 19—27).—Deep well waters in the Braila district of Rumania contain considerable proportions of NaHCO<sub>3</sub> and of org. matter, including easily hydrolysed Na humates. Such waters are unsuitable for boiler use, producing priming and at higher pressures a heavy deposit of org. matter resulting from the decomp. of humates. If such water must be used it should be preheated, treated with CaO and  $Fe_2(SO_4)_3$ , and then freed from Ca by zeolite or alkali treatment. C. I.

PATENTS.

Muffle furnace. P. MÖHRING, Assr. to F. KRUPP A.-G. (U.S.P. 1,833,088, 24.11.31. Appl., 20.8.30. Ger., 8.11.29).—The muffle is heated by metallic bells placed over flues in a bottom of ceramic ware. The bells at the sides are tall and practically form part of the walls; the intermediate ones are mere lids and the goods are placed over them. B. M. V.

Smelting apparatus. A. W. WAERN, Assr. to CHAMPION FIBRE Co. (U.S.P. 1,833,386, 24.11.32. Appl., 15.3.30).—A rotary drum furnace discharges into a fixed vertical furnace. The claims refer to the watercooling of the drum and gas-tight connecting ¬-piece between the drum and the fixed furnace. B. M. V.

Chemical heating appliances. R. S. FERGUSON (B.P. 375,521, 5.6.31).—In a heater utilising the latent heat of crystallisation of a supersaturated solution, a valve for admitting air only when desired and for exhausting air or vapour when excess pressure arises is described. B. M. V.

Heat exchanger. C. R. EWING and T. MONTGOMERY, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,833,871, 24.11.31. Appl., 3.1.29).—The inner fluid makes a large no. of passes in series through groups of vertical tubes and the outer fluid makes one pass horizontally. B. M. V.

Tubular heat exchangers for use in oil-fuel installations and applicable also for use as feedwater heaters, evaporators, condensers, and coolers. T. T. BROWN, and SWINNEY BROS., LTD. (B.P. 375,132, 28.8.31).—The tube-plate cover is surrounded by an outer cover and used as additional heat-transmitting surface. A scraper for the outside of the tubes is described. B. M. V.

Rock crushers of the gyratory or other types. L. MELLERSH-JACKSON. From TRAYLOR ENG. & MANUFG. Co. (B.P. 375,520, 4.6.31).—The crushing space is divided into zones by imaginary horizontal planes spaced in such a way that the distance apart of the jaws when open on a plane below is equal to the distance on the next plane above when the jaws are closed, and the crushing space must be so curved and tapered that the vol. of the zones so defined does not decrease in a downward direction. B. M. V.

Process and apparatus for grinding or crushing. N. NIELSEN (B.P. 375,480, 23.4.31).—A grinding mill is discharged partly by an air current working in a closed circuit through separators etc., and partly by a mechanical conveyor (e.g., a worm) collecting material discharged through apertures in the mill shell and delivering

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Filtration.—See XVII.

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

them to a screening machine, the oversize being returned to the mill. B. M. V.

Hammer mill. M. McMILLAN, R. S. MACMILLAN, and (Assee.) G. A. ROALFE (U.S.P. 1,832,468, 17.11.31. Appl., 24.2.30).—The mill has a vertical shaft and downward flow of material. The stationary lining contracts downwardly and is provided with both horizontal and vertical ribs, the latter being rather less prominent. B. M. V.

Pulveriser mill. H. R. CARR (U.S.P. 1,833,560, 24.11.31. Appl., 10.2.30).—An outer ring of rollers runs against a tyre and an inner ring of rollers runs in the spaces between the outer rollers; all the rollers have axes which are short compared to their diam. and are placed in the space between two horizontal rotating discs, being driven from the lower one by gravitational friction only. The material is fed through apertures in the upper disc and conveyed away in an air current. Scoops are provided to lift back any coarse material which drops out of the grinding zone. B. M. V.

Aëration of slurry. W. A. NEILL and W. A. STED-MAN, ASSTS. to DORR CO., INC. (U.S.P. 1,832,469, 17.11.31. Appl., 1.6.29).—Air outlets (fixed or rotating) at the bottom of a deep tank of slurry are provided with filters of porous tile which, on cessation of the air supply, may permit liquid but not slurry to enter the pipes. B. M. V.

**Removal of adhering liquids from solid bodies.** SIEMENS & HALSKE A.-G. (B.P. 375,138, 3.9.31. Ger., 18.9.30).—Methods of blowing the moisture off articles as they emerge from baths are described. B. M. V.

Separation of intermixed divided materials. R. PEALE, Assr. to PEALE-DAVIS Co. (U.S.P. 1,832,048, 17.11.31. Appl., 2.5.28).—The lower stratum from a pneumatic shaking table is guided into a fairly compact stream and, while falling freely off the edge or end of the table, is subjected to an upwardly inclined air blast whereby the finer particles are blown to a different collector or, preferably, to a subsidiary table. The apparatus is especially suitable for material differing greatly in size but not much in sp. gr., *e.g.*, coal and dirt. B.M.V.

Centrifugal separator. J. H. WEBB (B.P. 1,832,269, 17.11.31. Appl., 18.6.30).—The basket is inverted frusto-conical and is double-walled with the coarser mesh inside; thus three products are obtained.

B. M. V.

Filter-press. P. W. PRUTZMAN (U.S.P. 1,833,335, 24.11.31, Appl., 3.3.30).—The press is constructed on the plate-and-frame principle but its axis is vertical and it is enclosed in a casing. An axial passage is left for discharge of cake (the plates being annular) and each space for cake is provided with a rotating scraper.

Filtering medium. H. H. BURHANS (U.S.P. 1,833,315, 24.11.31. Appl., 21.12.23).—A filter, preferably of the rotary-drum type, is provided with a medium of woven fabric having in addition a very pronounced nap like bristles which is smoothed down during the filtering and washing stages but lifted to a perpendicular position by internal air pressure when discharging. B. M. V. Distillation apparatus. W. SIECK, JUN., Assr. to W. GARRIGUE & Co., INC. (U.S.P. 1,831,887, 17.11.31. Appl., 2.1.30).—The apparatus is suitable, *e.g.*, for the distillation of glycerin under 29 in. vac. The still is heated by a steam coil and also by injected steam derived from returned aq. condensate and superheated in a tubular exchanger in the outgoing mixed vapour; some condensed glycerin is collected from the exchanger. The mixed vapours pass up to a reflux tower heated at the bottom (whence the remainder of the glycerin is removed) and provided with a partial condenser at the top; the remaining vapours are finally condensed, the condensate containing traces of glycerin being fed to the boiler for injected steam so that the vapours removed by the vac. ejector contain no glycerin.

B. M. V.

Condenser. J. N. VANDEGRIFT, ASST. to INTERNAT. BITUMENOIL CORP. (U.S.P. 1,833,664, 24.11.31. Appl., 10.11.27).—A metallic spiral is sandwiched between two discs and the whole submerged in cooling liquid.

B. M. V.

Concentration of liquids. H. W. THOMPSON, ASST. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,831,892, 17.11.31. Appl., 11.2.28).—The liquid is admitted in divided form to the top of a tower to the lower part of which cold air is admitted and hot gases of combustion to an intermediate point, the mixture rising at a moderate temp. performing the evaporation. The vapours from the tower may be passed downwards through a recuperator tower concurrently with liquid in the same or an earlier stage of evaporation. B. M. V.

Purification and concentration of liquids. W. G. LAIRD, ASST. to HEAT TREATING CO. (U.S.P. 1,833,717, 24.11.31. Appl., 13.1.27).—A const.-boiling mixture is further separated by rectification at reduced partial pressures produced by the presence of an inert gas and/or a vac. followed by treatment at increased pressure. B. M. V.

Device for determining the specific gravity of liquids. E. SACHS, ASST. to R. BOSCH A.-G. (U.S.P. 1,832,792, 17.11.31. Appl., 27.7.26. Ger., 26.10.25).— A hydrometer, e.g., of the syringe type, is provided with a float of prismatic form and a surrounding vessel of prismatic form having a multiple of the no. of sides and not much clearance from the float; e.g., a triangular float is guided by a hexagonal tube. The stem of the float and that part of the vessel surrounding it are circular to prevent distortion of the scale. B. M. V.

Viscosimeter. B. S. TESCHNER, ASST. to TESCHNER-MYERS Co., INC. (U.S.P. 1,831,980, 17.11.31. Appl., 29.11.27).—The viscosimeter is of the type in which the time of discharge of a certain vol. is measured. The claims refer to the inter-connexion of the outlet valve with a stop-clock and with a lever to open it, the latter being released electrically and promptly closed by a spring. B. M. V.

Apparatus for treating liquids or semi-liquids [e.g., reconstitution of cream]. H. and B. HIBBERT (B.P. 375,317, 17.3.31).—A hand-operated pump at the bottom of a vessel forces the liquid through a fine aperture formed in the bore of the pump ram. B. M. V.

B. M. V.

Means for introducing gas into a liquid. SVENSKA AKTIEB. GASACCUMULATOR (B.P. 375,164, 12.10.31. Swed., 29.11.30).—An automatic valve for causing a steady flow to become intermittent is described; this is found to be the most effective way of introducing a gas into a solvent. The valve comprises a springloaded disc upon which the pressure of the entering gas acts over only a small area when the valve is closed, but when once opened by the accumulating pressure of the steady stream the pressure acts on the whole area. The discharge is restricted to a sufficient extent to give a suitable period to the puffs. B. M. V.

Mixing vat. J. R. KUENEMANN, ASST. to L. S. LOEB (U.S.P. 1,833,220, 24.11.31. Appl., 9.4.28).—A dusttight closure is described. B. M. V.

Conversion of liquefied gases into gas at atmospheric temperature and any desired pressure. K. S. MURRAY, and BRIT. OXYGEN Co., LTD. (B.P. 375,479, 23.4.31).—The liquid is evaporated at a temp. rather above that of the final bottling and the excess pressure used to operate a pump to charge the bottles. The apparatus is started by working the pump by hand a few times. B. M. V.

Regenerative chambers of open-hearth or reheating furnaces. UNITED STEEL Cos., LTD., and A. MCKENDRICK (B.P. 377,342, 14.5. and 9.10.31).

Furnace-charging apparatus. E. E. BROSIUS (B.P. 376,764, 10.4.31. U.S., 9.5.30).

[Controlling temperature in] furnaces. K. E. LUNDBERG (B.P. 377,061, 18.11.31. Swed., 22.11.30).

Means for consuming smoke in boiler furnaces A. DRAKEFORD (B.P. 374,988, 30,12,31).

Pressure-temperature method and apparatus for control of heating. SARCO CO., INC., Assees. of T. N. ADLAM (B.P. 377,522, 4.11.31. U.S., 8.11.30).

Tilting thermostats with a light and a heavy liquid. S. J. BENNETTER (B.P. 376,786, 15.4.31).

Connecting device for supply and discharge pipes of centrifugal separators. AKTIEB, SEPARATOR (B.P. 377,560, 7.1.32, Swed., 8.1.31).

Screw pumps or packing-glands employing a viscous liquid. C. J. S. Appleyard, B. E. A. VIGERS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 375,400, 25.3.31).

Air compressors of the multi-cylinder reciprocating type. H. R. RICARDO (B.P. 377,314, 28.4.31).

Air or gas compressors. BRIT. THOMSON-HOUSTON CO., LTD., and F. W. ASHLEY (B.P. 377,202, 20.4.31. Addn. to B.P. 346,450).

Containers for liquefied gases. L'AIR LIQUIDE Soc. ANON. POUR L'ETUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 376,822, 16.4.31. Fr., 17.4.30).

Distribution of fluids [into several paths in definite ratio]. BRIT. CELANESE, LTD., and E. KIN-SELLA (B.P. 374,886, 6.2.31).

Separators.—See II. Catalytic reactions.—See VII. Kilns.—See IX. Fusion of substances. Separating dust from gases.—See XI.

# II.—FUEL; GAS; TAR; MINERAL OILS.

Autoxidation of fossil coals. I. BLUM (Bul. Chim. Soc. Române Stiin., 1929, 32, 45—49).—Humic acids are considered to be the best index of tendency to inflammability. Tests on lignites showed that humic acid and lignin rapidly absorb  $O_2$  in presence of alkali and air and that if they are removed the residue absorbs  $O_2$  only at the same rate as true coal. C. I.

Rotary low-temperature carbonisation plant of Salerni. A. THAU (Brennstoff-Chem., 1932, 13, 228—231; cf. B., 1932, 167, 487).—The plant is said to be an application of known results of the pioneer work of German investigators. The design of the plant, however, is adversely criticised and doubt is thrown on the possibility of its successful economic operation. A. B. M.

Heat of coking and total heat expenditure in coke ovens. E. TERRES and O. DOERMANN (Brennstoff-Chem., 1932, 13, 221-228).-Heat balances have been drawn up for two ovens (an end oven and a middle oven, respectively) of a vertical chamber-oven battery, determinations being made of (a) the amount of coal carbonised, (b) the coke used for firing the oven, (c) the heat of coking of the coal (cf. B., 1931, 427), (d) the sensible heat of the flue gases leaving the recuperator (cf. B., 1929, 512), (e) the heat lost by radiation and conduction (by means of a Lamort heat-loss meter), and (f) the heat expended in the production of watergas in the oven. Of the total heat supplied to the oven 33% represented the nett heat required for carbonisation, 37% was lost by conduction and radiation, 27% represented the sensible heat of the flue gases, and the remainder was made up of the sensible heat of the generator clinker, experimental errors, etc. By comparing the heat expended with the oven full with that with the oven empty a val. for the heat of coking was obtained (361 kg.-cal./kg.) which agreed well with that determined in the laboratory (353.6 kg.cal./kg.). The heat expended in the production of water-gas in the ovens (calc. from the observed total heat expenditure when the charge was steamed) amounted to approx. 800 kg.-cal. per cu. m. of watergas. The calc. coking time agreed well with the observed val.; assuming the latter val. for the coking time, the results gave a val. of 0.0032 for the mean thermal conductivity of the coal and coke during carbonisation (cf. B., 1929, 461). A. B. M.

Pintsch-Hillebrand continuous water-gas plant of the Hamburg Gasworks. STIEF (Gas- u. Wasserfach, 1932, 75, 581-586).-A study of the heat balance of the water-gas process shows that, unless pure O, is used, external heat must be supplied. In the discontinuous process this is obtained by burning coke in the generator, in the process under review, by regeneration and combustion of part of the gases produced. The Hamburg plant for combined carbonisation and gasification of brown coal briquettes is described. The chequer-brick regenerators are heated to 1300° by burning part of the gas produced and serve to heat saturated gas from the carbonisation chamber, situated above the generator, to 1280°. The water-gas formed leaves at 700-750°, is cooled in condensers, and sent

to the mains except for the part which is used for heating the regenerators. The efficiency of the process is 52% but could probably be raised to 70%, compared with 65% for the discontinuous method. R. N. B.

Treatment of Estonian oil shale. H. I. WATERMAN, S. C. DE JONG, and A. J. TULLENERS (J. Inst. Petroleum Tech., 1932, 18, 183—186).—A scheme of treatment of shale oil by catalytic hydrogenation is discussed. The crude oil is separated into gasoline, kerosene, and a residual liquid oil and each fraction treated with H<sub>2</sub> under pressure and at high temp. in presence of a colloidal Mo-C catalyst. Phenols were almost entirely removed from the two lighter fractions, in both of which the colour and odour were improved, the sp. gr. was lowered, and the NH<sub>2</sub>Ph point raised. Similar treatment of the residual oil in a Bergius autoclave gave a pale brown oil with green fluorescence, of reduced phenol content and lower sp. gr., from which gasoline and kerosene were obtained by distillation. H. S. G.

Elementary composition of neutral oils obtained from the crude tar from Rumanian lignites, and determination of the olefinic, aromatic, and saturated hydrocarbons in the fractions of these oils. N. DĂNĂLĂ and T. D. IONESCU (Bul. Chim. Soc. Române Stiin., 1929, **31**, 229—273).—The % yield and properties of the coke, tar, aq. liquor, gas, and benzine condensed from the gas are given for 3 kinds of lignite distilled at various temp., with and without steam. The products resulting from the fractionation of the tar have been examined, and the neutral oils obtained by this means and by condensation from the gas have been analysed and their physical consts. (b.p., d,  $[\alpha]$ ) measured.

## D. R. D.

Physical method for the analysis of gas mixtures, especially those consisting of hydrocarbons. W. J. D. VAN DYCK (J. Inst. Petroleum Tech., 1932, 18, 145-178) .- A special rectifying apparatus is illustrated and described in which the difference in solubility of gases in a liquid is utilised for the analysis of mixtures thereof, the gases being removed in order of their solubilities, starting with the most sol. The apparatus is especially suitable for field handling and requires only cooling H<sub>2</sub>O and electric current for its working. For hydrocarbon mixtures the results are accurate to within 0.5%, whilst the min. quantity of a component which can be identified with certainty is about 2.5%. The method of operation, and examples of the analysis of both hydrocarbon mixtures and others difficult to analyse by chemical means are given in detail. H. S. G.

[Determination of] total sulphur in benzols. G. CLAXTON, E. G. HANCOCK, and W. H. HOFFERT (Gas J., 1932, 199, 94—96).—In a modified standard lamp apparatus the burner consists of a piece of tubing containing an ignited piece of asbestos tape. The size of the flame is controlled by a SiO<sub>2</sub> sleeve raised or lowered by a Cu spiral. A closed 50-c.c. burette contains the benzol solution (20 c.c. + 80 c.c. EtOH) and is connected to the burner by a capillary tube fitted with a 3-way tap at its lowest portion. Air enters near the bottom of the burette through a fine jet and maintains the liquid level in the burner and the size of flame

const.; losses due to evaporation are a min. A better titration end-point is obtained with bromophenol-blue as indicator. Tests on solutions of various S compounds were satisfactory but low when the blank correction was added. This is a general result due to incomplete combustion. R. N. B.

Preparation of higher fatty acids by oxidation of mineral oils and paraffins with air at ordinary and increased pressure with and without catalysts. N. DĂNĂILĂ and (MISS) M. BOLTUȘ-GORUNEANU (Bul. Chim. Soc. Române Stiin., 1929, 31, 132-200).-Solid fatty acids cannot be obtained by oxidation of paraffins by air at ordinary pressure, except in the presence of Cu as catalyst, when a small yield of very impure product is obtained. With or without Cu, various mineral oils yielded, on heating with air at ordinary pressure at 135-145° for 96 hr., a mixture of OH-acids, lower fatty acids, acids analogous to humic or asphaltic acid, etc. The products are objectionable in colour and odour, and useless in the manufacture of soap. Soap (Na salts of acids similar to stearic) is obtained on heating paraffin wax or oil with aq. Na<sub>2</sub>CO<sub>3</sub> and air at high temp. and pressure (max. 170°/30 atm.) in a Bergius bomb. The starting material must be free from unsaturated compounds and paraffins containing a tert. C atom. No catalyst need be added when an Fe bomb is used, the Fe probably acting as such. 53% of the paraffin may be oxidised in one operation to Na salts of solid fatty acids. D. R. D.

Electrochemical oxidation of paraffin and mineral oils. I. A. ATANASIU (Bul. Chim. Soc. Române Stiin., 1929, 31, 75—102).—On electrolysing an emulsion of molten paraffin wax and 20%  $H_2SO_4$ , the former is oxidised at the anode to fatty acids, primarily to those of similar mol. wt., secondarily to lower fatty acids. No OH-acids are formed. Max. yields of higher fatty acids were obtained by periodic removal of the acid formed, by addition of Ce(SO<sub>4</sub>)<sub>2</sub>, V<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, or K<sub>2</sub>CrO<sub>4</sub>, by increasing the degree of emulsification, and by employing the max. possible ratio of paraffin to electrolyte. D. R. D.

Composition of the products resulting from the cracking of Rumanian mazouts. T. D. IONESCU (Bul. Chim. Soc. Române Stiin., 1930, 33, 37—43; cf. B., 1931, 707).—The olefine and aromatic contents of the above products, of the original petroleum fractions, and of the primary neutral oils obtained from Rumanian lignite are compared. Cracking increases the olefine content but has little effect on the aromatic hydrocarbon content. A. A. L.

Utilisation of aromatic hydrocarbons from petroleum fractions. II. N. POPA and A. VELCULESCU (Bul. Chim. Soc. Române Stiin., 1930, 33, 1-9; cf. B., 1926, 476).—The removal of aromatic hydrocarbons from Rumanian petroleum fractions with conc.  $H_2SO_4$ has been studied. Agitation in the liquid state is more efficient than treatment in the gaseous state. The hydrocarbons are regenerated with superheated steam and the  $H_2SO_4$  may be recovered and used again. A. A. L.

Influence of lead tetraethyl on knock rating. L. E. HEBL and T. B. RENDEL (J. Inst.!Petroleum Tech., 1932, 18, 187—196).—The improvement in H.U.C.R. is an exponential function of the concn of PbEt<sub>4</sub> added. to a gasoline, the equation of which contains a const., K, that is substantially identical over a wide range of different gasolines, the average val. being 0.75. The susceptibility of a gasoline to the action of PbEt<sub>4</sub> can be deduced from the equation S = I/0.75N, where Iis the increase in H.U.C.R. for the addition of N c.c. of PbEt<sub>4</sub> per (U.S.) gal. of benzine, if the H.U.C.R. of a gasoline and a mixture of it with one concn. of PbEt<sub>4</sub> is known. Tables are given showing the H.U.C.R. and Pb susceptibilities of gasolines made from different crudes, and of the effect of the volatility and composition of the gasolines. H.S.G.

Influence of lead tetraethyl on knock rating. F. B. THOLE and R. STANSFIELD (J. Inst. Petroleum Tech., 1932, 18, 526).—The const. vals. for K found by Hebl and Rendel (preceding abstract) may be due to the E. 35 engine used by them; with the Armstrong engine K varied over a wide range. Furthermore, in the Armstrong engine the Pb susceptibility decreases as the fuel becomes more volatile, or as natural gasoline is added, which is the reverse of that reported for the E. 35 engine. These differences are not due to engine design as they also hold for gasolines tested in overheadvalve engines. H. S. G.

Hydrogenation.—See III. Cementing oil wells. —See IX. Tar-distillate sprays. Spray oils.—See XVI.

## PATENTS.

Pneumatic separators [for coal]. G. B. SADLER (B.P. 375,046, 26.5.31).—The shaking deck is composed of spaced bars through which the lower stratum is adapted to pass, and below it is a screen conveyor composed of alternate finely-reticulated and imperforate sections upon which further separation of the fine coal and refuse is effected. B. M. V.

Intermittent vertical chamber ovens. GAS CHAM-BERS & COKE OVENS, LTD., and N. J. BOWATER (B.P. 375,300, 15.1.31).—Each setting is supplied independently with hot gas conveyed from a separate external producer by an insulated main. Air recuperators are kept to a min. size or are eliminated so that a max. vol. of steam may be raised in waste-heat boilers.

#### R. N. B.

Horizontal chamber ovens for production of gas and coke. C. OTTO & Co. G.M.B.H. (B.P. 375,608, 31.8.31. Ger., 29.8.30).—Longitudinal air passages are provided between the refractory brickwork of the regenerators and the concrete sub-structure, which is thereby thermally insulated. These air passages are open to the atm. or are connected to a compressed-air supply and lead into the main chimney flue. R. N. B.

Apparatus for manufacturing combustible gas. H. O. LOEBELL, ASST. to H. L. DOHERTY (U.S.P. 1,826,007, 6.10.31. Appl., 7.12.23).—A gas generator is provided with means for feeding in fuel, preferably a mixture of coal and coke, at the top, and withdrawing ash and unburned fuel at the bottom. A blast of air and steam, preheated in recuperators or regenerators, is blown down a conduit mounted axially in the upper part of the generator and extending approx. to the middle of the charge. The major part of the blast gases pass radially through the fuel column and thence through a circumferential outlet to a waste-heat boiler and to the regenerators. A part of the blast gases pass up through the fuel in the upper part of the generator, carbonising the coal therein, the enriched gases being withdrawn from the top of the generator. The bottom of the generator, which is rotatable, is provided with helically sloped steps which direct the ash and unburned fuel towards a central opening below which is a movable circular table, which supports the central section of the fuel column and controls the rate of removal of material from the generator. A. B. M.

**Gas purification.** KOPPERS Co., Assees. of G. M. CARVLIN (B.P. 375,237, 10.2.32. U.S., 12.2.31).—H<sub>2</sub>S is removed from fuel gases by washing with a solution of a salt of the Sn group of qual. analysis, especially 0.5-1.0% As<sub>2</sub>O<sub>3</sub>, in the presence of alkali. The Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> formed is removed as crystals by evaporation to half vol. and cooling. The remaining solution (d < 1.25) containing As is reactivated by aëration etc. and used again, the S formed being separated.

R. N. B.

Removal of naphthalene and gum-forming constituents from fuel gas. KOPPERS Co., Assees. of A. R. POWELL (B.P. 374,975, 23.3.31. U.S., 22.7.30).— Fuel gas, especially water-gas, is passed upwards through a tower down the lower half of which contaminated gas oil is flowing.  $C_{10}H_8$  and most of the indene, styrene, and coumarone are removed. Further traces present are removed in the upper half by treatment with an intermittent supply of fresh oil, < 1% of the recirculated oil (> 200 gals. per 10<sup>6</sup> cu. ft.). The contaminated oil may be used for carburetting water-gas. R. N. B.

Means for regulating combustion in furnaces. Rossman Patents, Inc. (B.P. 377,305, 28.1.31. U.S., 29.1.30).

Separating divided material.-See I.

# III.—ORGANIC INTERMEDIATES.

Suida process for acetic acid recovery. E. P. Poste (Ind. Eng. Chem., 1932, 24, 722-726).—Two American plants formerly working the  $Ca(OAc)_2$  process have been converted to the Suida method, the AcOH being separated first and the MeOH separated from the vapours leaving the scrubbing column. The dehydrated tar is distilled to give light oil, heavy oil, and scrubbing oil, the last being used for AcOH extraction. The AcOH produced is of 92% concn., not quite waterwhite, but sufficiently pure to be used directly for the manufacture of solvent esters. The original 60 plates of the Suida column have been reduced to 20 by the return to the upper portion of the column of a small proportion of distillate from the condenser. C. I.

Hydrogenation of aromatic hydrocarbons at high pressure with nickel on kieselguhr as a catalyst. G. F. SCHOOREL, A. J. TULLENERS and H. I. WATERMAN (J. Inst. Petroleum Tech., 1932, 18, 179– 182).—C<sub>6</sub>H<sub>6</sub> heated at 190° under an initial pressure > 100 kg./sq. cm. was almost entirely converted into C<sub>6</sub>H<sub>12</sub>, but raising the temp. to 460° for 1 hr. transformed the C<sub>6</sub>H<sub>12</sub> into (probably) methylcyclopentane.

Similar treatment of *p*-cymene at  $< 200^{\circ}/140$  kg./sq. cm. yielded menthane, which on continued heating at 470° for 1 hr. decomposed into CH<sub>4</sub>, C<sub>3</sub>H<sub>8</sub>, and low-boiling products free from olefines and aromatic compounds.

H. S. G.

Fatty acids for mineral oils.—See II. BuOH-COMe<sub>2</sub> fermentation.—See XVIII.

# PATENTS.

Manufacture of aliphatic anhydrides. BRIT. CELANESE, LTD., S. J. GREEN, and T. P. DEE (B.P. 371,946, 31.1.31).—An aliphatic (acetic) acid vapour (2 mols.) and COCl<sub>2</sub> are passed at 280—310° through a packed tube. C. H.

Separation of acetic anhydride and acetic acid. G. B. ELLIS. From C. F. BOEHRINGER & SÖHNE, G.M.B.H. (B.P. 372,434, 17.2.31).—A suitable acetate (Na, K, Ca) is added, preferably together with PhMe or other solvent for  $Ac_2O$ , and  $Ac_2O$  is distilled off the solid double acetate (which also retains any  $H_2O$  present). The AcOH may be recovered from the double acetate by distillation or extraction. C. H.

Manufacture of acetamide and acetic acid [from formamide or carbon monoxide and ammonia]. Soc. FRANC. DE CATALYSE GÉNÉRALISÉE (B.P. 371,555, 18.3.31. Fr., 26.3.30).—HCO·NH<sub>2</sub> vapour and H<sub>2</sub>, with or without steam, are led through a tube at 250— 300°, preferably filled with pumice, C, Cu powder, Al<sub>2</sub>O<sub>3</sub>, or Cu and Al<sub>2</sub>O<sub>3</sub>, and exposed to ultra-violet radiation; a mixture of CO and NH<sub>3</sub> may be used in place of HCO·NH<sub>2</sub>, the temp. then being 100—300°/14 atm. C. H.

Continuous rectification of acetic acid. Soc. DES ETABL. BARBET (B.P. 372,507, 31.3.31. Fr., 31.3.30).— The crude acid is fed through a column into a boiler, the vapours from which and from the bottom of the column pass into a second reflux column, from which pure AcOH is collected. The light impurities from the top of the first column and the heavy residue from the boiler are fractionated. Suitable apparatus is figured. C. H.

Concentration of dilute aliphatic acids. W. W. GROVES. From E. B. BADGER & SONS Co. (B.P. 371,554, 18.3,31).—In the extraction process a mixture of  $Pr_{2}^{9}O(e.g., 60-80\%)$  and EtOAc is used as solvent, the partition coeff. of which is high for the whole range of concns. C. H.

Manufacture of ester acetates direct from calcium acetate. E. G. CORBETT (B.P. 371,586, 13.4.31).—  $H_2SO_4$  is added in excess to a stirred mixture of  $Ca(OAc)_2$  and monohydric alcohol (EtOH) and the ester is distilled off. C. H.

Manufacture of alkamines. I. G. FARBENIND. A.-G. (B.P. 371,490, 2.2.31. Ger., 31.1.30).—Alkamines, Ar·CH(OH)·CHR·NR'R", where Ar = aryl (which may carry OH or substituted OH), R = alkyl, R' = alkyl, alkenyl, aralkyl, aralkenyl, or H, and R" = aralkyl or aralkenyl, are synthesised. Examples are the condensation of:  $\beta$ -methylamino- $\alpha$ -phenyl-*n*-propyl alcohol with cinnamyl bromide (B,HCl, m.p. 175°) or *p*-methylbenzyl bromide (B,HCl, m.p. 207°);  $\beta$ -amino- $\alpha$ -phenyl*n*-propyl alcohol with CH<sub>2</sub>PhBr (B,HCl, m.p. 192—193°; allylated, B,HCl, m.p. 188—189°) or cinnamyl brommide (m.p. 101—102°; B,HCl, m.p. 228—292°);  $\beta$ -methylamino- $\alpha$ -*p*-hydroxyphenyl-*n*-propyl alcohol with CH<sub>2</sub>PhBr (2 mols.) (B,HCl, m.p. 178—179°); 2-benzyl-amino- $\alpha$ -phenyl-*n*-propyl alcohol with CH<sub>2</sub>PhBr (B,HCl, m.p. 212—213°) or cinnamyl bromide (B,HCl, m.p. 210°).  $\beta$ -*p*-Methylbenzylanethylamino- $\alpha$ -phenyl-*n*-propyl alcohol (B,HCl, m.p. 207°) is obtained by reduction of the propiophenone. C. H.

Chlorination of organic compounds. N. V. DE BATAAFSCHE PETROLEUM MAATS., Assees. of S. C. CARNEY (B.P. 372,028, 16.3.31. U.S., 8.10.30).—Chlorination is effected continuously within a rectifying column, the temp. being adjusted by introduction. of a suitable liquid, preferably one of the condensates, into the reaction zone. The chlorination of  $C_4H_{10}$  is described. C. H.

Manufacture of derivatives of [hydr]oxy-fatty acids [wetting agents]. Soc. CHEM. IND. IN BASLE (B.P. 371,088, 16.2.31. Switz., 25.3.30).—Hydroxylated fatty acids or their derivatives are heated with a sulphophthalic acid or anhydride in absence of sulphonating agents, preferably in an org. solvent, and the products are sulphonated if necessary. Wetting agents are thus obtained from castor oil and 3-sulphophthalic anhydride, with or without  $C_2HCl_3$ , and with or without subsequent sulphonation with 100%  $H_2SO_4$  at  $0-5^\circ$ . C. H.

Manufacture of sulphonated condensation products [wetting agents]. COMP. NAT. DE MAT. COL. & MANUF. DE PROD. CHIM. DU NORD RÉUNIES ETABL. KUHLMANN (B.P. 372,159, 9.7.31, and Addn. B.P. 373,006, 21.7.31. Fr., [A] 1.8.30, [B] 24.9.30).—(A) The residues from the distillation of MeOH obtained from CO and  $H_2$  are sulphonated together with an aromatic or hydroaromatic hydrocarbon ( $C_{10}H_8$  etc.). (B) The above products are applied as free acids, or as salts of alkali or alkaline-earth metals, NH<sub>3</sub>, or amines, as wetting, dispersing, or emulsifying agents. C. H.

Manufacture of wetting, cleansing, emulsifying, and the like agents. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 371,822, 26.1.31).—" Montane derivatives" are treated with sulphonating agents. *E.g.*, montan wax is chlorinated, hydrolysed with NaOH, and sulphonated with ClSO<sub>3</sub>H in Et<sub>2</sub>O; or is amidated and the amide is converted into sulphamic acid with  $C_5H_5N$  and ClSO<sub>3</sub>H; or is hydrogenated and the resulting alcohols are treated with ClSO<sub>3</sub>H in Et<sub>2</sub>O; or is esterified with EtOH and reduced to alcohols and treated with  $H_2SO_4$  and  $Ac_2O$ ; or is esterified with CH<sub>2</sub>Ph·OH or tolyl  $\beta$ -hydroxyethyl ether and sulphonated with ClSO<sub>3</sub>H in C<sub>2</sub>HCl<sub>3</sub>. C. H.

Manufacture of wetting, cleansing, and dispersing agents. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 372,389, 28.1.31).—Amines or amides which have 1 or more aliphatic or alicyclic groups above  $C_7$ , and which may carry aryl, halogen, OH,  $CO_2H$ , or other substituents, are converted into their sulphamic acids, *e.g.*, by CISO<sub>3</sub>H and pyridine. Examples of starting materials are : palmitamide, naphthenamide, decylamine, and monopalmitylethylenediamine. Similar

products may be obtained from  $\rm NH_2 \cdot SO_3H$  and acid chlorides. C. H.

Manufacture of ester-like wetting agents, foaming agents, and dispersing agents. I. G. FARBEN-IND. A.-G. (B.P. 372,005, 25.2.31. Ger., 25.2.30. Addn. to B.P. 366,916; B., 1932, 540).-A hydroxylated or halogenated aliphatic or aromatic sulphonic acid (other than hydroxy- or halogeno-ethanesulphonic acid) is condensed with a saturated or unsaturated fatty acid above C5. Examples are products from : Na hydroxybutanesulphonate and stearyl chloride; CH2Cl·SO3Na and Na oleate; CH.O-bisulphite and olev1 chloride; Na hydroxypropanesulphonate and palmityl chloride; Na ricinoleate and Na w-chlorotoluene-p-sulphonate; Na p-phenolsulphonate, Schäffer salt, resorcinol-4:6disulphonic acid, or COMe<sub>2</sub>-bisulphite, and oleyl chloride; montan wax acid chloride or naphthenoyl chloride and Na hydroxybutanesulphonate; PhCHO-bisulphite and stearyl chloride; Na β-hydroxy-γ-o-toloxypropanesulphonate and oleyl chloride. C. H.

Manufacture of aldehydes and intermediate products [perfumes]. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 372,013, 3.3.31).-The condensation of halogenoacetic esters with cyclic aldehydes or with ketones in presence of NaNH<sub>2</sub>, NaOEt, etc. (Darzens' reaction) takes place smoothly in a suitable solvent (liquid hydrocarbons and their liquid halogen derivatives, e.g., light petroleum, cyclohexane, tetra- and decahydronaphthalenes, C6H6, PhMe, PhCl). The resulting glycidic esters are hydrolysed to salts, which give aldehydes in good yield when acidified and steamdistilled. Examples are: CH, Ph·CHO, b.p. 80-82°/ 10 mm., from PhCHO via Et phenylglycidate, b.p. 128-130°/4-5 mm.; p-chlorophenylacetaldehyde, m.p. 39-40° (hyacinth odour), from p-C6H4Cl·CHO via Et p-chlorophenylglycidate, b.p. 155-160°/4 mm.; hydratropaldehyde from COPhMe via Et β-phenyl-βmethylglycidate, b.p. 132-134°/5 mm. (strawberry odour); azi-trimethylundecaldehyde, b.p. 106-110°/3 mm. (floral odour), from hexahydro-\u00fc-ionone via the Et glycidate, b.p. 160-165°/4-5 mm.; decahydro-β-naphthaldehyde, b.p. 100-102°/4 mm. (ambergris odour), from β-ketodecahydronaphthalene via the Et glycidate, b.p. 152-157°/5 mm.; γ-2:2:6-trimethylcyclohexyl-a-methylbutaldehyde, b.p. 115-120° (odour of nuts and leaves), from tetrahydroionone via the Et glycidate, b.p. 170-175°/4-5 mm. Many other products having odour of raspberries, lilac, mimosa, musk grains, hay, anise, etc. are described. C. H.

Manufacture of styrene and its homologues. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 371,335, 14.11.30. Addn. to B.P. 340,587; B., 1931, 335).—The catalysts of the prior patent are replaced by dehydrogenating catalysts comprising > 3% of a heatstable compound of a metal the oxides of which are easily reducible; preferably steam and/or CO<sub>2</sub> are present. Examples are: PhEt and steam over CuO, Fe<sub>2</sub>O<sub>3</sub>, or Fe<sub>2</sub>(CrO<sub>4</sub>)<sub>3</sub> and Florida earth at 600°; 2- and 4-chloroethylbenzene and steam over ZnO and CuO at 600°. C. H.

Manufacture of a carbazole compound [2hydroxy-7-sulphonic acid]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 371,827, 26.1.31).— 2-Hydroxycarbazole-7-sulphonic acid is obtained by alkaline fusion of the 2:7-disulphonic acid, *e.g.*, at 260—280°, or with H<sub>2</sub>O at 270—290°. C. H.

Manufacture of isocyanates [carbinides]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 372,355, 4.2.31).—Aminoazo compounds are treated with COCl<sub>2</sub> at temp. rising to 100° in presence of an org. solvent, whereby the NH<sub>2</sub> group is replaced, via the carbamyl chloride, by ·N:CO. Examples are ·NCO compounds from : p-aminoazobenzene (m.p. 97—98°; carbamyl chloride, m.p. 110—112°); p-anisidine  $\rightarrow$  m-toluidine (m.p. 80°); o-chloroaniline  $\rightarrow$  cresidine (m.p. 153°); o-phenetidine  $\rightarrow \alpha$ -naphthylamine. C. H.

Production of leuco-1:4:5:8-tetra-aminoanthraquinone and derivatives thereof. VER. F. CHEM. U. MET. PROD. (B.P. 371,594, 16.4.31. Czech., 17.4.30).— Diamino-anthrarufin- or -chrysazin-mono- or -di-sulphonic acids are heated under pressure with  $NH_3$  or a primary or secondary amine, and sufficient  $Na_2S_2O_4$  to form and preserve the leuco-compound. C. H.

Distilling apparatus.—See I. Carbazole derivatives.—See IV. Products from sugar-cane sprouts. —See V. Wetting etc. agents.—See VI. Decomp. products from leather.—See XV. Ca gluconate. 3:5-Di-iodo-4-pyridone. N-Alkyldi-iodochelidamic acids.—See XX.

## IV.—DYESTUFFS.

## PATENTS.

Manufacture of vat dyes of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 372,123, 2.6.31. Ger., 3.6.30. Addn. to B.P. 350,575; B., 1931, 965).— In the process of the prior patent, the dichlorophenyltriazine is replaced by an alkyl-, aralkyl-, or cycloalkyldichlorotriazine. Examples are: 1-aminoanthraquinone condensed with 4:6-dichloro-2-methyl-1:3:5-triazine (greenish-yellow) or dichlorocyclohexyltriazine (yellow); 1-amino-4-benzamidoanthraquinone with dichloro-Nbutyl-1:3:5-triazine (blue-red); 1-amino-4-methoxyanthraquinone with dichlorobenzyltriazine (orange).

C. H.

Production of vat dyes [from 3-fluoro-2-aminoanthraquinone]. L. J. HOOLEY, R. F. THOMSON, D. A. W. FAIRWEATHER, and SCOTTISH DYES, LTD. (B.P. 371,420, 18.10.30).—3-Fluoro-2-acetamidoanthraquinone, obtainable by acetylation, is treated with pyridine-SO<sub>3</sub> and Cu in pyridine to give, after removal of the Ac group with NaOH, 3-fluoro-2-aminoanthrahydroquinone disulphuric ester. This ester is converted by alkaline oxidation, e.g., with NaOCl, into the tetrasulphuric ester of 3 : 3-difluorotetrahydrodianthraquinoneazine, yielding 3 : 3-difluoroindanthrone by acid oxidation (e.g., on the fibre). The tetra-ester on acidification gives the di-ester of 3 : 3-difluoroanthrahydroquinonedihydroazine, which similarly yields indanthrone by acid oxidation. C. H.

Manufacture of thioindigoid dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 371,459, 24.1.31).— A polynuclear aryl Me ketone having a free *o*-position is treated with S<sub>2</sub>Cl<sub>2</sub>, preferably in an org. solvent. Examples are:  $\alpha$ -C<sub>10</sub>H<sub>7</sub>·COMe and S<sub>2</sub>Cl<sub>2</sub> in PhNO<sub>2</sub> at 140—150° for  $\beta$ -naphthathioindigo; and thioindigos from  $\beta$ -C<sub>10</sub>H<sub>7</sub>-COMe, acetylphenanthrenes, m.p. 70–71° and 145°, respectively, and 1-methoxy-4-naphthyl Me ketone. C. H.

Manufacture of [thio]indigoid vat dyes. Soc. CHEM. IND. IN BASLE (B.P. 372,640, 10.8.31. Switz., 8.8.30).-6-Chloro-5-methylthioindoxyl, m.p. 159-160°, made from 2-chlorotoluene-4-sulphonyl chloride via the 4-thiol, b.p. 117-118°/14-15 mm., and the 4-thioglycollic acid, m.p. 86°, gives with p-NO C6H4 ·NMe2 the p-dimethylaminoanil, m.p. 220°, of the corresponding thioisatin, m.p. 147°. 6-Bromo-5-methylthioindoxyl, m.p. 158-159°, from 2-bromo-p-tolylthioglycollic acid, m.p. 81°, gives a p-dimethylaminoanil, m.p. 247-248°. The chloromethylthioindoxyl is condensed with acenaphthaquinone (scarlet; yellower on bromination), isatin  $\alpha$ -anil (heliotrope), 5:7-dibromoisatin (red-brown), 5:7dibromoisatin  $\alpha$ -chloride (violet), and  $\alpha$ -p-dimethylaminoanils of thioisatin (red), 5-chloro-7-methylthioisatin (blue-red), 6-ethoxythioisatin (blue-red), a-naphthathioisatin (blue-bordeaux), and \$\$-naphthathioisatin (violet). 6-Chloro-5-methylthioisatin  $\alpha$ -p-dimethylaminoanil is condensed with 6-chloro-4-methylthioindoxyl (bluepink) and  $\beta$ -naphthathioisatin (yellow-bordeaux).

C. H.

Manufacture of azo dyes. I. G. FARBENIND. A.-G. (B.P. 372,562, 14.5.31. Ger., 14.5.30).—The disulphuric esters of anthrahydroquinone-2-sulphamic acids, obtainable by action of ClSO<sub>3</sub>H on the amine ester, are diazotised and coupled with components free from SO<sub>3</sub>H or CO<sub>2</sub>H groups, especially 2:3-hydroxynaphthoic arylamides. The products may be developed on the fibre with acid oxidants. Examples are the coupling of disulphuric esters of anthrahydroquinone-2-sulphamic acid or its 3-Br-derivative with 2:3-hydroxynaphthoic *o*-anisidide (violet, becoming red on development),  $\beta$ -naphthol (brown-red, becoming orange), and 1-phenyl-3-methylpyrazolone (red, becoming red-yellow). C. H.

[Manufacture of] azo dyes for leather. IMPERIAL CHEM. INDUSTRIES, LTD., M. MENDOZA, and J. HANNON (B.P. 371,866, 28.1.31).—Chocolate or nigger shades on leather are obtained by the application of dyes of the type: NH<sub>2</sub>Ph (or homologue or Cl-, NO<sub>2</sub>-, or SO<sub>3</sub>H derivative)  $\Rightarrow \alpha$ -C<sub>10</sub>H<sub>7</sub>·NH<sub>2</sub> or an aminonaphthol or a sulphonic acid thereof  $\Rightarrow$  m-C<sub>6</sub>H<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub> (or 4-alkyl-, -alkoxy-, or -halogeno-derivative)  $\leftarrow$  NH<sub>2</sub>Ph (etc.), the finished dye containing at least 2 SO<sub>3</sub>H groups. Examples are : NH<sub>2</sub>Ph  $\Rightarrow$  Cleve acid  $\leftarrow$  m-C<sub>6</sub>H<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub>  $\leftarrow$  p-nitroaniline-o-sulphonic or sulphanilic acid ; metanilic acid  $\Rightarrow$  $\alpha$ -C<sub>10</sub>H<sub>7</sub>·NH<sub>2</sub>  $\Rightarrow$  m-C<sub>6</sub>H<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub>  $\leftarrow$  aniline-2: 5-disulphonic acid; NH<sub>2</sub>Ph  $\Rightarrow$  J-acid  $\Rightarrow$  m-C<sub>6</sub>H<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub>  $\leftarrow$ sulphanilic acid. C. H.

Manufacture of chromiferous azo dyes. Soc. CHEM. IND. IN BASLE (B.P. 371,449, 23.1.31. Switz., 23.1.30).—Dyes of the type 5-nitro-o-aminophenol  $\rightarrow$ O-arylsulphonyl-J-acid are prechromed, and give level green shades on viscose silk. C. H.

Manufacture of azo dyes [pigments and ice colours]. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 372,338, 30.12.30).—Arylamides of salicylic acid, carrying in 4- and 5-positions 2 Me groups or Me and Cl, are coupled with a diazo, diazoazo, or tetrazo compound of the C<sub>6</sub>H<sub>6</sub> or Ph<sub>2</sub> series (which may have alkyl, alkoxyl,

halogen, or NO<sub>2</sub> substituents), or of the C<sub>10</sub>H<sub>8</sub> series, or with a diazoanthraquinone, especially with a halogenated diazo compound of the C6H6 series. Brown shades are Examples include :  $2:5-C_6H_3Cl_2\cdot NH_2 \rightarrow$ obtained. 4:5-dimethylsalicylic  $\alpha$ - or  $\beta$ -naphthylamide, m-chloroanilide, 4-chloro-o-anisidide, 4-nitro-o-anisidide, 4-methoxy-a-naphthylamide, 5-nitro-a-naphthylamide, 1:5naphthylenediamide, p-phenylenediamide, or dianisidide;  $2:5-C_6H_3Cl_2\cdot NH_2 \rightarrow 5$ -chloro-4-methylsalicylic anilide, 5-chloro-o-toluidide, p-chloroanilide, 5-chloro-o-anisidide, α-naphthylamide, or p-xylylene-2:5-diamide; 2:5- $C_6H_3Cl_2 \cdot NH_2 \rightarrow 4$ -chloro-5-methylsalicyclic 5-chloro-otoluidide, 2:5-dimethoxyanilide, 2-carbazolylamide, pphenylenediamide, etc. C. H.

Manufacture of water-insoluble azo dyes and intermediate products therefor [pigments and ice colours from N-substituted 3-hydroxycarbazole-2carboxylic arylamides]. A. CARPMAEL. From I. G. FARBENIND, A.-G. (B.P. 372,301, 3.2., 21.9., and 27.10.31). -N-Substituted 3-hydroxycarbazoles, obtained from 3-alkoxycarbazoles, are converted by Kolbe reaction into their 2-carboxylic acids, the arylamides of which are coupled in substance or on the fibre with suitable diazo compounds. Examples are : 3-ethoxy-5-methylcarbazole, m.p. 85°, de-ethylated (m.p. 167-168°), carboxylated (m.p. 239°), and converted into o-anisidide, m.p. 188-190° [coupled with diazotised m-C6H4Cl·NH2 (deep yellow-brown), 4-chloro-o-toluidine (red-brown), or 4'-nitro-4-amino-2: 5-dimethoxyazobenzene (dark blue)]; 3-hydroxy-5-ethylcarbazole, m.p. 109-110°, carboxylated (m.p. 229°), and converted into o-toluidide, m.p. 187° [coupled with diazotised p-NH2.C6H4.NO2 (redbrown), 4-benzamido-2:5-diethoxyaniline (violet)]. Other compounds described are: 3-hydroxy-5-methylcarbazole-2-carboxylic p-chloroanilide, m.p. 276°, anilide, m.p. 254°, o-toluidide, m.p. 193°; 3-hydroxy-5-phenylcarbazole-2-carboxylic o-toluidide, m.p. 135°. C. H.

Hair dyes.-See VI.

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

Oxidation by gaseous oxygen of cotton impregnated with sodium hydroxide solution. G. F. DAVIDson (J. Text. Inst., 1932, 23, 95-133 T).-In all cases, the rate of absorption of O, increases for some time and then becomes practically const.; this is explained by the hypothesis that the oxidation consists of several consecutive reactions. Since the rate is not const. for any concn. of NaOH it cannot be expressed by a single no., but an idea of the relative rates of oxidation at 20°, 40°, and 60° is given by the fact that when  $10 \cdot 3N$ -NaOH is used under the conditions standardised the vol. of  $O_2$ absorbed in 48 hr. at 20° is absorbed in about 6 hr. at 40° and in about 1.2 hr. at 60°. The rate at both 40° and 60° increases rapidly as the concn. of NaOH is increased from 2.5 to 5N, less rapidly up to 10N, where a welldefined max. rate is observed, and then falls. The presence of Fe in the soda-cellulose has a pronounced catalytic effect on the rate of oxidation, but the effect decreases with increasing concn. of NaOH and is small for concns. above 10N. Fe dissolved in the NaOH is conc. on cotton by adsorption during the steeping process, and a method of purifying aq. NaOH from Fe based

on this effect is described. Cu and Ni have an accelerat ing action < that of Fe, but Mn greatly retards the oxidation of cotton impregnated with 5N-NaOH. The oxidation leads to increase of fluidity in cuprammonium and Cu no., the increase of both being rapid at first and then slowing off as the absorption of O<sub>2</sub> increases. The products of the oxidation are similar to those obtained by the action of NaOBr on cotton, and the oxidation provides a controllable method of preparing chemically modified cottons of relatively low Cu no.

B. P. R.

Detection and estimation of chemical damage in wool. P. KRAIS (J. Text. Inst., 1932, 23, 144— 146 P).—Estimation of damage by means of colour reactions, the action of aq. Cl<sub>2</sub>, determination of sol. N, or other chemical reactions should be accompanied by tests for mechanical strength and elasticity, since otherwise false impressions of the extent of damage may be given. In some cases damage by alkali may be indicated by the sol. N val., but may have no effect on the mechanical properties unless a subsequent acid treatment ( $H_2SO_4$  etc.) is given. Examples showing the relation between the results of chemical and mechanical tests are given. Coarse wool is less affected by chemical treatments than is finer wool. B. P. R.

Chemical tests in the wool industry. E. HILL (J. Text. Inst., 1932, 23, 124—139 P).—The various chemical tests relative to wool and its manufacture previously given (lit.) are collected and practical details are given. B. P. R.

Testing of linen fabrics from the point of view of their uses. J. A. MATTHEW (J. Text. Inst., 1932, 23, 113–123 P). B. P. R.

Testing of textiles, with special reference to investigation of defects in yarns and fabrics. W. E. KING (J. Text. Inst., 1932, 23, 102-112 P).

B. P. R.

Commercial strength standards for linen yarns and fabrics. W. J. COWDEN (J. Text. Inst., 1932, 23, 139-144 P). B. P. R.

Theory of sulphite cooking. E. Hägglund (Svensk Kem. Tidskr., 1932, 44, 163—169).—An aq. solution of sucrose containing solid ligninsulphonic acid is inverted much more rapidly than corresponds with its  $p_{\rm H}$ ; this is ascribed to the hydrolytic activity of the solid acid. H<sub>2</sub>O dissolves ligninsulphonic acid from cellulose containing it at a rate which approx. obeys a unimol. law, and the temp. coeff. between 80° and 90° is 1.9. Moreover, carbohydrate is found in the solution, and it is concluded that the process is not simple dissolution, but is a hydrolysis catalysed by the solid acid. A.G.

Acetylation of cellulose and acetylation plant. F. Ohl (Chem. Fabr., 1932, 5, 233-236).—The principles of efficient acetylation are discussed and available plant is reviewed with brief details of construction and method of operation. The requirements of the plant are stringent. The metal used in its construction must not be costly, must have good heat conductivity, and must resist the action of both the acetylation and cooling mixtures commonly used. The castings must be free from pores which serve as corrosion nuclei. A -special bronze has so far proved most generally successful, but is heavy and costly. The present tendency is towards the use of larger acetylators capable of taking charges of 1000 kg., in the construction of which acidresisting synthetic materials are largely used. Efficient mixing and temp. control are essential at all stages. V. E. Y.

Effect of calcium and phosphorus on adhesive strength of paper-coating casein. S. P. GOULD and E. O. WHITTIER (Ind. Eng. Chem., 1932, 24, 791—792). —The strength of Hammarsten casein was determined by the Sutermeister "wax test" before and after addition of various amounts of Ca (as hydroxide, borate, acetate, etc.) and P (as phosphate). It is shown that the weakening effect of ash in commercial casein is chemical rather than mechanical, being due to P and to a small extent to Ca, whilst the KCl commonly present is without effect. Thorough washing is essential for the production of casein of greatest strength. S. S. W.

Parchment paper.—See XIX.

#### PATENTS.

Manufacture of articles [hollow bodies] from cellulosic compositions. BRIT. CELANESE, LTD. (B.P. 373,037, 2.10.31. U.S., 2.10.30).—Sheets composed of an org. derivative of cellulose are wound on a mandrel and made to unite by means of a softening or swelling agent applied by spreading or immersion; subsequently the tubes etc. may be subjected to heat and pressure.

F. R. E.

Method of coating [sheets or films]. W. H. CHARCH and N. A. CRAIGUE, Assrs. to DU PONT CELLO-PHANE Co., INC. (U.S.P. 1,826,698, 6.10.31. Appl., 18.6.30. Ger., 7.10.29).—A moisture-proof transparent material with a uniform coated surface is made by subjecting a sheet of regenerated cellulose, having beads on its longitudinal edges, to a humidifying atm. and applying a moisture-proofing composition consisting of a cellulose derivative, a wax, and a volatile solvent, the excess of which is removed first from the zones adjacent to the beads and then from the entire width of the sheet maintained under uniform tension. The solvent is then evaporated at a temp. approx. that of the m.p. of the wax, and the coated material deodorised and humidified. F. R. E.

Dry-spinning device for artificial fibres. "CHATILLON" SOC. ANON. ITAL. PER LA SETA ARTIFICIALE (B.P. 372,503, 30.3.31. Italy, 7.5.30).—The evaporative medium is circulated in a closed path through the cell and a cooling chamber by convection alone and made to impinge on the filaments issuing from the nozzle from one side only of the cell. F. R. E.

Purification of cellulose fibre. G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,829,110, 27.10.31. Appl., 28.5.30).—Cuprammonium solution, *e.g.*, containing  $2\cdot 8 - 5\cdot 6$  g. Cu and 196 g. NH<sub>3</sub> per litre, is used for dissolving out degraded celluloses. Thus by treating bleached sulphite pulp containing 85% of  $\alpha$ -cellulose with the solution for 30 min. at 20° a 90.8% yield of purified pulp containing 92.5% of  $\alpha$ -cellulose is obtained. D. J. N.

Manufacture of artificial staple fibre. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 372,323, 4.2.31). -Filaments of an org. derivative of cellulose are wound on a thin rod while rendered plastic by moistening with a solvent, by addition of vinyl acetate, or by heating the rod so as to acquire a curved set, and are cut from the rod continuously with winding. F. R. E.

Manufacture of [hollow or voluminous] artificial filaments, threads, and the like. H. DREYFUS (B.P. 372,835, 19.2.31).—A solution of an org. derivative of cellulose, which may contain the coagulant, is dry-spun and a coagulant (hydrocarbons, petroleum of b.p. 60— 120°) less volatile than the solvent in the spinning solution is applied to the filaments etc. immediately after their extrusion or to the spinning nozzle itself; the solvent is subsequently rapidly evaporated at a temp. above the b.p. of the solvent, but below that of the coagulant. F. R. E.

Production of cellulose and hydrocarbons from sugar-cane sprouts and refuse and burnt and frozen cane. E. J. FRANCK (U.S.P. 1,832,375, 17.11.31. Appl., 8.1.29. Argent., 6.7.28).—The raw material is extracted with cold aq. Na<sub>2</sub>S or CaS and Na<sub>2</sub>CO<sub>3</sub> or NaOH, and the fermentation products are recovered by distillation of the liquor. The treated mass is heated at 100° in an autoclave for 1—2 hr. with a liquor containing saltpetre, Na<sub>2</sub>CO<sub>3</sub>, and NaCl, after which a chloride of S is added and distillation products are collected; finally the heating is continued under pressure, and the cooked product is bleached with aq. Cl<sub>2</sub>. The cooking liquor is evaporated to dryness and the residue is drydistilled, yielding COMe<sub>2</sub>, MeOH, C<sub>5</sub>H<sub>5</sub>N, etc. B. P. R.

Manufacture of cellulose [alkyl] compounds. L. LILIENFELD (B.P. 374,964, 14.3.30).—Cellulose is treated with one or more alkylating agents (alkyl sulphates), without heating or while cooling, in presence of an amount of alkali smaller than the wt. of  $H_2O$ ; the resulting cellulose alkyl ether, which is sol. in alkali but insol. in  $H_2O$ , is dissolved in alkali, brought to the desired form, and coagulated in a bath containing 35—80% of  $H_2SO_4$ . F. R. E.

Manufacture of mixed esters or ether-esters of polymeric carbohydrates [cellulose]. I. G. FARB-ENIND. A.-G. (B.P. 372,122, 2.6.31. Ger., 4.6.30).— Cellulose and its derivatives containing free OH groups are esterified with org. acid halide in absence of acidbinders under non-hydrolysing conditions, viz., exclusion of H<sub>2</sub>O and removal of the H halide formed either by vac. or a current of gas or a solvent. Thus, cellulose hydroxypropyl ether is distilled with PrCOCl in vac. at  $60-65^{\circ}$ ; or cellulose diacetate is heated at  $50-70^{\circ}/15-30$  mm. with coconut oil acid chlorides; or cellulose  $\beta$ -hydroxyethyl ether is kept several days with AcCl in light petroleum at 15°. C. H.

**Preparing [wood] fibres.** J. A. WIENER and J. B. HARMON (U.S.P. 1,831,940, 17.11.31. Appl., 12.5.30).— Waste wood is steeped in hot  $H_2O$  to soften the fibres, shredded, the fibres are disintegrated by means of air currents, mixed in a stock tank with pulp-mill refuse, and the whole is refined and pressed into sheets.

B. P. R.

Freeness tester [for paper pulp]. F. M. WILLIAMS (U.S.P. 1,828,210, 20.10.31. Appl., 11.5.27).—The apparatus consists of an upper part which holds the test sample of pulp, a screen to retain the fibres, and a valve to prevent discharge of liquid through the screen until the test is to be made. Two openings are provided such that the  $H_2O$  is discharged preferentially through one, and only through the other when the flow exceeds a predetermined val. B. P. R.

Treating fibrous material. L. A. GOODMAN (U.S.P. 1,829,702, 27.10.31. Appl., 7.4.30).—A fibrous base made from mitsumata, kodzu, gampi, or similar fibre is impregnated with a solution of a cellulose derivative, *e.g.*, the acetate, dried, and treated with a solution of NaOH, *e.g.*, in EtOH, to form a film of regenerated cellulose. The product has the desirable properties of natural gut and is suitable for food purposes, *e.g.*, sausage skins. D. J. N.

**Preconditioning of [pulp] cooking solutions.** T. L. DUNBAR (U.S.P. 1,832,367, 17.11.31. Appl., 17.5.30).—During the cooking of pulp part of the liquor is released from the digester into a pressure chamber where the liquid is separated from the gaseous part and fresh cold cooking liquor is passed through a coil contained in the liquid part, thus effecting indirect exchange of heat. The hot gaseous fraction from the pressure chamber is then introduced into the fresh cooking liquor after the latter has passed through the hot liquid fraction. B. P. R.

**Operating on [paper] pulp streams.** W. THALER, Assr. to AMER. VOITH CONTACT Co., INC. (U.S.P. 1,832,882, 24.11.31. Appl., 9.1.30. Ger., 14.1.29).— In a beater the pulp is thrown upwards in an inclined direction and strikes a hood having two curvatures one of which deflects the upper part of the pulp stream downwards through the lower part of the same stream.

B. M. V.

Elimination of foam in paper manufacture. H. R. RAFTON, ASST. to RAFFOLD PROCESS CORP. (U.S.P. 1,831,928, 17.11.31. Appl., 29.6.27).—Paper stock containing  $CaCO_3$  and  $Mg(OH)_2$  is treated with gelatinised starch and alum. B. P. R.

Manufacture of paper and pasteboard. E. CASA-NOVAS (B.P. 374,892, 17.3.31. Spain, 24.3.30).—A web of pulp produced in a cylinder paper machine is superimposed on another produced in a Fourdrinier machine, and the composite web is then subjected to suction and pressure. F. R. E.

Cellulosic material. A. W. SCHORGER, Assr. to C. F. BURGESS LABORATORIES, INC. (U.S.P. 1,829,763, 3.11.31. Appl., 21.2.30).—Paper, moulded articles, etc. made from partly hydrated stock or from a pulp mixture containing mechanically gelatinised cellulose are dried by heating them in an org. medium which is liquid at room temp. and is capable of displacing interstitial  $H_2O$  and  $H_2O$  of hydration. Suitable media are COMe<sub>2</sub>,  $C_6H_{6^3}$  naphtha, and particularly EtOH. Excessive shrinkage during drying is thus avoided and the

dried articles show improved softness, opacity, and resistance to  $H_2O$ . D. J. N.

Stencil sheet. S. HORH (U.S.P. 1,825,798, 6.10.31. Appl., 6.9.30).—A base of fibrous material (Yoshino paper) is coated with a solution in a volatile solvent (COMe<sub>2</sub>, AcOEt) of polysaccharide esters (starch acetate, cellulose nitrate), blown oils (camelia, tsubaki, sasanqua, or tea seed), and tempering agents (oils, waxes, Et phthalate, tolyl phosphate, etc.). F. R. E.

Stencil sheet. S. HORH (U.S.P. 1,829,750, 3.11.31. Appl., 1.8.30. Jap., 28,6.30).—A metal (Ca) salt of naphthenic acid is added as a stabilising agent to a coating composition comprising a polysaccharide ester, an alkyl naphthenate, and a tempering agent. D. J. N.

Manufacture of mineral fibre paper. A. L. KENNEDY, Assr. to PLASTIC, INC. (U.S.P. 1,830,607, 3.11.31. Appl., 22.6.27).—The kelp for treatment as in U.S.P. 1,820,538 (B., 1932, 502) is purified before use by grinding it with aq. Na<sub>2</sub>CO<sub>3</sub>, adding sufficient 10%aq. H<sub>2</sub>SO<sub>4</sub> to neutralise the alkali, and washing. By thus removing S compounds and mineral salts from the kelp the finished product shows improved dielectric properties. D. J. N.

Composite sheet material. G. A. RICHTER, Assr, to BROWN CO. (U.S.P. 1,829,157, 27.10.31. Appl.. 29.9.28).—The breaking strength of loosely felted sheets of unbeaten pulp is increased without materially reducing their absorptivity for impregnating agents by adhesively uniting them to a strong paper made from well beaten stock. D. J. N.

Paper-coating composition. D. B. BRADNER, Assr. to CHAMPION COATED PAPER CO. (U.S.P. 1,826,726, 13,10.31. Appl., 30.8.28).—Small amounts of pine oil and sulphonated castor oil are added to coating pastes consisting of mineral pigment and casein to prevent pin-hole and "grease" defects. B. P. R.

Manufacture of compound papers and pasteboards impregnated with caoutchouc. V. ANTOINE (B.P. 375,157, 30.9.31. Belg., 1.10.30).—A solution of caoutchouc is introduced between two or more thin lengths of paper or pasteboard which are then passed between pressure rollers; caoutchouc solution is afterwards circulated through the composite material by pressure and/or suction. F. R. E.

Process and apparatus for the de-pulping of fibre-containing parts of agaves and similar plants. F. KRUPP GRUSONWERK A.-G. (B.P. 377,545, 25.11.31. Ger., 27.11.30).

Shuttles.-See IX. Coating cords etc.-See XIII.

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

Faults in piece-dyed woollen and worsted materials. L. L. LLOYD (J. Text. Inst., 1932, 23, 97-101 P). --Local concn. of alkali or acid in the fabrics, nonuniform drying before carbonising, suspended metallic impurities in boiler liquors, etc. cause unlevel dyeing in the materials. B. P. R.

Faults in mercerised cotton hose. J. LOMAX (J. Soc. Dyers and Col., 1932, 48, 185–190).—A description is given of the various faults, their causes, and differentiation. A. J. H.

Use of soap in dry-cleaning. R. B. BROWN (J. Soc. Dyers and Col., 1932, 48, 190-195).-The efficiency of benzine-sol. soaps may be determined on a small scale by shaking 2.5 g. of soiled fabric with  $\geq 200$  c.c. of dry-cleaning solvent ("white spirit" + soap) in a simple machine (described). The fabrics are soiled by shaking with a mixture of fine carbon, olive oil, and white spirit. In dry-cleaning, the white spirit itself is able to effect 20-30% of the cleaning; the presence of excess of soap may decrease the cleaning considerably. In a single treatment with white spirit and soap (K oleate containing 10% of free acid) the max. effect is produced in 20-30 min.; the cleaning effect increases from 10° to 30° and then diminishes rapidly. Although the cleaning efficiency is decreased by adding H<sub>o</sub>O to the white spirit, the natural moisture content of wool favours its cleaning; the presence in white spirit of > 1:200 of methylated spirit or > 1:400 of  $H_2O$  decreases its cleaning action. A K soap containing 5-10% of free acid is the most satisfactory for dry-cleaning; NH4 soaps and triethanolamine soaps are inferior. The addition of pine oil to the dry-cleaning spirit is beneficial, but cyclohexanol is unnecessary. In short dry-cleaning trials solvent naphtha, trichloroethylene, CCl4, benzine, "Mex Rubber Solvent," and white spirit had decreasing efficiency in the order named, but when the trial period was extended from  $\frac{1}{2}$  to  $\frac{1}{2}$  hr. white spirit occupied third place. Fabrics coloured with vat dyes are especially liable to bleed in dry-cleaning liquors containing C<sub>6</sub>H<sub>6</sub> or solvent naphtha. A. J. H.

Oxidation of cotton.-See V.

#### PATENTS.

Treatment of fibrous materials [wetting agents]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 372,325, 27.11.30).-Non-acylated amines or the corresponding quaternary bases, or salts of these, having at least 1 aliphatic or alicyclic group above C7, preferably C10-C20, and at least 1 alcoholic OH, are used in aq. solution or dispersion as softening, dispersing, and wetting agents. Examples are : N-B-hvdroxyethyl-N-octadecylamine acetate : NN-dimethyl-N-hydroxyoctadecylamine sulphate (from octadecanediol monosulphate and NHMe<sub>o</sub>); N-β-hydroxyethyl-N-dodecyl, -octadecyl-, or -pentadecyl-amine ; product from phosphates of coconut oil alcohols and N-cyclohexyl-Nhydroxypropylamine or di-β-hydroxyethylamine; product from C12 alcohols, b.p. 110-170°/12 mm. (from naphthenic acids), and β-hydroxyethylamine. C. H.

Dyeing [with blue vat dyes]. R. G. KNOWLAND, Assr. to NASHUA MANUFG. Co. (U.S.P. 1,832,907, 24.11.31. Appl., 30.1.28).—In after-soaping cotton dyed with indanthrene-blue dyes, reddening of shade is prevented by adding to the soap bath 0.2-0.5% of an oxidising substance, e.g, CaOCl<sub>2</sub>, H<sub>2</sub>O<sub>2</sub>. A. J. H.

Production of fast colour reserves by means of ester salts of leuco-vat dyes without steaming. DURAND & HUGUENIN A.-G. (B.P. [A] 372,190, 13.8.31. Ger., 13.8.30, and [B] Addn. B.P. 372,224, 9.10.31. Ger., 10.10.30).—(A) The goods, impregnated with a 2:3-hydroxynaphthoic arylamide, are printed with a leuco-ester and an acid-reacting salt, e.g.,  $ZnSO_4$  or  $Al_2(SO_4)_3$ . The ground colour is first developed with a

diazotised *p*-aminodiphenylamine, and then the coloured reserve by an acid oxidant without steaming. Examples are : anilide, Soledon-jade-green, and  $\text{ZnSO}_4$ , developed with diazotised 4-amino-4'-methoxydiphenylamine and with  $\text{CuSO}_4$  and dil.  $\text{H}_2\text{SO}_4$  for green on dark blue; anilide, leuco-ester of 1:2:6:7-dibenzpyrene-3:8quinone, and  $\text{ZnSO}_4$  similarly for yellow on dark blue. (B) Al(ClO<sub>3</sub>)<sub>3</sub> is used as the acid-reacting salt; the ester salt reserve can be developed by a drying operation before development of the ground colour. C. H.

Compositions for dyeing hair. E. BASCH (B.P. 371,677, 24.6.31).—The usual ingredients, e.g., m- and  $p-C_6H_4(NH_2)_2$ , are mixed with a hydroxyalkylamine soap, e.g., stearates of tri- $\beta$ -hydroxyethylamine and glycol Et ether, preferably with enough thickener to give a paste. C. H.

Hair-rinse preparation. W. H. H. DAVIS (B.P. 375,218, 28.12.31).—An aniline dye (<1 pt.) is mixed with malic acid (20 pts.). H. R.-D.

Printing of cotton. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 371,826, 26.1.31. Addn. to B.P. 343,597; B., 1931, 628).—In the printing pastes of the prior patent, alkali salts of naphthoic or hydroxy-naphthoic acids are substituted for alkali benzoates or hydroxybenzoates. Examples are vat dye printing pastes containing: Na 2:3-hydroxynaphthoate and 6:6'-diethoxythioindigo, 6:6'-dichloro-4:4'-dimethylthioindigo, or Caledon-jade-green; Na 1-hydroxy-2-naphthoate and Caledon-jade-green or *iso*dibenzanthrone. C. H.

Printing of cotton. I. G. FARBENTND. A.-G., and A. CARPMAEL (B.P. 372,828, 12.2.31. Addn. to B.P. 343,102; B., 1931, 584).—The vat dyes of the prior process are replaced by sulphide dyes. Examples are printing pastes containing Hydron-blue or Indocarbon CL reduced with  $Na_2S_2O_4$  in presence of  $H_2O$  and glycerol or thiodiglycol and  $NH_3$ , with the usual thickeners etc. C. H.

[Bituminous] coated fabric. A. N. PARRETT, Assr. to E. I. DU PONT DE NEMOURS & Co (U.S.P. 1,833,471, 24.11.31. Appl., 9.9.27).—Artificial leather fabric is manufactured by calendering a flexible fabric, embossing with a grain after applying on one side a composition containing a bituminous material, an indurator (vulcanised oil, rubber), a softening agent (petroleum), and a filler, then heating at about 123°, and finally coating with a varnish. A. J. H.

Waterproofing of sheets of cellulosic material. COURTAULDS, LTD., and W. H. GLOVER (B.P. 375,451, 1.4.31).—Paper, film, or fabric made from cellulose or cellulose derivative fibres is impregnated with an org. solution, free from wax (cf. B.P. 309,017; B., 1930, 11), containing a cellulose ester or ether, petroleum jelly, a gum or resin, and preferably a plasticiser. A suitable solution consists of BuOAc (374), nitrocellulose (52), methylated spirit (26), ester gum (34), Bu phthalate (10), and petroleum jelly (4 pts.). A. J. H.

**Transfer.** H. S. SADTLER (U.S.P. 1,832,660, 17.11.31. Appl., 28.5.27).—Paper transfer sheets are printed in the form of a design with a resist, composed of a mixture of wax, NaHSO<sub>3</sub>, Zn dust, and anthraquinone, which is transferred to fabrics by hot-ironing, so that when the fabric is subsequently dyed a white design on a coloured ground is obtained. Alternatively, vat dyes are mixed with the resist so that coloured effects may be obtained. B. P. R.

Preparing lichens to be used for floral decorations. A. C. DUNBAR (B.P. 375,498, 15.5.31).—Lichens are treated with a wetting agent, dyed with a coal-tar dye, partly dried, and sprayed with or dipped in pure or aq. glycerin. A. J. H.

Apparatus wherein articles or materials are treated with dyes or other liquids. MELLOR, BROMLEY & Co., LTD., and T. C. BROMLEY (B.P. 377,428, 24.7.31).

Wetting agents.—See III. Coloured lacquers. Coloured masses.—See XIII.

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

Contact sulphuric acid plant [at Trail, B.C.]. W. M. COBLEIGH (Ind. Eng. Chem., 1932, 24, 717-721).-The plant described, handling Zn smelter gases, consists of a Grillo unit with Pt catalyst and 3 larger units with V catalyst, the total daily capacity being 375 tons of 100% acid. Zn flotation concentrates are "flash roasted" after calcination with controlled secondary air admission, no fuel being required. The excessive dust is collected by a cyclone. The gas then passes through a plate-and-wire Cottrell plant to dil. acid scrubbers, and is then distributed to the 4 units. In these it is washed with H<sub>2</sub>O, freed from mist in a Cottrell plant, and dried in 2 stages with 80% and 93% H2SO4. It then passes through coke filters to the preheaters and converters, which are in 2 stages. At this point the gas contains 5.5% SO<sub>2</sub>, the original 9.0% having been reduced by inward leakage. The SO<sub>3</sub> absorption towers are of steel and are fed with 98% H<sub>2</sub>SO<sub>4</sub>. The final product is diluted to 93% and is used for manufacture of  $(NH_4)_2SO_4$ ,  $(NH_4)H_2PO_4$ , and "triple super-phosphate." The  $NH_3$  is produced by the Fauser process using electrolytic H<sub>2</sub>. C. I.

Causticisation of sodium carbonate. PICKER (Chem.-Ztg., 1932, 56, 610-611).-A modern continuous causticising plant includes a rotary CaO slaker fed with aq. Na<sub>2</sub>CO<sub>3</sub>, a Dorr jigger for the removal of sand etc., 3 causticisers, a Dorr thickener, a washing vessel, and a rotary filter for completion of washing. The filter cake is burned in a rotary kiln and the CaO used again. A min. of liquor should be used for slaking if the CaCO<sub>3</sub> is to settle well. For the same reason the stirring time in the causticisers should be > 0.5 - 1.0 hr. The temp. should be < 85°. The separation of CaCO<sub>3</sub> from the NaOH liquor is the step in which variations most affect the economics of the process. Good separation depends on points mentioned above, also on the burning of the CaO, concn. of NaOH, and velocity of stirring. C. I.

Stability of bleaching powder and hypochlorite solutions. J. H. WALTON and C. S. R. AYYAR (Agric. and Live Stock India, 1932, 2, 309-313).—After storage for 3 months at 27-32°, losses of available Cl from aq. solutions of bleaching powder (I), solutions of (I) in brine, and electrolytic hypochlorite solutions were 14.3, 13.6, and 7.8% respectively. Losses from solutions of (I) were considerably reduced by cold storage. Dry (I) is less stable than a 2-3% solution. A. G. P.

AcOH.—See III. Oxidation of cotton.—See V. Bottle glass and  $H_2SO_4$ .—See VIII.  $H_2$ .—See XI.

#### PATENTS.

Method of conducting catalytic exothermic gaseous reactions [ammonia synthesis.] A. T. LARSON, ASST. to DU PONT AMMONIA CORP. (U.S.P. 1,833,188, 24.11.31. Appl., 12.4.27).—In, e.g., the synthesis of NH<sub>3</sub> the pure fresh mixture of N<sub>2</sub> and H<sub>2</sub> is passed through a space between the wall of the apparatus and the catalyst container in order to prevent damage to the former by NH<sub>3</sub>, and, at the completion of that pass, is mixed with returned gases containing NH<sub>3</sub> and passed through the catalyst. B. M. V.

Manufacture of alkali cyanides.E. HENE (B.P.375,636, 22.5.31).—HCN is passed at  $360^{\circ}$  over  $Na_2CO_3$ formed by heating NaHCO<sub>3</sub> below 500°.The productcontains > 98% NaCN.A. R. P.

**Extraction and refining of alumina.** VEREIN. ALUMINIUM-WERKE A.-G., and H. GINSBERG (B.P. 375,476, 21.4.31).—Bauxite, FeS<sub>2</sub>, and coke are smelted in an electric furnace to produce Fe–Si–Ti alloy and an  $Al_2O_3$ – $Al_2S_3$  slag containing 14%  $Al_2S_3$ . The liquid slag is tapped into a second furnace and cooled slowly to 1400° to allow a crust of relatively pure  $Al_2O_3$  to form; the charge is then reheated at 1900° for 10 min., treated with more FeS<sub>2</sub>, Fe, and/or coal, and again cooled slowly. The purified  $Al_2O_3$  is then withdrawn and the residue returned to the first stage together with any sludge obtained by washing the  $Al_2O_3$  with HCl to remove sulphides and Fe. A. R. P.

Ca fluosilicate.  $H_2SO_4$  and NaCN.—See XVI. I preps.—See XX.

# VIII.—GLASS; CERAMICS.

Decomposition of bottle glass by sulphuric acid. L. FARCY (Ann. Falsif., 1932, 25, 290-291).— A bottle used for the storage of battery acid splintered but did not disintegrate. The fragments contained crystals of K alum mixed with leaflets of mixed silicates. T. McL.

Solubility of lead glasses in water. S. MORIYASU (J. Soc. Chem. Ind., Japan, 1932, 35, 260-265 f; cf. B., 1931, 1094).—The solubility of Pb glasses in H<sub>2</sub>O depends not only on the % of K<sub>2</sub>O and Na<sub>2</sub>O present, but also on the ratios K<sub>2</sub>O/Na<sub>2</sub>O and PbO/SiO<sub>2</sub>. J. W. S.

Flow of glass in tanks. F. C. FLINT and A. K. LYLE, JUN. (J. Amer. Ceram. Soc., 1932, 15, 410–418).

Town's gas in the pottery industry. O. W. Roskill (Ind. Chem., 1932, 8, 277–282).

#### PATENTS.

Manufacture of sheet glass by rolling. Soc. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNY & CIREY (B.P. 377,637, 20.5.32. U.S., 23.5.31. Addn. to B.P. 361,839). Manufacture of double-walled flasks and vessels of glass in one operation at the glass furnace. GLASFABR. SOPHIENHÜTTE R. BOCK G.M.B.H., and W. HODECKER (B.P. 377,550, 8.12.31).

Device for manufacturing glass objects. N. V. MAATSCH. TOT EXPLOIT. VAN UITVINDINGEN (B.P. 377,408, 9.7.31. Holl., 12.7.30).

Manufacture of glass bands, tubes, rods, etc. Comp. Internat. Pour la Fabr. Mécanique du Verre, Assees. of Deuts. Libbey-Owens Ges. F. Glasherstel-LUNG A.-G. (B.P. 376,990, 22.8.31. Ger., 25.8.30).

Methods of connecting branch pipes to quartz tubes and similar quartz bodies of comparatively great dimensions. JENAER GLASWERK SCHOTT & GEN. (B.P. 377,071, 4.12.31. Ger., 9.12.30).

# IX.—BUILDING MATERIALS.

Production of free lime in Portland cement by heating. G. E. BESSEY (Cement, 1932, 5, 233-235).— Decomp. of  $3CaO,SiO_2$  in Portland cement takes place at temp. down to  $700^{\circ}$  but was not observed on heating at  $550^{\circ}$  for 6 hr.  $3CaO,SiO_2$  is unstable at  $<1350^{\circ}$ but decomp. occurs only with any observable velocity at approx.  $1050^{\circ}$  when heated alone or above  $600^{\circ}$  in the presence of catalysts, *e.g.*, free CaO and gypsum. C. A. K.

Behaviour of Portland cement when heated. G. A. ASHKENASI (Cement, 1932, 5, 240–245).—The proportion of CaO liberated by heating cement at about 1000° fluctuates between 9 and 11% and the composition of the residue which remains after dissolution of the CaO corresponds with the composition of a Ca disilicate assuming that  $Al_2O_3$  and  $Fe_2O_3$  are combined with 3CaO and 2CaO, respectively. Portland cement may therefore be considered as a disilicate with CaO in solid solution rather than as 3CaO,SiO<sub>2</sub>. C. A. K.

Hardening of Portland cement. F. F. TIPPMANN (Cement, 1932, 5, 236-239).—Evidence supporting the existence of Ca(OH)<sub>2</sub> crystals in cement in process of hardening is re-stated and the existence of Ca hydrosilicate is regarded as an assumption (cf. B., 1932, 64). C. A. K.

Constancy of volume of Portland cement. A. C. DAVIS (Cement, 1932, 5, 253-265).—A no. of accelerated tests for unsoundness of cement are described. These include the various hot- and cold-plunge tests on pats and the Le Chatelier expansion test. C. A. K.

Cementation of oil wells with the use of an accelerator. KOREPANOV and MUCHINSKI (J. Inst. Petroleum Tech., 1932, 18, 197—212).—The process of setting of cement mixes and slurries  $(23 \cdot 5 - 70\%)$  H<sub>2</sub>O) was studied at 18—90°, with and without the addition of 0.5-5% of CaCl<sub>2</sub> as accelerator. The higher the temp. the more rapid is the setting, although with CaCl<sub>2</sub> present the influence of temp. is not so marked. The higher temp. also increased the strength of all mixtures tested. In presence of H<sub>2</sub>O with increase of pressure, the initial and final sets take place earlier. H. S. G.

Slănic trass. II. A. STEOPOE (Bul. Chim. Soc. Române Stiin., 1929, 31, 275-280).-Mortars were prepared

containing sand 3 pts., cement 1 pt., the latter being composed of trass and CaO in varying proportions. The strongest product contained 20-25% of trass in the cement and was mixed with only 10% of H<sub>2</sub>O. When Portland cement was used instead of CaO, the strongest mortar was that containing 20% of trass in the cement. D. R. D.

Influence of time of agitation and of addition of trass on the strength, setting time, and contraction of fluid cement mixes. A. STEOPOE (Bul. Chim. Soc. Române Stiin., 1929, 32, 55-58, 73-78, 89 -94, 95-98). Comparative tests on 1:1 cement mixes with 25% H<sub>2</sub>O with or without 25% addition of trass mixed for 5-170 min. show that the effect of time variation differs with different makes of cement and each case must be separately studied. The mixtures with trass vary less than pure mixtures, but inversely. Addition of 25% of trass (on the wt. of cement) produced strength reductions of the order of 25%. Mixes including lime and tufa debris after 7 days' setting were too weak for satisfactory use. After longer periods addition of trass improves the strength of common Portland cement somewhat, but not that of very finelyground cement. Contraction is reduced by trass addition in the case of common cements, as also by prolonged contact with H<sub>2</sub>O. With fine cements trass increases the contraction. C. I.

Wood preservation in the sea. J. H. ORTON (J. Brit. Wood Pres. Assoc., 1932, 2, 81-86).—The failure of many preservatives and anti-fouling compositions on exposure to sea-water was due to erosion of the paints, which indicates that the vehicle is as important as are its poisonous constituents. Coal tar and coal tar diluted with naphtha were good preservatives, as was also a green anti-fouling paint which contained As, Cu, and Zn. The former do not prevent superficial growths. C. A. K.

Wood structure and penetration of preservatives. F. J. POPHAM (J. Brit. Wood Pres. Assoc., 1932, 2, 87—91).—Preservatives are classified as to whether they do or do not enter the cell  $H_2O$ . Swelling did not occur unless creosote contained > 20% of tar acids, so that creosote as ordinarily applied enters the wood only by means of the cell spaces. The penetration rate of  $H_2O$  into wood is extremely slow and the length of time for impregnation by salt solutions is uneconomical. Little penetration of green timber is effected if it contains > 25% of moisture. It may be assumed that lighter woods, *i.e.*, those with greater void space, require more preservative solution than the denser varieties.

## PATENTS.

# C. A. K.

Kilns and associated plant for the heat-treatment of [refractory etc.] materials. H. C. and R. C. ERITH (B.P. 375,404, 26.3.31).—A range of brick kilns is arranged on a U-shape plan, and a portable brick-making machine is brought to the entrance of each kiln in turn by an overhead crane and the fuel and other materials by a conveyor. The bricks are discharged direct into rail wagons. B. M. V.

Natural or artificially coloured lime or cement mortar or plaster. H. PLAUSON (B.P. 375,144, 11.9.31. Ger., 6.10.30).—A colouring agent is coated with a thin layer of  $SiO_2$  gel either by direct addition or by forming it *in situ*, and the dried product is mixed with cement and/or CaO. C. A. K.

Production of glimmering or iridescent appearance on cement or like coatings or surfaces. A. WEITHALER (B.P. 375,045, 26.5.31).—A facing of cement, which may contain trass, casein, dialysed SiO<sub>2</sub>,  $Ca(OH)_2$ ,  $CaCO_3$ , etc. to give a glassy surface, is etched with a mordant to cause a light-interference effect. The surface is then washed and given a protective coating. C. A. K.

Preparation of concrete. M. A. MORRISSEY and A. F. MOORE (U.S.P. 1,832,875, 24.11.31. Appl., 1.10.30).—A cement is mixed with an aggregate and  $H_2O$ , and after complete hydration of the cement excess  $H_2O$  is removed by pressure or suction.

C. A. K. **Protecting and curing concrete.** A. H. STEVENS. From AMER. REINFORCED PAPER Co. (B.P. 375,196, 25.11.31).—During the curing period newly-laid concrete is covered by a multi-ply waterproof sheet which consists of sheets of paper cemented together with a waterproof adhesive. C. A. K.

Production of binding agents for constructing or covering roads etc. G. A. LASSEUR and T. V. TARALOU (B.P. 375,460, 7.4.31. Fr., 7.4.30).—A mixture of CaO 15,  $H_2O$  45, an alkali silicate 100, and tar 200 pts., with or without 30 pts. of pitch, forms a colloidal slip which is used for spraying roads. C. A. K.

Material for roads and ways, floors, walls, sports grounds, etc. E. O. COWPER (B.P. 375,288 and 375,298, 13.3.31. [A] Addn. to B.P. 338,247).— (A) Volatile mineral oils, particularly those which possess a softening action on rubber, e.g., a mixture of  $C_6H_6$  and anthracene oil, are added to facilitate the coating and penetration of the road mixture. (B) An oil-treated aggregate is combined with crushed waste rubber, which may also be treated with oil and bonded with rubber latex and/or bitumen. C. A. K.

Shuttles from wood. R. ENGELHARDT, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,832,971, 24.11.31. Appl., 27.6.30. Ger., 31.7.29).—Wood for use as shuttles is impregnated with a stable chlorinated hydrocarbon, e.g., chlorinated  $C_{10}H_8$ . C. A. K.

[Fluted or ribbed] non-conducting coverings for heat and sound. A. JACKSON and R. CLEGG (B.P. 377,020, 2.10.31).

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

Reactions in the galvanising of iron. H. GRU-BITSCH (Monatsh., 1932, 60, 165—180).—Solubility curves of five pearlitic steels (0.09-0.50% C) in pure Zn are similar and show a max. between 480° and 520° which is practically independent of C content. A 0.87% C steel of pearlitic, sorbitic, and martensitic structure has a very different solubility in Zn with max. at 450° and 495° and a rapid increase above 530°. The thickness and nature of layers of Zn-Fe alloys on Armco Fe and on the pearlitic steels, studied from solubility and by photomicrographs, vary greatly with and are characteristic for temp., and are related to solubility of Fe in Zn (cf. B., 1931, 976). E. W. W.

Primary graphitisation of cast irons containing molybdenum. J. COURNOT and J. CHALLANSONNET (Compt. rend., 1932, 195, 46–48).—Addition of up to 2% Mo (as 20% Mo cast Fe) to cast Fe containing C 3.7, Si 2.3, Mn 1.4, S 0.03, P 0.07% causes no separation of carbide. With 2.9-7.7% Mo the free C diminishes progressively, giving rise to complex cementite cast Fe. Nodular graphite was found only in the thinner ingots, and change in its condition seems due only to conditions of melting and pouring, and not to the presence of Mo (cf. B., 1931, 250). C. A. S.

Deoxidation process in the production of silicon steel (transformer steel). P. BARDENHEUER (Naturwiss., 1932, 20, 405-407).-Total O and O combined with Si in mild steel have been determined during 90 min. after addition of ferrosilicon to deoxidise the metal, which is practically free from Mn, C, P, and S; 5-10 min. after the addition the SiO, content of the metal reaches a max., but only after 60 min. is conversion of the O into SiO<sub>2</sub> complete. Transformer steel with 4% Si has a min. watt loss when all FeO and MnO has been reduced. Total O is determined by heating with C in vac. in an induction furnace, O present as SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> by treatment of the metal at 400° with dry Cl<sub>2</sub> and determination of the SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> in the insol. residue, and O present as MnO and FeO by reduction in H<sub>2</sub>. The last-named procedure gives low results in the presence of Si and Al, and in this case MnO and FeO must be determined by difference from the results obtained in the first two methods. A. R. P.

Kinetics of the transformations during steel hardening. F. WEVER (Naturwiss., 1932, 20, 412— 416).—The mechanism of the 3 stages in the decomp. of austenite (cf. B., 1932, 426) has been investigated by magnetic methods and micrographic examination of the structure. In the first stage (pearlite transformation) the rate of diffusion of the C is sufficient to cause complete decomp. of the austenite into Fe<sub>3</sub>C and ferrite with a low C content; in the second stage the austenite is transformed into a metastable intermediate product of unknown composition, which then decomposes into ferrite and cementite, and in the third stage spontaneous transformation of the austenite into tetragonal martensite takes place and the martensite then slowly decomposes into ferrite and highly dispersed carbide.

A. R. P.

Creep limit of metals at high temperatures and the effect thereon of heat-treatment. W. ROHN (Z. Metallk., 1932, 24, 127—131).—The creep limit of Ni, Fe, and numerous Fe and Ni alloys with Mo, Cr, Mn, and Al has been determined in an electric tube furnace (described), the results being shown graphically. Previous annealing of all the metals tested at temp. well above the recrystallisation point increases appreciably the creep limit, especially at temp. above the region of cryst. recovery and recrystallisation.

A. R. P.

Potentiometric analysis in the steelworks laboratory. P. DICKENS and G. THANHEISER (Naturwiss., 1932, 20, 407-409).—S is determined by combustion of the steel in  $O_2$ , oxidation of the  $SO_2$  to  $H_2SO_4$  by  $H_2O_2$ , and potentiometric titration of the acid with NaOH; Mn in Co steel by oxidation to HMnO<sub>4</sub> with PbO<sub>2</sub> in HNO<sub>3</sub> and potentiometric titration with  $H_2C_2O_4$ ; Mo by titration of the Na<sub>2</sub>MoO<sub>4</sub> solution, obtained in the usual way, with Pb(ClO<sub>4</sub>)<sub>2</sub> or SnCl<sub>2</sub>; and V by potentiometric titration with FeSO<sub>4</sub> after oxidation with KMnO<sub>4</sub> followed by destruction of the excess with  $H_2C_2O_4$ . A great advantage of this process is the possibility of determining several elements one after the other in the same solution; examples of this are outlined, A. R. P.

Determination of small quantities of antimony in copper and its alloys. W. BOEHM and W. RAETSCH (Z. anal. Chem., 1932, 88, 321–324).—In the analysis of Cu alloys, addition of aq.  $NH_3$  to the  $HNO_3$  solution effects complete pptn. of the Sb provided that sufficient Fe<sup>TT</sup> is present. Blumenthal's statement to the contrary (B., 1928, 573) is attributed to the use of pure Cu and KSbO<sub>3</sub> in his work instead of a synthetic alloy. A. R. P.

Precision X-ray methods in alloy research. J. WEERTS (Z. Metallk., 1932, 13, 8—141).—A review of modern methods of determining the structure of alloys by X-rays and an account of recent work on Au-Pt and Cu-Al alloys and of the results of X-ray examination of alloys at elevated temp. A. R. P.

Magnetic analysis of internal strain. M. KERSTEN (Z. Physik, 1932, 76, 505—512).—It is shown that the magnetisation curves of stretched Ni wires may be used to measure internal strain. A. B. D. C.

Application of the ideal mass-action law to metallurgical processes. F. KÖRBER [with W. OELSEN] (Naturwiss., 1932, 20, 409—412).—The deviations from the ideal mass-action law found by van Laar and Lorenz (A., 1925, ii, 866) in the reactions between two molten metals and their chlorides are shown to be due to insufficiently rapid quenching of the specimens taken for analysis. When the quenching is rapid the equilibrium curves are in good agreement with the ideal law, the equilibrium const. is a function of the temp. throughout the system, and no change of direction of the curves can be detected. A. R. P.

Corrosion of metals by [vegetable] tan liquors. III. Corrosion of submerged metals in movement. M. P. BALFE and H. PHILLIPS (J. Soc. Leather Trades' Chem., 1932, 16, 345—353; cf. B., 1932, 552).—Fe was seriously corroded by and caused discoloration of tan liquors, e.g., a weak suspender liquor containing H<sub>2</sub>SO<sub>4</sub> and lactic acid, a similar liquor containing subhited mimosa bark extract also, unused quebracho and mimosa bleaching extracts, and an unused chestnut extract. Similar corrosion to a smaller extent was produced on a Ni–Fe alloy. Ni–Cu alloys were definitely attacked. Resistance to chestnut extract was in the order monel metal > brass > Cu or phosphor-bronze. In presence of SO<sub>2</sub>, Ni and monel metal are more corroded than is brass. Fe–Cr and Fe–Cr–Ni alloys are least corroded of any metals by vegetable tan liquors.

D. W.

Lead ore slags. W. STAHL (Z. anorg. Chem., 1932, 206, 356).—The mol. ratio basic oxides/ $SiO_2$  is 2/1·199. The slag consists, therefore, of orthosilicates containing some metasilicate. E. S. H.

Determination of iron in aluminium. L. Szegö (Giorn. Chim. Ind. Appl., 1932, 14, 226-228).-To 2 g. of the Al turnings mixed with about 25 c.c. of H<sub>2</sub>O 10% aq. NaOH is gradually added, the liquid being finally warmed. When evolution of H<sub>2</sub> ceases, the solution is diluted, cooled, and filtered, the residue (Fe) being washed with hot H<sub>2</sub>O and dissolved in 1:10 HCl. The acid solution (about 25 c.c.) is boiled, decolorised with a few drops of SnCl<sub>2</sub> solution, diluted with boiled H<sub>2</sub>O, treated with 5 c.c. of saturated HgCl, solution and 5 c.c. of an acid solution of MnSO4, and titrated with 0.05N-KMnO<sub>4</sub>: 1 c.c.  $\equiv 0.002792$  g. Fe. This method gives good results with Al containing 0.4% Ti and 0.2% Si. T. H. P.

Electrometallurgy of aluminium. P. P. FEDO-TÉEV (Z. anorg. Chem., 1932, 206, 267-269).—In the electrolysis of fused Na<sub>3</sub>AlF<sub>6</sub> in Al<sub>2</sub>O<sub>3</sub> the former is the electrolyte and the latter simply a more or less non-ionised solvent. Na travels to the cathode and forms Na<sub>3</sub>AlO<sub>3</sub>, liberating Al, whilst the liquid around the anode becomes richer in AlF<sub>3</sub>. By the intermixing of the two fused salts Na<sub>3</sub>AlF<sub>6</sub> is formed again. It is an advantage to have excess of AlF<sub>3</sub> to lower the m.p. of the bath, to replace the AlF<sub>3</sub> which volatilises, and to encourage the re-formation of Na<sub>3</sub>AlF<sub>6</sub> and so prevent the separation of metallic Na formed by the electrolysis of NaF or Na<sub>3</sub>AlO<sub>3</sub> absorbed by the C lining of the bath. M. S. B.

Controlling influences in the electrostatic purification of blast-furnace gas. K. GUTHMANN (Stahl u. Eisen, 1932, 52, 529-539).—The effects of temp., moisture and  $CO_2$  content of the flue gas, rate of flow, size of particle and chemical composition of the suspended dust, deposition potential, and state of the electrodes on the efficiency of purification of the gas are shown in a series of graphs. The best results are obtained when the gas and dust particles have a certain definite conductivity which is obtained when the gases are just saturated with moisture at the temp. at which they are admitted to the precipitator. The relative costs of the three processes, dry, combined wet and dry, and wet, are tabulated ; from these data the last-named process appears to be the most economical. A. R. P.

Importance of X-ray interference in metallurgy. E. SCHMID (Naturwiss., 1932, 20, 530-536).

#### PATENTS.

Operating reverberatory furnaces and smelting ores therein. E. W. DAVIS, ASST. to DAVIS STEEL PROCESS CORP. (U.S.P. 1,833,321-2, 24.11.31. Appl., [A] 23,11.29, [B] 25.11.30. [A] Renewed 18.7.31).— (A) A sloping charge is maintained at the sides of the furnace which serves practically as a hopper feed, and the material exposed on the furnace bed lies at an angle less than the angle of repose of the charge. (B) Openings are provided in the side of a furnace to provide a feed which fulfils these conditions. C. A. K. Roasting furnace. J. B. READ and M. F. COOL-BAUGH, Assrs. to COMPLEX ORES RECOVERIES CO. (U.S.P. 1,833,590, 24.11.31. Appl., 29.7.25).—In a multiplehearth furnace the top and lowest hearths are provided with air outlets, as is also one of the middle hearths, to cause a flow of air both upwards and downwards. C. A. K.

Treatment of [roasting zinc] sulphide ores. A. E. WHITE. From NICHOLS COPPER Co. (B.P. 372,771, 6.2.31).—Blende is roasted in a multiple-hearth furnace the discharge from which is heated above 900° by the hot gases containing 10—15% SO<sub>2</sub> produced by burning molten S with air preheated at 250°. In this way ZnSO<sub>4</sub> is completely decomposed. A. R. P.

Cooling the port structures of open-hearth furnaces. TERNI SOC. PER L'INDUSTRIA E L'ETTRICITA, and F. FIORELLI (B.P. 374,333, 20.8.31).—Ducts are provided for admitting cold inert gas into the gas stream to cool it sufficiently to protect the masonry of the furnace head. A. R. P.

[Electric] furnace for treating metal. E. BOR-NAND and H. A. SCHLAEPFER (U.S.P. 1,833,065, 24.11.31. Appl., 29.11.29. Fr., 23.1.29).—In an arc furnace with a no. of stationary electrodes the current is conveyed to an electrode passing through a refractory block capable of vertical movement through the cover. The block has a cavity on its bottom face and is lowered intermittently into the metal bath to assist intimate mixing. C. A. K.

Manufacture of metals, metal mixtures, or alloys [e.g., iron-chromium alloys]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 374,560, 2.4.31).— Fe powder from  $Fe(CO)_5$  is heated with  $CrCl_3$  or other heavy-metal chloride at 900° in an atm. of N<sub>2</sub> to obtain a sintered Fe-Cr or other similar alloy. A. R. P.

[Improving the yield point of] iron and steel alloys. F. KRUPP A.-G. (B.P. 374,953, 19.3.31. Ger., 22.1.31).—Fe or steel free from Si but containing Mn, Ni, and/or Cr is alloyed with < 1% Ti and the castings are either quenched from 1300—700° and tempered at 250—650°, or cooled very slowly from above 700°. A. R. P.

Nitrogenisation of iron, steel, and alloys thereof. ELECTRO METALLURG. Co., Assees. of J. J. EGAN (B.P. 374,880, 12.3.31. U.S., 14.3.30).—The articles are surrounded by filings, powder, or gauze of steel containing about 1% Al which has previously been nitrogenised by heating in  $NH_3$  at 460°, and the whole is heated in  $NH_3$  at 460—580°. A. R. P.

Pickling or cleaning of iron or steel. T. W. COSLETT (B.P. 375,599, 15.8.31).—The pickle comprises aq.  $H_2O_2$  with HNO<sub>3</sub>, HCl, or a mixture of HNO<sub>3</sub> with  $H_2SO_4$  or  $H_3PO_4$ . A. R. P.

Imparting a clean, bright surface to ferrous articles [by pickling]. HANSON-VAN WINKLE-MUN-NING Co., Assees. of G. B. HOGABOOM and W. R. KING (B.P. 375,573, 16.7.31. Canad., 9.10.30).—The articles are treated anodically at 25—35° with a c.d. of 80—250 (150) amp. per sq. ft. in 40—50% H<sub>2</sub>SO<sub>4</sub> containing preferably a little CrO<sub>3</sub>. A. R. P.

British Chemical Abatracts

Coating metals such as iron and steel with lead. J. W. PRITCHARD, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 375,361, 24.3.31).—The metal is coated with molten Pb in an atm. of  $Cl_2$  or HCl, or the Pb is melted on to the metal by means of a  $H_2$ - $Cl_2$  burner.

A. R. P. Manufacture of sintered [carbide] alloys. L. MELLERSH-JACKSON. From DEUTS. EDELSTAHLWERKE A.-G. (B.P. 374,710, 2.9.31).—Hard alloys are made by sintering mixtures of one or more carbides of Ti, V, Mo, W, or U with org. salts of Co, Ni, or Fe, e.g., NiC<sub>2</sub>O<sub>4</sub> or CoC<sub>2</sub>O<sub>4</sub>, at 600—800° in H<sub>2</sub>, crushing the product, pressing into shape, and again sintering in H<sub>2</sub>.

A. R. P.

**Refining of metals [steel, copper, aluminium].** J. W. FLANNERY (B.P. 372,326, 28.11.30. U.S., 29.11.29). —The refining slag is placed at the bottom of the furnace and the metal melted down above it by means of heat applied at the top of the furnace; rise of the slag through the molten metal effects a thorough removal of impurities. For steel the refining material comprises hydroxides or carbonates of K, Na, or Ba with or without CaC<sub>2</sub> or a mixture of CaCO<sub>3</sub> and C; for Cu and Al it consists of a mixture of SiO<sub>2</sub> with KOH or Na<sub>2</sub>CO<sub>3</sub>. A. R. P.

Refining and preparing for casting magnesium and its alloys. F. BADGER, and BRIT. MAXIUM, LTD. (B.P. 375,743, 18.8.31).—Refining is effected at 800° with a flux comprising a mixture of 40% MgF<sub>2</sub> and 60% MgCl<sub>2</sub> containing 2—20% H<sub>2</sub>O, and the metal is cast from below the solid crust of flux while a current of inert gas is passed into the space between the crust and the metal surface. A. R. P.

Production of metal [aluminium] powders such as bronze colours. HARTSTOFF-METALL A.-G. (HAME-TAG) (B.P. 373,784, 27.5.31. Ger., 28.5.30).—Al is pulverised in an atm. of N<sub>2</sub>, CO<sub>2</sub>, or mixtures thereof containing 1—3% O<sub>2</sub> or H<sub>2</sub>O vapour, so that the ground particles immediately become covered with a protective Al<sub>2</sub>O<sub>3</sub> film. A. R. P.

Production of oxide coatings on aluminium and its alloys. VEREIN. ALUMINIUM-WERKE A.-G. (B.P. 374,806, 27.1.32. Ger., 18.12.31).—The metal is subjected to anodic oxidation in 8—10% aq.  $H_2C_2O_4$  using d.c. and a.c. alternately. A. R. P.

Production of thin films of metals [beryllium]. ELECTRICAL RESEARCH PRODUCTS, INC., Assees. of H. W. WEINHART (B.P. 374,447, 9.3.31. U.S., 12.3.30).—Pellets of Be are welded on to a thin W wire, which is then heated to incandescence in vac. so that the Be vaporises and condenses on the article to be coated. A. R. P.

Preparation of metals for painting. AMER. CHEM. PAINT Co., Assees. of A. DOUTY (B.P. 374,142, 5.3.31. U.S., 12.3.30).—The metal is sprayed with a suspension of colloidal clay (250 lb.) in a mixture of  $H_2O$  (32 gals.), 70%  $H_3AsO_4$  (9 gals.), EtOH (30 gals.), and BuOH (15 gals.) and the coating is dried at 100° and brushed off together with the oxide film and any dirt, grease, or oil. A. R. P.

Locally reinforcing the plating of [silver-]plated metal articles [e.g., spoons]. HANSEATEN BINDERWERK G.M.B.H. (B.P. 373,015, 12.8.31. Ger., 14.8.30).— Depressions are made in the parts to be reinforced and these are filled with Ag by electrodeposition while the remainder of the surface is coated with an insulating film; the blank is then cleared, pressed into spoonshape (during which operation the deposit is consolidated and hardened), and plated with Ag in the usual way. A. R. P.

Alloys. HANDY & HARMAN, Assees. of R. H. LEACH (B.P. 377,097, 8.1.32. U.S., 14.5.31).—See U.S.P. (1,829,903; B., 1932, 684.

Reheating furnaces [for billets]. F. KAUBA (B.P. 377,005, 7.9.31).

**Tuyères for cupola furnace.** R. STOTZ, O. BROSSARD, and R. GERISCH (B.P. 377,294, 24.4.31. Ger., 23.7.30).

Casting of copper bars. AMER. METAL Co., LTD., Assees. of W. F. EPPENSTEINER (B.P. 373,003, 16.7.31. U.S., 19.7.30).

Means for detecting flaws in metallic objects. SPERRY PRODUCTS, INC., Assees. of H. C. DRAKE (B.P. 377,500, 29.9.31. U.S., 13.10.30).

Welding rods.-See XI.

## XI.—ELECTROTECHNICS.

Transformer for small induction furnace. R. KÜMMICH (Z. Elektrochem., 1932, 38, 402-407).—Full details are given of the design of transformers for supplying small (3—12-kw.) induction furnaces; such instruments are unduly large, since the wt. of Fe required is inversely proportional to the square of the secondary load. A 10—12-kw. transformer is described.

H. F. G.

The Fauser cell for the electrolysis of water. C. PASTONESI (L'Ind. Chimica, 1932, 10, 583-590).— The asbestos fabric double diaphragm surrounding each electrode prevents the mixing of the generated gases which are obtained in a pure state. The danger of explosion is minimised. Active circulation of the electrolyte produced by the evolved gases diminishes the ohmic resistance and lowers the consumption of energy. Details of construction, operation, and running costs are given. O. F. L.

Manufacture of hydrogen by electrolysis. G. F. JAUBERT (Bull. Soc. Ing. Civ. France, 1931, 1997-2032). -In the electrolytic manufacture of H<sub>2</sub> the energy efficiency under the best conditions is 80%; the balance is chiefly lost as heat, and the loss is measured by the excess cell voltage necessary above 1.47, the theoretical figure. It depends on the internal resistance, which is modified by the accumulation of gas bubbles in the liquid. This efficiency is nevertheless much higher than that of chemical processes of manufacture. The Schuckert cell without diaphragm is employed by the French military authorities. The gas as collected contains 98.5% H<sub>2</sub> and is purified by treatment with porcelain impregnated with reduced Pd or Ni. The latter requires a temp. of 300° to effect combination of  $H_2$  and  $O_2$ . It is not suitable for  $O_2$  purification. A recent improvement consists in adding 70 g. of Mg(OH)2 per litre to the electrolyte. This coalesces the gas bubbles and improves the separation. Diaphragm cells which give a more complete separation employ a brass or Ni gauze,

or a colloid liquid on a metal armature. They may be uni- or bi-polar; in the latter case they are constructed similarly to closed filter-presses. In the case of the author's colloid diaphragm the two divisions of the cells are separated initially by a metallic gauze, the colloid is added to the solution and collects as semiliquid at the diaphragm; the purity of the gases rises steadily until after 42 hr. it has reached 100%. The theory of this is discussed. The diaphragms are impermeable to gas mols. and yet afford a free passage to ions. C. I.

Oxidation of paraffin etc.—See II. Al. Blastfurnace gas.—See X.  $p_{\rm H}$  determinations.—See XV.

## PATENTS.

Electric resistance furnaces. ALLMÄNNA SVENSKA ELEKTRISKA AKTIEB., and L. HÖGEL (B.P. 375,562, 7.7.31).—Resistance elements having the shape of corrugated bands are arranged along, and supported by, the furnace walls so that one broad side of the band radiates heat into the furnace chamber. J. S. G. T.

Fusion of substances in electric furnaces. ELEC-TRIC FURNACE Co., LTD., Assees. of E. F. NORTHRUP (B.P. 375,304, 16.3.31. U.S., 15.3.30).—The furnace lining in contact with the charge consists of the same material as the charge, but less pure, or, if desired, of ore, matte, or slag, and melting of the charge is indicated by descent of a rod resting on the charge. J. S. G. T.

Electrodes or welding rods for use in electric arc welding. QUASI-ARC Co., LTD., and A. P. STROH-MENGER (B.P. 374,160, 9.3.31).—A metal core is coated with extruded paste and wrapped with asbestos.

J. S. G. T.

Electrostatic apparatus for separating particles of dust in suspension in a gaseous fluid. J. BRÉ-MOND (B.P. 374,145, 6.3.21. Belg., 24.3.30).—The concave or hollowed parts of bent or corrugated electrodes, formed from a single length of metal and arranged so that their sharp vertical edges are tranverse to the gas current, are placed opposite the ionising electrodes and further therefrom than are the vertical edges, in order to act as passages down which deposited material falls. J. S. G. T.

Oxide cathodes for use in thermionic valves etc. TELEFUNKEN GES. F. DRAHTL. TELEGRAPHIE M.B.H. (B.P. 374,241, 20.5.31. Ger., 20.5.30).—A layer of W, oxides of W, and/or Cu oxide, applied to a core by means of "Cellon" or other dope, is heated in Ba vapour. J. S. G. T.

Indirectly-heated cathode for thermionic valves. S. LOEWE and B. WIENECKE (B.P. 375,481, 25.4.31).---Whistling, humming, etc. are prevented by coating non-emitting parts of the cathode with rough or porous non-conducting material, e.g., ZrO<sub>2</sub> or MgO, preferably mixed with a binder, e.g., water-glass. J. S. G. T.

Electric-discharge tubes. N. V. PHILIPS' GLOEI-LAMPENFABR. (B.P. 374,007, 23.2.31. Holl., 25.2.30). —One at least of the cold electrodes of a discharge tube filled with a gas mixture, *e.g.*, Ne and another gas, *e.g.*, A, of lower ionisation potential than an excitation potential of the former, contains an alkali metal arranged on a layer of alkali oxide, *e.g.*, Cs<sub>2</sub>O. J. S. G. T. [Filament for gas- and vapour-filled] electric Jamps. GEN. ELECTRIC CO., LTD., Assees. of PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 374,505, 7.7.31. Ger., 8.8.30).—A refractory core, e.g., of W, is coated with, or contains, electron-emitting material. J. S. G. T.

Photoelectric cells. N. V. PHILIPS' GLOEILAMPEN-FABR. (B.P. 375,595, 375,614, and 375,639, [A] 5.8.31, [B] 9.9.31, [C] 1.10.31. Holl., [A, B] 27.9.30, [C] 1.10.30). -(A) An anode, consisting, e.g., of a thin coating of Pt on glass, is arranged parallel to a substantially plane photoelectric electrode consisting, e.g., of Ag, the oxidised surface of which is coated with Cs, and is transparent to rays which liberate electrons from the photoelectric electrode. (B) An electrode consisting of photo-sensitive material, e.g., Cs, and, if desired, W particles, deposited upon a transparent layer, e.g., of CaF<sub>2</sub>, coating the wall of the cell is claimed. (c) A screen, e.g., of black paper, which is transparent to electrons but not to fluorescent radiation, is arranged between a photoelectric electrode constructed, e.g., in accordance with (B) and a fluorescent electrode consisting of ZnS or Zn silicate, mixed, if desired, with vaseline, applied to a thin metal sheet, e.g., of Pt.

J. S. G. T.

**Porous or permeable coatings for use in galvanic** cells. G. LÜDECKE (B.P. 375,388, 24.3.31. Ger., 26.3.30).—A mixture of cellulose fibres and acetylcellulose together with, if desired, EtOH and a solvent for acetylcellulose, *e.g.*, EtOAc, is claimed. J. S. G. T.

[Insulating] composition and article impregnated and coated therewith. W. H. WRIGHT, Assr. to SCHENECTADY VARNISH Co. (U.S.P. 1,833,810, 24.11.31. Appl., 22.11.28).—The product of the reaction between a mixture of rosin or other resin and acaroid gum with glycerol (e.g., 5%) is used as a coating composition for transformer coils. E. L.

Electrodes and other metal elements for vacuum electric devices. A. J. MANDELL (B.P. 376,727, 30.10.30).—See U.S.P. 1,769,229; B., 1931, 167. [Stat. ref.]

Electric ozone generator. J. M. DALLY (B.P. 376,788, 15.4.31).—See U.S.P. 1,803,600; B., 1931, 1140.

Gas-filled discharge tubes. N. V. Philips' GLOEI-LAMPENFABR. (B.P. 377,041, 24.10.31. Holl., 31.10.30).

X-Ray apparatus [with multi-filament tubes]. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of L. L. CALL (B.P. 377,496, 25.9.31. U.S., 27.9.30).

[Helical, zig-zag, or U-shaped] hot cathode forsingle and multi-grid thermionic valves. RADIOAKT.-GES. D. S. LOEWE, and P. KAPTEYN (B.P. 377,564, '13.1.32. Ger., 14.1.31).

Incandescence electric lamps. WESTINGHOUSE LAMP Co., Assees. of W. E. ANDERSON (B.P. 377,554, 14.12.31. U.S., 13.12.30).

[Combined arc and incandescence] electric lamps. Brit. Thomson-Houston Co., Ltd., Assees. of R. F. Strickland (B.P. 375,477, 22.4.31. U.S., 22.4.30).

Furnace for treating metal. Fe articles. Coatings on Al. Ag plate.—See X.

# XII.-FATS; OILS; WAXES.

Changes in fats during frying. F. R. PORTER, H. MICHAELIS, and F. G. SHAY (Ind. Eng. Chem., 1932, 24, 811—813).—Very little free fatty acid is produced when fats are heated evenly (in an oven) at  $176 \cdot 5^{\circ}$  and 232°, or even when some local over-heating occurs. The rate of formation of acid is small up to approx. 0.75% acid, but thereafter increases rapidly. If H<sub>2</sub>O is continuously present, even at  $176 \cdot 5^{\circ}$  acid formation is rapid, and acrid vapours (acraldehyde) are evolved. The flavour of the food is impaired when the free acid exceeds about 2-4.4%. E. L.

Goats' tallow of Swiss origin. J. PRITZKER and R. JUNGKUNZ (Pharm. Acta Helv., 1932, No. 2, 6 pp.).— Rendered offal fats from she-goats ( $\frac{1}{2}$ —3 years old) resembled ordinary tallow, were lacking in any special odour, and had m.p. 45—48°,  $d^{100}$  0.8570—0.8565, acid val. 0.4—1.1, sap. val. 196.5—195.3, I val. 38.6—32.4. Reichert-Meissl val. 0.77—1.1, Polenske val. 0.7—0.8, fatty acids 95—95.3%, solid acids (Bertram) 54.5—62.9% (on fat), "isooleic" acid (Grossfeld, vaccenic acid?) 1.2—2.8% (on fat). Arachidic acid was absent and the fat behaved like beef or mutton tallow as regards the Bömer no. and the Kreis-Roth fractional pptn. E. L.

Hydrogenation of fats. I. Influence of catalyst concentration on selective hydrogenation of cottonseed oil. D. R. DHINGRA, T. P. HILDITCH, and A. J. RHEAD. II. Course of hydrogenation of cottonseed oil by the Bolton-Lush continuous hydrogenation process (drip method). T. P. HILDITCH and A. J. RHEAD. III. Relative amounts of solid and liquid oleic acids present at different stages of the hydrogenation of olive and cottonseed oils. T. P. HILDITCH and E. C. JONES (J.S.C.I., 1932, 51, 195-198 т, 198-202 т, 202-203 т).--I. The amounts of saturated, isooleic, oleic, and linoleic acids in the mixed acids from cottonseed oils (400 g.) hydrogenated to various stages by the agitation process at 130°, 170°, and 200° in presence of varying amounts of Ni-kieselguhr (2, 5, 10 g.) have been determined, mainly by the method of Cocks, Christian, and Harding (A., 1931, 935). The results confirm earlier data in that selective hydrogenation of linoleic glycerides becomes more marked with increasing temp. of reaction, and that formation of isooleic derivatives is also more evident at the higher temp. Selectivity (and, to a less extent, isooleic production) is favoured by increasing concn. of catalyst in the oil.

II. Similar analyses to the above of partly hydrogenated cottonseed oils carried out by the continuous process ("drip" method) with a stationary Ni catalyst confirmed the observations of Lush (J.S.C.I., 1923, 42, 219 T) that under these conditions the hydrogenation process is less selective in its general results and that the amount of *iso*oleic derivatives produced is also less than at corresponding stages in the agitation process. Detailed examination of the glyceride structure of three partly hydrogenated fats revealed the presence of much more completely saturated glycerides than at corresponding stages of the agitation process, but in the nonfully-saturated part of the oil, hydrogenation had proceeded for the most part selectively and similarly to the course of the action in the agitation process. It is suggested that the difference in composition of the products results from the difference in mechanical conditions in the two processes.

III. In the agitation process, (i) selective hydrogenation of polyunsaturated glycerides generally leads to the production of more *iso*oleic than ordinary oleic derivatives; (ii) hydrogenation of monoethylenic derivatives (whether oleic, *iso*oleic, or a mixture of the two) is usually accompanied firstly by changes (usually augmentation) in the ratio of solid to liquid oleic acids, secondly by a period in which this ratio reaches an upper limit but during which its rate of variation is relatively slow, and finally the ratio may again decline to a certain extent.

Solvent extraction of castor-oil seeds. M. C. SWISHER and G. W. FIERO (J. Amer. Pharm. Assoc., 1932, 21, 579-582).—Extraction of crushed castor-oil seeds with a MeOH-EtOH mixture, removal of the solvent in vac., deodorisation by steam, and decolorisation by clay-C mixtures at 65-75° gives an oil satisfying U.S.P. requirements (except I val.) and more stable than commercial expressed oils. In sunlight at 30-35° it slowly develops a disagreeable odour and taste and becomes acid.  $CO_2$  hastens and  $N_2$  retards formation of acid. R. S. C.

Influence of climate during ripening on the quality [composition] of the oil of Linum usitatissimum. S. IVANOV (Allgem. Oel- u. Fett-Ztg., 1932, 29, 149—150).—The same (Nolinsk) flax seed was grown under different conditions; the oils from the crop obtained in Nolinsk (57.8° N. lat.), Davos (1550 m. above sea-level, cold and wet season), and Liebefeld (550 m. alt., cold season) had I vals. 185—188, 189.6, and 188.4, respectively (CNS vals. 99.9, 104.3, 104.5), but the oil from a crop ( $\frac{1}{2}$  g. seed) bred in a hot-house (25—35°) had I val. only 92.57. E. L.

Composition of Philippine rice oil (Ramai variety). A. O. CRUZ and A. P. WEST (Philippine J. Sci., 1932, 48, 5—12; cf. B., 1932, 560).—The  $\text{Et}_2\text{O}$ -extracted oil (18% dry basis) from the bran (polishing) was refined with kieselguhr, suchar, etc. and had  $d_4^{*0}$  0.9059,  $n_D^{*0}$  1.4662, acid val. 42.2, sap. val. 185.9, unsaponifiable matter 4.02%, I val. 99.3[?]; oleic and linoleic acids were identified. The saturated acids consisted chiefly of palmitic acid, with minor amounts of stearic acid and traces of myristic, arachidic, and lignoceric acids. E. L.

Composition of Philippine soya beans and soyabean oil. A. O. CRUZ and A. P. WEST (Philippine J. Sci., 1932, 48, 77-88).—A short survey of the nutritional factors of soya beans is given. The Philippine beans are rich in fats (20%), proteins ( $39\cdot1\%$ ), and carbohydrates ( $24\cdot96\%$ ). The oil had I val. 128.4, sap. val. 193.3, unsaponifiable matter 1.0%, unsaturated fatty acids (corr.)  $81\cdot5\%$  (I val. 151.3), saturated fatty acids (corr.)  $12\cdot65\%$ . The composition of the fatty acids is given as: ( $\alpha$ -)linolenic 2.1%, linoleic 53.0%, oleic  $30\cdot1\%$ , palmitic 8.9%, stearic 3.8%, and arachidic 0.6%.

Composition of Philippine talisay oil from the seeds of *Terminalia catappa*, Linn. A. O. CRUZ

and A. P. WEST (Philippine J. Sci., 1932, 48, 13–19). Et<sub>2</sub>O-extraction of the kernels yielded 50% (3% of whole seed) of edible oil, also known as "Indian almond oil." The earth-clarified pale yellow oil had:  $d_{1}^{*0}$ 0.9046,  $n^{30}$  1.4644, I val. (Hanus) 75.4, sap. val. 193.2, unsaponifiable matter 0.54%, acid val. 2.5, saturated acids (corr.) 32.62%, unsaturated acids (corr.) 61.01% (I val. 122.9). The oil consisted of : acids as glycerides : linoleic 22.9%, oleic 40.9%, myristic 1.0% palmitic 28.5%, stearic 4%, arachidic 0.75%. E. L.

Iodine value and refractive index of perilla oil. C. A. LATHRAP (Ind. Eng. Chem., 1932, 24, 826—827).— 6 samples of cold-pressed oils from authentic clean seed (Manchurian and Korean) had  $d_{15}^{15}$  0·9324—0·9355,  $n^{25}$  1·4800—1·4820, acid val. 0·5—1·5, sap. val. 191·1—191·8, I val. (Wijs) 192—208·6, (Hanus,  $\frac{1}{2}$  hr.) 187·1—200·4, Lovibond-yellow 30—40, -red 1·8—1·1. Routine analyses of commercial samples gave I val. (Wijs) 207—193·3 (aver. 200·4),  $n^{25}$  1·4802—1·4818 (aver. 1·4811, 20% being > 1·4815). The relation of n to I val. was approx. linear. E. L.

Soap manufacture. R. KRINGS (Allgem. Oel- u. Fett-Ztg., 1932, 29, 157–160).—An appreciation of the new principle of obtaining rapid and complete saponification of fat by intimate mixing (at about  $40-70^{\circ}$ ) with powdered alkali and a min. of  $H_2O$  (cf. U.S.P. 1,831,610; B., 1932, 778). Saponification is completed (with spontaneous rise of temp.) within 1 hr. and the product is cooled and worked directly between chilled rolls. E. L.

Fatty acids and mineral oils.—See II. Soap in dry-cleaning.—See VI. Oil boiling. "Olovine." —See XIII. Rubber seed.—See XIV. Sulphonated castor oils.—See XV.

#### PATENTS.

**Purifying [bleaching] oils and fats.** METALLGES. A.-G. (B.P. 375,381, 20.3.31. Ger., 4.4.30).—The oil (e.g., maize oil) is neutralised with NaOH and then bleached by heating or hardening, and the fatty acids produced are removed by steam-distillation in vac. during or after the heat-treatment. E. L.

Manufacture of preparations containing higher fatty acid derivatives. A. G. BLOXAM. From Soc. CHEM. IND. IN BASLE (B.P. 371,023, 13.1.31).-Emulsions of fatty esters above  $C_7$  are obtained by heating them with > 0.5 mol. of a diamine having 1 tert. and 1 primary or secondary NH2-group (with or without conversion into quaternary salt by alkylation or aralkylation), and pouring the product into dil. acid, or into conc. acid and subsequently diluting. According to the amount of acid used, acid, neutral, or alkaline reacting emulsions are obtained. Examples are : boiled linseed, olive, or castor oil, lard, beeswax, or montan wax with  $\beta$ -diethylaminoethylamine; castor oil with β-amino-β'-diethylamino-n-propyl alcohol or N-cyclohexyl-N-ethylethylenediamine; neatsfoot oil with 1-βaminoethylpiperidine or *m*-aminodimethylaniline; raw linseed oil with N-benzyl-N'N'-diethylethylenediamine. C. H.

Distilling apparatus.—See I. Drying oils.—See XIII.

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

Olovine. New medium for oil varnishes and enamels. T. H. BARRY and L. LIGHT (Brit. Plastics, Synth. Appl. Finishes, 1932, 3, 3-4, 28-29).--"Tekaols" consist of linseed stand oils from which the low-polymerised, highly dispersed portion has been removed. On exposure they do not oxidise, but polymerisation is pronounced ; films dry more rapidly, with or without driers, than stand-oil films and are more durable, since linoxyn formation is reduced. Zn white and Ti white settle much less rapidly in "tekaol" than in stand oil having the same  $\eta$ . "Olovine" varnish is prepared by heating a "tekaol" with a polyvinyl ester (acetate). The product is in chemical union, not colloidally dispersed, is sol. in a wide range of solvents, and polymerises to form colourless, hard, H<sub>2</sub>O-resistant, very durable films which do not subsequently yellow. Drying is accelerated by Co. "Olovine" in BuOAc can be used in conjunction with nitrocellulose. S. M.

Effect of carbon dioxide in oil and varnish boiling. R. BÜRSTENBINDER (Farbe u. Lack, 1932, 357).—When a stream of  $CO_2$  is conducted almost to the bottom of the boiling pot the contents are stirred, local overheating is prevented, and loss of volatile matter reduced. The product has lower acid val. and its colour is lighter than when the  $CO_2$  is introduced just below the surface or merely replaces the air above the oil. S. M.

Phenol-acraldehyde ["acrolite"] resins. B. V. MAXOROV and K. A. ANDRIANOV (Ind. Eng. Chem., 1932, 24, 827-832).-The effects of catalysts on the viscosity of these resins, and the dependence of rate of condensation, colour, and general properties on the nature and proportions of the constituents were studied. With acid catalysts the rate of condensation reaches a max. when 5 mols. of acraldehyde are used per mol. of PhOH, and the reaction is rapid at first but slows up. With basic catalysts a 2.5:1 ratio gives max. velocity, the rate being slow at first and rising rapidly towards the end of the reaction. Cresols and xylenols give resins of higher m.p. than does PhOH, whilst the xylenol resins show the highest rate of conversion into the infusible, S. S. W. insol. condition.

Determination of plasticity of mouldable materials. M. FORRER (Brit. Plastics, 1932, 4, 19–20, 51–52).—The deformation of a plastic when compressed in a mould depends on nature of material, rate of polymerisation, presence of lubricants, shape and condition of mould, temp. and pressure applied, etc. Several methods for obtaining an arbitrary measure of plasticity of a given material, involving the variation of one of the three factors : deformation, time, and pressure, the other two being kept const., are described, and the need for a standard procedure is stressed. S. S. W.

Casein.—See V. Wood preservation.—See IX. Rubber seed.—See XIV.

## PATENTS.

Coating and impregnation of cords or ropes with plastic masses. CHEMIEPRODUKTE GES.M.B.H. (B.P. 375,433, 30.1.31. Ger., 6.2.30).—The cords are passed one or more times through plastic masses comprising chiefly petroleum-distillation residues, *e.g.*,

SINS.

protoparaffins, soft bitumens, and fillers, e.g., china clay, talc, heated to semi-liquid state. S. S. W.

Improvement of drying oils and coating or impregnating compositions made therefrom. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 375,002, 10.4.31).—Weather-proof coatings for stonework etc. are made by mixing drying oils with higher aliphatic alcohols, prepared by catalytic reduction of fatty acids or by the oxidation of paraffin hydrocarbons; in the latter case any accompanying acidic components may be converted, wholly or partly, into metal (e.g., Al, Zn) salts, and added in this form. E. L.

[Emulsion-]paint. H. E. LLOYD, ASST. to RUBEROID Co. (U.S.P. 1,833,612, 24.11.31. Appl., 18.2.27).—The paint comprises ammoniated fatty acid pitch, a neutral aq. solution of an alkali caseinate, and, if desired, pigment, asphalt, and/or a volatile hydrocarbon solvent. S. S. W.

Paints [of improved flow]. H. BERNARD, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,833,274, 24.11.31. Appl., 27.8.28. Ger., 2.9.27).—An unsaturated aliphatic carboxylic acid below  $C_{12}$ , and containing a double linking in the  $\alpha$ -position, e.g., crotonic or sorbic acid (about 5%), is incorporated in paints comprising drying oil (stand oil), diluent, drier, and pigment (ZnO).

S. S. W.

Preparation of lithopone-oil pulp or paint mixtures. J. F. BROEKER, ASST. to KREBS PIGMENT & COLOR CORP. (U.S.P. 1,832,476, 17.11.31. Appl., 27.11.27).—15.5 pts. of linseed oil (acid val. 7) or of other drying oil (acid val. >4) are added gradually at 82—88°, with vigorous agitation, to a suspension of 100 pts. of lithopone in 200 pts. of H<sub>2</sub>O which also contains Na<sub>3</sub>PO<sub>4</sub> or other alkaline saponifying agent. The H<sub>2</sub>O separates in the upper layer after 10—30 min. further agitation. S. M.

Soft-textured lithopone. F. G. BREYER, Assr. to KREBS PIGMENT & COLOR CORP. (U.S.P. 1,832,355, 17.11.31. Appl., 14.1.29).—The lithopone before calcination is intimately mixed with 0.25-2% of basic Mg carbonate [approx. 4MgCO<sub>3</sub>,Mg(OH)<sub>2</sub>,5H<sub>2</sub>O]. A dispersing agent, *e.g.*, Na resinate, soap, Turkey-red oil, or Na silicate, may also be added. S. M.

Manufacture of a brown pigment from sludge of titanium. N. SPECHT, ASST. to KREBS PIGMENT & COLOR CORP. (U.S.P. 1,832,666, 17.11.31. Appl., 22.6.26. Ger., 22.8.25).—The sludge obtained by treating titaniferous ores with  $H_2SO_4$  and then  $H_2O$  is nearly neutralised with alkalis, excess of ZnO is added, and the product washed, filtered, dried, and heated to 700— 850° according to the shade of brown required.

S. M.

[Non-agglomerating] pigment. W. J. O'BRIEN, Assr. to GLIDDEN Co. (U.S.P. 1,832,416-8, 17.11.31. Appl., [A] 28.5.27, [B, C] 1.2.28).—Finely-divided pigments, e.g., lithopone, the particles of which are coated with 0.5-1 wt.-% of (A) oleic acid, (B) stearic acid or other fatty acid, C<sub>16-18</sub>, are claimed. (c) For use in water paints the particles are coated with 0.1-1 wt.-% of an alginate, preferably the NH<sub>4</sub> salt. The protective colloid is added to the wet pigment slurry, and the pigment dried and ground. S. S. W. Manufacture of varnishes and coatings therefrom. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 375,320, 19.3.31).—Solutions of polymerised styrene and natural and/or artificial resins compatible therewith, e.g., rosin, dammar, linseed oil-glyptal, and preferably hydrogenated resinic acid esters, e.g., Me tetrahydroabietate, together with, if desired, softeners, pigments, dyes, etc., are claimed.

S. S. W.

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Manufacture of coloured nitrocellulose lacquers and masses. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 372,525, 16.4.31).—Cellulose nitrate lacquers and masses are coloured light-fast green shades, stable



to sublimation, by the use of oxazine dyes of annexed formula, in which R = Me or  $NEt_2$  Et, X = H or Me, and Y = Hor OEt, together with a 2-nitroarylamine-4-sulphonate

or 2:6-dinitrodiarylamine-3': 4-disulphonate of a hydroaromatic base, e.g., mono- or di-cyclohexylamine. C. H.

Nitrostarch lacquer compositions. W. R. LAMS and J. A. WYLER, ASSTS. to TROJAN POWDER CO. (U.S.P. 1,833,526, 24.11.31. Appl., 30.4.28).—Lacquers comprising nitrocellulose, nitrostarch, 5% (on their combined wts.) of an homogeniser, e.g., alkali palmitates, HCO<sub>2</sub>Na, NH<sub>2</sub>Ph,HCl, *p*-aminoacetanilide, and a suitable solvent, e.g., EtOAc, are claimed. S. S. W.

[Manufacture of] compositions containing polymerised vinyl compounds and the application thereof. BRIT. CELANESE, LTD., and W. H. Moss (B.P. 372,327, 24.12.30).—As plasticisers there are used phenols or their esters or ethers, e.g.,  $\beta\beta$ -di-(p-hydroxyphenyl)propane (I), Ph or tolyl phosphate, 1:1-di-(p-hydroxyphenyl)cyclohexane. There may also be present a natural or synthetic resin, e.g., shellac, or the product from CH<sub>2</sub>O and (I) and/or volatile solvents. C. H.

Removal of colour substances from resin. I. W. HUMPHREY, Assr. to HERCULES POWDER Co. (U.S.P. 1,832,864, 24.11.31. Appl., 23.8.26).—Wood resin is repeatedly distilled at 270—330°, under reduced pressure, avoiding substantial decomp., the vapours being preferably passed through a fractionating column maintained at 260—290°. The resulting product does not discolour materially in presence of alkali and  $O_2$ (as in soap making). S. S. W.

Manufacture of hydrophobe resins by condensation of urea with aldehydes. K. RIPPER, Assr. to SYNTHETIC PLASTICS CO., INC. (U.S.P. 1,833,868, 24.11.31. Appl., 25.2.26. Austr., 3.3.25).—Urea is warmed with an aldehyde in a neutral medium, and the solution of the intermediate H<sub>2</sub>O-sol. condensation product is acidified and condensation continued with the addition of urea or thiourea. E. L.

Manufacture of condensation products of urea, thiourea, and formaldehyde. POLLOPAS, LTD., Assees. of KUNSTHARZFABR. DR. F. POLLAK G.M.B.H. (B.P. 375,609, 31.8.31. Ger., 1.9.30).—A mixture of CO(NH<sub>2</sub>)<sub>2</sub> (1 mol.) and CS(NH<sub>2</sub>)<sub>2</sub> (1 mol.), pretreated in non-acid solution with CH<sub>2</sub>O (2 mols.), is gradually British Chemical Abstracts-B.

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introduced into a hot solution of  $CO(NH_2)_2$  (1 mol.) and  $CH_2O$  (< 2 mols.) containing an acid condensing agent, until 1 mol. of combined  $CO(NH_2)_2$  and  $CS(NH_2)_2$  corresponds to  $1\cdot4$ — $1\cdot8$  mols. of  $CH_2O$ , and the condensation is continued until hydrophobic masses, pptg. on cooling, are formed. The product is separated and dried etc. by known means. S. S. W.

Making artificial [thermoplastic] resins and articles therefrom. H. ROMMLER A.-G. (B.P. 374,914, 3.3.31. Austr., 15.12.30).—1 mol. of thiourea (with or without admixture of urea) is allowed to react at room temp. with  $1\frac{1}{2}$  ( $\leq 2$ ) mols. of aq. CH<sub>2</sub>O, at  $p_{\rm H} < 5$ (preferably 3); fabric, asbestos, etc. is impregnated with the solution, to which volatile solvents, e.g., EtOH, may be added, and dried, preferably without application of heat, and pressed and united under heat, or disintegrated to form moulding and die-casting powders.

E. L.

Preparation of artificial resin compression mixtures. A. Nowack A.-G., and R. HESSEN (B.P. 375,396, 25.3.31. Ger., 27.3.30).—Intimate mixtures of finely-powdered precondensed resin, e.g., PhOH-CH<sub>2</sub>O, and fillers, together with pigments and stearine, if desired, are subjected to high pressure and temp., e.g., by forcing at 150—200 kg./sq. cm. through nozzles 10 cm. long, heated to 200°. The mixture is then rapidly cooled while releasing the pressure, the resin still remaining in the "resol" stage. S. S. W.

Manufacture of artificial resin. J. V. MEIGS, Assr. to MEIGSOID CORP. (U.S.P. 1,832,038, 17.11.31. Appl., 17.7.22).—A carbohydrate is heated with a phenol in presence of mineral acid or other suitable agent, e.g., HCl,  $H_2C_2O_4$ , citric acid,  $H_2O$  being removed from the reactants, and the initial resin product is heated with a substance, free from free mineral acid, capable of reacting therewith, e.g., NH<sub>3</sub>, natural resin.

S. S. W.

**Production of soluble resinous products.** I. ROSENBLUM (B.P. 370,916 and 370,946, 7.10.30).— (A) An oil-sol., fusible resin is obtained by heating glycerol (etc.) with a dry-distilled natural resin for partial esterification and then with an acidic phenolaldehyde or -ketone condensation product, preferably produced in presence of a metal org. salt, particularly Zn abietate or a mixture of ZnO and resin. (B) The glycerol is partly esterified with a non-resinous acid, *e.g.*, linoleic or phthalic acid, instead of resin. C. H.

Synthetic resins and coating compositions manufactured therefrom. IMPERIAL CHEM. INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & Co. (B.P. 374,876, 14.3.31).—The product obtained by incorporating (by heating at, e.g., 170°) 1—10% of litharge in a phthalic acid-fatty acid-glycerol type resin is used to produce hard-drying coatings free from aftertack. E. L.

Manufacture of coloured masses and solutions thereof. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 372,761, 31.12.30. Addn. to B.P. 293,067; B., 1928, 681).—Coloured org. sulphonic or carboxylic acids are substituted for the acid dyes of the prior patent. Examples are: mono- or di-cyclohexylamine 2-nitroaniline-4-sulphonates, 2:6-dinitrodiphenylamine-3':4disulphonates, 2:4 - dinitrodiphenylamine - 3' - carboxylates, 2:6-dinitro-4'-hydroxy-3'-carboxydiphenylamine-4:5'-disulphonates, or 5-chloro-2-nitroaniline-4sulphonate (all yellow or brown); 2:4-dinitrodiphenylamine-3-sulphonate of Rhodamine B (scarlet) or Rhoduline-blue 6G (blue-green); 2-nitroaniline-4-sulphonate of Methyl-violet B extra (violet). C. H.

Pigments.—See IV. Metals for painting. Bronze colours.—See X. Insulating composition.—See XI. Vulcanised goods.—See XIV. Ink for films.—See XXI.

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

Products from rubber seed. II. Applications and economics. T. R. DAWSON and T. H. MESSENGER (J. Res. Assoc. Brit. Rubber Manufrs., 1932, 1, 45—53). —Information on the possibilities of rubber-seed oil for such applications as the manufacture of paint and varnish, rubber substitute, soap, linoleum, etc. is reviewed. Rubber-seed cake has received extensive consideration as a cattle food. In present circumstances there is little hope of successful commercial exploitation of rubber seed. D. F. T.

Rubber latex. Recent scientific and technical developments. V. N. MORRIS and H. W. GREENUP (Ind. Eng. Chem., 1932, 24, 755—770).—After a review of the scientific knowledge of rubber latex an account is given of methods for its concentrating and compounding, and for its application to manufactures by such processes as impregnation, dipping, filtration, electrodeposition, gelling, extruding, moulding, or coagulation and milling. The uses in sp. industries are recorded and the characteristics of various types of product, *e.g.*, adhesives and cements, porous and sponge rubber, electrical insulation, etc., are indicated. D. F. T.

Elastic constants of rubber at great stresses. R. WEISE (Kautschuk, 1932, 8, 106-110).-Test-pieces of heat-vulcanised sheet of " pure rubber " quality were submitted for 2 hr. to loads varying from 100 g. to 1000 g. (approx. 0.01-0.1 kg./sq. mm.), the corresponding elongation ranging from 110.6% to 410.6% of the original length. On release and recovery for 2 hr. the residual extension for these two pieces was 0.14% and 1.76%, respectively, the corresponding vals. being 0.05% and 1.19% after 24 hr. Test-pieces which had not been stretched beyond 100% extension (at which stage Röntgen interference first occurs) recovered practically completely in 1 yr. The average val. of Poisson's ratio up to a total extension of 870% was 0.490. The elasticity modulus, which at low extensions was 0.102kg./sq. mm., attained a min. val. of 0.036 kg./sq. mm. at 70% extension and ultimately increased to approx. 1000 kg./sq. mm. A kinematographic method of observation was applied as an aid to the measurements.

D. F. T.

Compression stress-strain curve of rubber. J. R. SHEPPARD and W. J. CLAPSON (Ind. Eng. Chem., 1932, 24, 782-790).—The relation existing between tensile forces and the equiv. compressive force is shown and with its aid a new method for determining the compression stress-strain curve of rubber is indicated involving the application of two-dimensional tensile stresses, viz., by inflation of a rubber sphere or balloon; the pressure of the gas and the dimensions of the sphere. provide the data for determining the compression stressstrain curve. This appears to be continuous with the ordinary elongation curve, when both are plotted in the same units, an inflexion occurring near the origin. It is not possible to compute the compression stress-strain curve from the ordinary unidimensional elongation stress-strain curve. D. F. T.

Cause of the alteration in viscosity of rubber solutions for dipping purposes on stirring. R. Drr-MAR (Chem.-Ztg., 1932, 66, 622).—Rubber solution in a bath for manufacture by dipping, if stirred vigorously, shows a marked increase in viscosity and does not regain its earlier condition until after several hr. In the light of recent views as to the presence of very elongated mols. in rubber solution this phenomenon is attributed to a disarrangement and gradual re-orientation of the mols. D. F. T.

Factors in deterioration of rubber when exposed to frictional contacts with other materials. V. A. COSTER (India-Rubber J., 1932, 84, 231–233).

#### PATENTS.

Mechanical working, manipulating, or mixing of rubber or similar mixes. T. J. DRAKELEY, F. H. COTTON, and D. BRIDGE & Co., LTD. (B.P. 374,271, 6.6.31).—To increase the plasticity or ensure more rapid and thorough mastication of the rubber,  $O_2$  or air, either continuously or at intervals, is introduced or circulated in a more or less enclosed chamber containing the rollers or other kneading apparatus. D. F. T.

Vulcanisation of rubber. RUBBER SERVICE LABS. Co., Assees. of R. L. SIBLEY (B.P. 374,123, 4.3.31. U.S., 6.3.30).—Vulcanisation is accelerated by a mixture of a condensation product of an arylamine (preferably primary), e.g., 2:4-diaminodiphenylamine, and a mercaptoarylenethiazole, e.g., mercaptobenzthiazole, with an org. nitrogenous basic activating accelerator, e.g., diphenylguanidine. D. F. T.

Vulcanisation of rubber articles. FIRESTONE TIRE & RUBBER CO., LTD., Assees. of E. T. HANDLEY (B.P. 375,446, 31.3.31. U.S., 31.3.30).—The internal oxidation of "water bags," such as are used to inflate rubber tyres during vulcanisation, is retarded by deoxidising the contained  $H_2O$ , e.g., with  $\frac{1}{2}$  oz. of Na<sub>2</sub>SO<sub>3</sub> per gal.

#### D. F. T.

Manufacture of vulcanised products with a fibrous mineral base, such as amianthus or asbestos and the like, combined with a binding material such as latex of caoutchouc etc. A. FERRETTI (B.P. 375,462, 10.4.31. It., 11.4.30).-Mineral fibres, with others if desired, are reduced to a pulp with H2O, vulcanising agents and antioxidants, together with protective agents, such as gelatin or soap, being also added if wished. After the incorporation of a coagulant for rubber latex, e.g., Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, rubber latex is introduced and the mixture is fed at once to a sheeting machine, e.g., of the paper-making type, a no. of sheets being subsequently plied together by pressure with or without previous successive application of a rubber coagulant D. F. T. and latex.

Production of compact soft and hard caoutchouc vulcanised goods. H. ZIEGNER (B.P. 374,085, 2.3.31).—

Products having the qualities of leather or linoleum are obtained by impregnating fibrous or absorbent substances such as sawdust with a rubber-swelling agent, *e.g.*, gasoline, before incorporation, with or without paraffin wax, in rubber. After evaporation of the swelling agent, the mixture is vulcanised under pressure. D. F. T.

Antioxidants for treatment of rubber. RUBBER SERVICE LABORATORIES CO., Assees. of J. R. INGRAM (B.P. 376,752, 13.4.31. U.S., 15.4.30).—See U.S.P. 1,816,851; B., 1932, 438.

Compound papers.-See V.

#### XV.—LEATHER; GLUE.

Wattle barks. III. M. B. WELCH, F. A. COOMES, and W. MCGLYNN (J. Proc. Roy. Soc. New South Wales, 1931, 65, 207-231).—The tannin content varies as the thickness of the bark under ordinary conditions. Green wattle (*Acacia mollissima*) may have a tannin content as high as  $53 \cdot 5\%$  with a bark thickness of 10 mm. *A. pauciglandulosa*, which has a thin bark, yields a max. of  $35 \cdot 1\%$  of tannin. Silver wattle (*A. arundelliana*) also yields a useful bark with a max. tannin content of  $43 \cdot 5\%$ , as does *A. decurrens*. All figures are based on a standard moisture content of  $11 \cdot 5\%$ . P. G. M.

Absorption of oxygen by [vegetable] tanning materials. E. W. MERRY (J. Soc. Leather Trades' Chem., 1932, 16, 358—377).—The absorption of  $O_2$  by solutions of commercial chestnut extract was accelerated by the addition or accumulation of Cu or Fe salts, NaHSO<sub>3</sub>, SnCl<sub>2</sub>, large amounts of  $p-C_6H_4(OH)_2$ , and retarded by  $H_2C_2O_4$ ,  $NaHC_2O_4$ ,  $K_2C_2O_4$ ,  $H_2SO_4$ , HCl, PhSO<sub>3</sub>H,  $H_3PO_4$ , quinhydrone, and  $p-C_6H_4O_2$ . The rate of oxidation was generally reduced by a marked reduction in the  $p_{\rm H}$ , or by an increase in the amount of added acid. The effect of  $H_2C_2O_4$  was partly due to its effect on the  $p_{\rm H}$  val. and partly sp.  $H_2C_2O_4-p-C_6H_4O_2$  mixtures had a greater effect than either separately. A list of substances which had no effect on the oxidation is given. D. W. J.

(A) Improved Procter-Hirst reaction for detection of sulphite-cellulose in tannin extracts and (B) effect of humic acids in decayed wood on the test. O. GERNGROSS and H. HERFELD (Collegium, 1932, [A] 237-246, [B] 247-250).-Humic acids isolated from lignite are insol. at pH 8 (in aq. NH3). They are pptd. at  $p_{\rm H}$  5.4 from pure solutions and at lower vals. in presence of tannin, and by 2.5% and 28.4% solutions of NH, Ph, HCl. The Procter-Hirst reaction is sensitive to 0.01% of humic acids. The ppts. given by tannins with NH<sub>2</sub>Ph,HCl are sol. in excess of that reagent, whereas the ppt. with sulphite-cellulose is insol. in excess of the reagent. The test fails with extracts of decayed wood because the humic acids yield a ppt. with NH2Ph, HCl which is insol. in excess of the reagent, but the failure is obviated if 10 c.c. of the clear, filtered tan liquor are acidified with 0.2 c.c. of HCl (25%), the mixture is vigorously shaken, filtered after 10 min., 5 c.c. of the clear filtrate are treated with 0.5 c.c. of pure NH,Ph, well shaken, then 2 c.c. of conc. HCl are added and the whole is left for 15 min. for the turbidity to develop. The sensitivity of the test is reduced from 1% sulphite-cellulose to 5%. D. W.

Oxidation phenomena in the determination of moisture in tannin extracts by the indirect method. L. POLLAK and W. SPRINGER (Collegium, 1932, 255— 266. Cf. B., 1931, 266; 1932, 157).—A higher figure is obtained by the indirect method for  $H_2O$  in pyrogallol tannin extracts owing to oxidation, and a lower figure for  $H_2O$  in pyrocatechol tannin extracts, due to complete oxidation of the non-tans, particularly the sugars. Both types of oxidation may proceed simultaneously. D. W.

Intermittent contact electrode for the determination of  $p_{\rm H}$  in sulphited [tanning] extracts. W. R. INGLE (J. Soc. Leather Trades' Chem., 1932, 16, 354—355).—A palladised Pt electrode is suspended in a glass tube in a steady stream of H<sub>2</sub>, which escapes through a circular aperture in the tube 0.125 in. below the electrode. The apparatus is lowered into the liquor to be tested and the flow of H<sub>2</sub> so controlled that bubbles escape through the aperture every 4 sec. and the liquor rises to make contact at the point only of the electrode. After 5 min. bubbling, circuit is made with the galvanometer. D. W.

Preliminary hydration of hides in relation to the chemico-physical qualities of heavy leathers. G. BAGGINI and B. AVENATI-BASSI (Boll. Uff. Staz. sperim. Ind. Pelli, 1932, 10, 162-172).-Hydration of a hide during the preliminary washing, and, especially, the alkaline swelling due to liming, tends to give a swollen product saturated with tanning material and hence heavier and less durable. Hides tanned in their naturally hydrated state fix less tannin and give a lighter and more elastic leather of dense fibre and more durable, but the tanning is more lengthy and is difficult with present methods of working. The best results are obtained by perfecting the system of liming so as to achieve complete and rapid depilation with a min. of swelling and hence of hydration of the collagen. These conclusions are supported by photomicrographs. T. H. P.

Raw hides and skins: salting problems. Report of the Pan-European Commission No. 5. M. BERG-MANN (Collegium, 1932, 285).—Previous reports (cf. B., 1932, 393) have referred to the salting of skins and particularly of calfskins. D. W.

Mode of action [on pelt] of pickles of different compositions. III. Effect of neutral salts on the swelling and hydrolysis of hide substance by alkaline solutions. A. KÜNTZEL and J. PHILIPS (Collegium, 1932, 267-284; cf. B., 1931, 358).-The swelling of collagen in NaOH is not comparable with that in HCl or HCl-NaCl. The swelling of collagen by alkali is only slightly repressed by neutral salts. The swelling of collagen in  $Ca(OH)_2$  is increased by the addition of NaCl to the  $Ca(OH)_2$  liquor owing to the formation of NaOH. The system NaOH<sub>2</sub>-Na<sub>2</sub>SO<sub>4</sub> resembles a pickle. The hydrolytic effect of NaOH on collagen is only slightly repressed by additions of NaCl to the NaOH. The effects of NaOH and Ca(OH)<sub>2</sub> on collagen are fundamentally The tensile strength of collagen fibres is different. increased by Ca(OH)2-swelling, but diminished by NaOH-swelling. The fibres are shortened by treatment with NaOH, but not with Ca(OH)2. D. W.

Darkening of vegetable-tanned leathers on exposure to light. M. C. LAMB and J. A. GILMAN (J. Soc.

Leather Trades' Chem., 1932, 16, 355–358).—Vegetable-tanned leathers tanned with pyrocatechol tanning materials and treated with buffers were darkened and reddened on exposure to light to a greater extent as the  $p_{\rm H}$  was lowered from 9.0 to 1.8. To avoid discoloration, shoe-lining leathers should be brought as near neutral point as possible. Darkening of pyrocatecholtanned leathers was not prevented by pretreatment with Na<sub>2</sub>SO<sub>2</sub> or NaHSO<sub>3</sub>. D. W.

Mechanical tests on leather. II. L. JABLONSKI and F. WAIGAND (Collegium, 1932, 416—431).—The modulus of elasticity has been determined for a no. of leathers of different tannages and compared with their analytical vals. All the low vals. were associated with lowquality leathers and the max. vals. with pure pit-tanned leathers. The vals. were affected by the mode of finishing of the leathers. The val. obtained by bending the grain was invariably greater than that obtained by bending the flesh side. The stretch val. was greater than the bending val. for small moduli of elasticity, but the reverse held for high moduli. D. W.

Löhlein-Volhard method of evaluating bates. A. KÜNTZEL (Collegium, 1932, 404-416).-A sample of the bate (e.g., 0.2 g. of Oropon R) is shaken with 10 c.c. of H<sub>2</sub>O for 15 min. at 37°, 20 c.c. of a 5% casein solution (1 c.c. N-NaOH per g. casein) are added, and after 1 hr. at 37° the reaction is stopped by adding 10 c.c. of 0.2N-HCl, the mixture well shaken, 10 c.c. of 10% Na<sub>2</sub>SO<sub>4</sub> solution are added, and 5 c.c. of the filtered liquid are titrated with 0.1N-NaOH (naphtholphthalein). A blank should be made. It is impossible to hydrolyse the whole of the casein however much bate is used and <60-70% should be hydrolysed to ensure reasonable accuracy. Tables are provided showing the % hydrolysed casein which corresponds to the particular final titration figure. In this way a sample bate can be compared with a standard product. The prescribed temp. must be closely adhered to. The activity of the bate is stimulated by NH4 salts and errors are caused by the doubtful end-point of the titration. D. W.

Fat-liquoring of chrome leather. II. Effect of various oils on oil adsorption and strength of leather. E. R. THEIS and F. HUNT (Ind. Eng. Chem., 1932, 24, 799-802; cf. B., 1931, 408).—Oil-adsorption curves were derived for various mixtures of raw and sulphonated neatsfoot, cod, and castor oils, moellon, and egg yolk. As the % of sulphonated oil in the mixture was increased, the amount of oil adsorbed by the leather increased, and the strength of the fatliquored leather was increased at first and then diminished; it was less with 100% sulphonated oil than with 100% raw oil although more oil had been adsorbed. Max. tensile strength was given to leather fat-liquored at  $p_{\rm H}$  4 with 25% moellon in sulphonated cod oil-moellon mixtures and the amount of adsorbed oil increased as the % moellon was increased. At  $p_{\rm H}$  9, max. oil adsorption and strength of the leather were given with 50% moellon. More oil was adsorbed from acid than from alkaline fat-liquors and the strength of the leathers fat-liquored with cod oil-sulphonated castor oil was greater from acid fat-liquors containing

more cod oil, whilst the reverse held for alkaline fatliquors. The adsorption of the oil was retarded by additions of salted egg yolk to fat-liquors; the tensile strength of the leather was unaffected at  $p_{\rm H}$  5, but was increased by such additions at  $p_{\rm H}$  9, attaining a max. with 50% of egg yolk. A max. of oil was adsorbed at 82·2° but max. tensile strength was imparted by fat-liquoring at 26·7°. The amount of oil adsorbed by the leather was increased in the range  $p_{\rm H}$  3—5 by the addition of emulsifiers to the fat-liquor. With moellon, cod oil, and emulsifiers, the adsorbed oil diminished as the H<sub>2</sub>O content of the leather increased, but it increased from sulphonated oil fat-liquors and was unaffected in egg yolk fat-liquors. D. W.

Behaviour of sulphonated castor oils in chrome leather fat-liquoring. F. STATHER and R. LAUFFMANN (Collegium, 1932, 391-404).-A portion of the sulphonated castor oil used in fat-liquoring chrome leather is so combined with the leather that it cannot be extracted therefrom with H<sub>2</sub>O, EtOH, or light petroleum. The total fat adsorbed by chromed hide powder is independent of the degree of sulphonation of the castor oil used for fat-liquoring, but the amount of "combined fat" increases as the degree of sulphonation of the oil is increased (cf. Schindler and Klanfer, B., 1931, 1063). Larger amounts of soap-like constituents are present in highly sulphonated oils and consequently soap-like Cr compounds will be more probably formed in larger quantities with such oils. The total fat adsorbed by chromed hide powder, the amount of combined fat and of extractable fat are all directly proportional to the amount of oil used in fat-liquoring. The combined fat is increased and the extractable fat decreased by increase in the Cr content of the leather, but the fat adsorbed is unaffected. These factors are not affected by the basicity of the Cr liquor employed in chroming, or by the  $p_{\rm H}$  of the fat-liquor. D. W.

Casein.—Sec. V. Corrosion by tan liquors.— See XV.

#### PATENTS.

Liberation of hair from hides or skins. H. A. BARE, M. J. KERREMANS, and H. M. J. A. DE COSTER (B.P. 374,112, 4.3.31. Belg., 4.3.30).—The hides or skins are immersed for 2 hr. at 70° in a 6%  $H_2SO_4$ solution containing a hair-protecting agent, *e.g.*, 0.005% AgNO<sub>3</sub>. D. W.

Treatment of animal fibres [skins]. A. MacKay, Assr. to AMER. SALPA CORP. (U.S.P. 1,833,029, 24.11.31. Appl., 5.7.29).—Raw skin or tanned skin which has been detannised is disintegrated and mixed with rubber latex and/or lignin from cellulose waste, and the H<sub>2</sub>O is removed, leaving the fibres in a felted condition. D. W.

Manufacture of decomposition products from leather. J. MICHELMAN (U.S.P. 1,830,921, 10.11.31. Appl., 3.4.24).—Leather scrap is distilled at 100° rising to >400° out of contact with air, the volatile distillate condensed and allowed to settle, the oily and tarry constituents are treated to isolate pyrrole, pyrocoll, etc., and the liquid portion of the distillate is treated to isolate OH- and  $CO_2H$ -derivatives of  $C_6H_6$  etc. D. W. Azo dyes.—See IV. Coated fabric.—See VI. Vulcanised goods.—See XIV.

# XVI.—AGRICULTURE.

Presence of mineral sulphides in an arable soil. G. BERTRAND and L. SILBERSTEIN (Compt. rend., 1932, 194, 2097—2101).—An infertile soil has been found to liberate small quantities of  $H_2S$  when treated with HCl. W. O. K.

Determination of the total surface area of soils, clays, and similar materials. V. Hysteresis in water adsorption. H. KURON (Z. Pflanz, Düng., 1932, 25A, 179–187).—Relationships between the process of H<sub>2</sub>O adsorption and the nature of the soil profile and depth of the horizons are examined. Profiles of different types show definitely different vals., but there is little change with varying depth in the same profile. The hysteresis phenomena recorded are dependent on the sensitiveness of the soil colloids to drying out, the action of enclosed air in reducing adsorption rates, and on the swelling of the colloids. A. G. P.

Comparison of methods for determining adsorbed bases [in soils]. I. N. ANTIPOV-KARATAIEV, A. S. KUZINA, and L. A. FROLOVA (Z. Pflanz. Düng., 1932, 25A, 187—185).—Among leaching methods the use of N-NH<sub>4</sub>Cl or -NH<sub>4</sub>OAc ( $p_{\rm H}$  7.0) for determining adsorbed Ca and Mg gives consistent and satisfactory results. Methods involving extraction with 0.05N- or 0.02N-HCl yield vals. for Ca which agree with the above, but Mg vals. show considerable divergence (up to 100%). In soils containing carbonates Hissink's modified method gives vals. for Ca similar to those obtained by the HCl methods, but is less satisfactory for Mg. A. G. P.

Relationships between the degree of saturation and buffering of a soil and its reaction value. I. Reaction and degree of saturation. H. NIKLAS, A. HOCK, and A. FREY (Z. Pflanz. Düng., 1932, 25A, 203-228).-In strongly acid soils there is a general parallelism between the  $p_{\rm H}$  (H<sub>2</sub>O),  $p_{\rm H}$  (KCl), the exchange and hydrolytic acidities; and the degree of saturation (Kappen). In feebly acid and neutral soils divergencies between these vals. are more prominent. The S val. of a soil may be determined by means of the abs. neutralisation capacity. The T val. may be calc. by multiplying the hydrolytic acidity by 6.5 for strongly acid soils or 3.0 for slightly acid and neutral soils. The degree of saturation indicated by Kappen's method is more closely related to the  $p_{\rm H}$  than that obtained by Gehring's method. The former method is more suitable for CaO-requirement measurements. A. G. P.

Electrical neutralisation of soils by methyleneblue and the adsorption capacity. A. RABINERSON (Z. Pflanz. Düng., 1932, 25A, 228–235; cf. Mattson, A., 1928, 1323).—In a no. of soil types, the amounts of methylene-blue necessary to neutralise the soil colloids (electro-osmosis measurements) have no relationship to their adsorption capacity. A. G. P.

Course of growth and nutrient intake of sugar beet in the first and second years of growth. II. H. WAGNER (Z. Pflanz. Düng., 1932, 25A, 129-155; cf. B., 1932, 696).—In neither the first nor second

growth years was there a decline in the dry wt. of leaves after the max. was reached. Max. leaf wt. was attained more quickly in the second than in the first year as a result of the utilisation of the reserve material of the root. In the first year there was a decrease in the % N in leaves with advancing growth (in cereals N, P, and K decrease), but during the period of rapid sugar accumulation in the root a slight decline in the % K and a notable decrease in the % Ca were observed. The % P205 remained practically const. throughout. In the second year the % N, P2O5, K, and Ca decreased steadily with growth. The general level of the P2O5 content was definitely higher in the second year than in the first. In the first year the total N and  $P_2O_5$  in the leaves rose to a max. and remained nearly const. with continued growth. After attaining a max. the total K declined slightly and the total Ca considerably. These later changes coincided with a further accumulation of sugar and mineral matter in the roots. In the second growth year the nutrient exchange between leaves, flowers, and seeds resembled that of annual plants. The intake of N, P, and K by leaves was paralleled, during the first year, by the increase in dry matter, but in the second year was in advance of the dry wt. increases, especially after the beginning of flowering. The intake of minerals by the roots was approx. parallel with drymatter production, although that of N was slightly and that of Ca considerably retarded. With increasing sugar accumulation the rate of intake of Ca rose. The total nutrient intake curve of whole plants up to the period of rapid sugar accumulation showed several points of inflexion corresponding with definite growth A. G. P. changes.

Intake of phosphoric acid by plants in water cultures. R. KREYZI (Z. Pflanz. Düng., 1932, 25A, 156-179).-The P intake of oat plants under uniform growth conditions is proportional to the wt. of the roots, and the intake per unit wt. of root is a measure of the resorptive power of the plants. The resorptive power of oats is much greater for P-deficient plants than for those receiving complete nutrients. From the latter plants PO<sub>4</sub> may be re-eliminated after transference into very dil.  $PO_4$  solutions. The % utilisation of the  $PO_4$  present in the nutrient declines with rising concn. of PO4 and with decreasing root wts. When CaCl2 is added to the nutrient complete utilisation of the PO4 may occur. With decreasing PO<sub>4</sub> concn. in the nutrient the resorptive power for  $PO_4$  declines and subsequently increases. The resorptive power of oat roots declines with age. The rate of intake of PO4 has no definite relationship to the nature of other ions present, although it is increased by adding small amounts of CaCl<sub>2</sub>.

#### A. G. P.

Percolation experiments with superphosphate and basic slag. C. KRÜGEL, C. DREYSPRING, and F. HEINRICH (Z. Pflanz. Düng., 1932, 25A, 195-203).— Leaching out of  $PO_4$  from soils treated with superphosphate or basic slag is insignificant even on light sands, unless quantities far above customary usage are applied. In soil the P of superphosphate must exist in a mobile condition capable of replenishing the soil solution at a much greater rate than the citric-sol.  $PO_4$ of basic slag. A. G. P. "Calcium fluosilicate compound " is not calcium fluosilicate. R. H. CARTER (J. Econ. Entom., 1932, 25, 707–709).—Material commonly reported as  $CaSiF_6$  in insecticidal investigations is a complex by-product of the fertiliser industry, and contains Ca phosphates,  $Fe_2O_3$ ,  $Al_2O_3$ , and approx. 20% of  $CaSiF_6.2H_2O$ . A. G. P.

Relative toxicity of some fluorine and arsenical insecticides. H. F. SMYTH and H. F. SMYTH, JUN. (Ind. Eng. Chem., 1932, 24, 229—232).—The relative chronic toxicities of F and As administered orally to rats over 16 weeks have been found to be PbHAsO<sub>4</sub> 100%, Na<sub>3</sub>AlF<sub>6</sub> 10%, and BaSiF<sub>6</sub> 8%. A daily dosage of 23 · 1 mg. of F (as Na<sub>3</sub>AlF<sub>6</sub>) or 20 · 6 mg. of F (as BaSiF<sub>6</sub>) per kg. body-wt. was the lowest definitely toxic dose, an equal degree of toxicity being obtained with  $1 \cdot 12$  mg. of As<sub>2</sub>O<sub>3</sub> (as PbHAsO<sub>4</sub>). Hence the use of F insecticides for fruit would give a wider margin of safety than do As materials, between the wt. of spray residue and the amount toxic to the consumer. H. J. D.

Tar-distillate sprays. F. Z. HARTZELL, P. J. PARROT, and L. R. STREETER (J. Econ. Entom., 1932, 25, 607—613).—The efficiency of tar-distillate sprays against a no. of insects is recorded. Some injury to swelling apple buds, even beforethe "si lver tip" stage, resulted from washes containing > 8% of tar oils, but with 4% no injury occurred. Combinations of tar and lubricating oils were very injurious to apple buds. A. G. P.

Efficiency of tar-distillate sprays in controlling San José and scurfy scales. W. S. HOUGH (J. Econ. Entom., 1932, 25, 613—617).—Only in concns. of 1 in 10—15 were tar-oil washes comparable in efficiency with 3% petroleum oil emulsions. Differences in the action of various tar-distillate oils of similar boiling range are recorded. A. G. P.

Chemical and physical properties of petroleum spray oils. J. R. GREEN (J. Agric. Res., 1932, 44, 773—787).—Plant injury by petroleum oils is associated with high sulphonatable fraction, S content, Br absorption, acidity, and ease of emulsification with  $H_2O$ . Oils having < 6% sulphonatable matter would not of themselves form a film on a  $H_2O$  surface and were the least injurious. Differences in surface tension of the oils examined were small. No relationships exist between the viscosity, flash point, fire point, or d of oils and their injurious properties. Sulphonation of oils does not entirely remove their toxic properties. A. G. P.

New sprays effective in the control of the locust borer. R. A. ST. GEORGE and J. A. BEAL (J. Econ. Entom., 1932, 25, 713—721).—Spring applications of paraffin emulsions containing Na<sub>3</sub>AsO<sub>3</sub>, pine-tar oil, o- or p-C<sub>6</sub>H<sub>4</sub>Cl<sub>2</sub> were effective. Later applications penetrated the wood. Mixtures of paraffin and o-C<sub>6</sub>H<sub>4</sub>Cl<sub>2</sub> killed the trees. A. G. P.

Waxes as possible carriers of insecticides. J. M. GINSBURG (J. Econ. Entom., 1932, 25, 599-607).— Spermaceti, candelilla, and paraffin waxes, emulsified with triethanolamine oleate, caused no injury to apple and pear foliage in concess. up to 4% and at 1% were appreciably toxic as contact insecticides but noneffective as stomach poisons. Emulsified waxes (1%) impregnated with pyrethrum or derris extracts showed ing inc high toxicity to both biting and sucking insects. The m

A. G. P.

Muckland potato spraying experiments. D. O. WOLFENBARGER (J. Econ. Entom., 1932, 25, 647—649). —Best results followed the use of Bordeaux mixture combined with Pb arsenates. A. G. P.

Reactions of the Japanese beetle to spray deposits on foliage. E. R. VAN LEEUWEEN (U.S. Dept. Agric. Circ., 1932, No. 227, 18 pp.).—Pb and Cu arsenates, Ca(OH)<sub>2</sub>, BaSO<sub>4</sub>, CaCO<sub>3</sub>, and china clay in spray deposits are repellent to the beetle. A. G. P.

Control of cabbage maggot (Phorbia brassicæ Bouché) on radishes. R. B. FRIEND (J. Econ. Entom., 1932, 25, 709—712).—Aq.  $HgCl_2$  can be satisfactorily replaced by dusts composed of gypsum and  $Hg_2Cl_2$ (96:4). The quantity of Hg adhering to washed roots is negligible. A. G. P.

Onion thrips. F. B. MAUGHAN (J. Econ. Entom., 1932, 25, 662—670).—Nicotine proved the most effective but good control was produced by kerosene (some foliage injury), pyrethrum,  $C_{10}H_8$ , and Cu preps. A. G. P.

Protection of celery from tarnished plant bug injury. L. L. HILL (J. Econ. Entom., 1932, 25, 671— 678).—Applications of finely-ground S,  $Ca(OH)_2$  dust, or S sprays prevented both feeding and oviposition. A. G. P.

Striped and spotted cucumber beetles. F. G. BUTCHER (J. Econ. Entom., 1932, 25, 658-662).—In trials with Cu preps., with and without addition of Ca arsenate, dusts proved more effective than wet sprays. Crop increases were greater after use of the combined spray than with CuSO<sub>4</sub>-CaO mixtures alone. A. G. P.

Arsenicals on beans for control of Mexican bean beetle. H. C. HUCKETT (J. Econ. Entom., 1932, 25, 620-625).—Best results were obtained with Mg arsenate, although basic Pb arsenate was nearly as satisfactory as regards freedom from plant injury. Ca arsenate and Zn arsenite were inferior except in conjunction with Bordeaux mixture, lime-wash, Cu-CaO dust, or Ca(OH)<sub>2</sub> dust. These materials reduced foliage injury by the arsenicals. A. G. P.

Control of the apple curculio in the Champlain Valley. O. H. HAMMER (J. Econ. Entom., 1932, 25, 569—575).—Standard Pb arsenate–CaO–S sprays were ineffective. Successful results were obtained by the use of Pb arsenate 6 lb., CaO–S  $2\frac{1}{2}$  gals., Ca(OH)<sub>2</sub> 25 lb., fish oil 1 quart, H<sub>2</sub>O to 100 gals. Fish oil produced slight foliage injury in some cases. A. G. P.

Control of *Empoasca fabæ*, Harris, on young apple trees. T. W. REED (J. Econ. Entom., 1932, 25, 587—591).—Bordeaux mixture (2:2:50) was effective. Higher concns. were not more toxic and the presence of of added  $Ca(OH)_2$  did not improve the action of the spray. Nicotine sulphate-soap preps. gave good control of nymphs. A. G. P.

Chloropicrin for nematode control. M. O. JOHNSON and G. H. GODFREY (Ind. Eng. Chem., 1932, 24, 311—313).—Applications of CCl<sub>3</sub>·NO<sub>2</sub> to soils infested with root-knot (*Heterodera radicicola*) have resulted in considerable reduction in the infestation and corresponding increase in the vigour and yield of pineapple plants. The most effective treatment consisted of applications in liquid form at the rate of 163 lb. per acre, afterwards covering with mulch paper. Heavier applications (204 lb./acre) gave inferior control. The increased yields appear to justify the extensive use of  $CCl_3 \cdot NO_2$  on badly infested land. H. J. D.

Relation of hydrocyanic acid concentration to the kill of the black fly (*Aleurocanthus woglumi*, Ashby). A. F. CAMP and R. J. WILMOT (J. Econ. Entom., 1932, 25, 476-483).—At 29-30°, exposure for 10 min. to an atm. containing 0.17-0.19% HCN killed all larvæ and pupæ. For adults 0.03% HCN sufficed for a complete kill. A. G. P.

Relation of hydrocyanic acid concentration to the kill of various stages of the green scale (Coccus viridis, Green). A. F. CAMP and R. J. WILMOT (J. Econ. Entom., 1932, 25, 483–486).—Complete control was obtained by exposure for 1 hr. to 0.04-0.05%HCN or for 5 min. to 0.19% HCN. A. G. P.

Reaction of sulphuric acid and sodium cyanide [in fumigation]. W. MOORE (J. Econ. Entom., 1932, 25, 729–730).—Erratic results recorded in fumigation trials of HCN from NaCN are ascribed to the use of too conc.  $H_2SO_4$  and the consequent production of various mixtures of CO and HCN. A. G. P.

Tests against the over-wintering stage of the codling moth. E. N. CORY and P. O. SANDERS (J. Econ. Enton., 1932, 25, 566-569).—Tar washes and pine-tar oils were effective against the hibernacula of the codling moth. Addition of  $p-C_6H_4Cl_2$  did not increase the toxicity of emulsified or "sol." petroleum oils but definitely improved the efficiency of pine and tar oils. A. G. P.

Comparison between nicotine tannate and lead arsenate as codling moth poisons. W. MOORE (J. Econ. Entom., 1932, 25, 554—559).—Nicotine tannate is more toxic than Pb arsenate as a stomach poison to codling moth larvæ and also acts as a contact poison to 3—4-day old eggs. Tannic acid (from Chinese galls only) is dissolved in H<sub>2</sub>O and free nicotine stirred in. Proportions recommended are tannic acid: nicotine: H<sub>2</sub>O, 4:1:1600 for larvæ and 4:1:2000 for eggs. Sprayed apple foliage retained its toxicity for at least 21 days. A. G. P.

Insecticidal efficiency of the pyrethrins, nicotine, and rotenone against the greenhouse red spider. H. H. RICHARDSON (J. Econ. Entom., 1932, 25, 592-599).-Without the use of wetting agents, these materials had little toxic action even at high concns. Incorporated with K oleate (0.25%), rotenone was slightly more toxic than were pyrethrins and nicotine was much less effective. Using sulphonated castor oil, pyrethrin and rotenone were about equally effective and nicotine was much inferior. The two wetting agents themselves had approx. the same toxicity. As a wetting agent soap was more effective with rotenone and pyrethrin, and sulphonated castor oil gave better results with nicotine. Nicotine (1 in 500) rendered sulphonated castor oil slightly alkaline but had little effect on the reaction of the soap solution. EtOH solutions of pyrethrin had an

acidic action on both sulphonated castor oil and soap.  $\text{COMe}_2$  extracts of rotenone did not appreciably affect the  $p_{\text{H}}$  of solutions of either wetting agent. A. G. P.

#### PATENT.

[Apparatus for] treatment of flowering bulbs and other similar plants to advance the flowering period. V. and H. J. DE WEYER, J. M. and T. H. VAN BUGGENUM (B.P. 375,184, 4.11.31).

# XVII.—SUGARS; STARCHES; GUMS.

High-density filtration [of sugar liquor]. J. P. GREVEN (Ind. Eng. Chem., 1932, 24, 833).—Raw sugar liquor of 66—67° Brix can be filtered safely and advantageously using only high-speed filter aid.

D. R. D.

Determination of reducing sugars present in small quantity, particularly in crude and refined sucrose. A. MOLHANT (Bull. Soc. chim. Belg., 1932, 41, 228–233).—Bertrand's method for determining reducing sugars is modified so as not to be vitiated by degradation of sucrose (present in large quantity) by the too strongly alkaline Cu reagent. R. S. C.

Phosphoric acid in potato starch. J. JANICKI (Rocz. Chem., 1932, 12, 381-402).—The viscosity n of starch solutions prepared from large granules is smaller than is that from small ones, which have also a higher P content. A certain proportionality exists between  $\eta$  and P content of different fractions of starch prepared from the same, but not from different, varieties of potato. The amylopectin content, according to Samec's method, of different potato starches varies from 84 to 89% whilst the dialysable and non-dialysable P contents of these starches, and the viscosity of their solutions, vary within much wider limits. The val. of  $\eta$  falls steadily with increase in time of storage of the starch before dissolution, without a corresponding change in the total and dialysable P contents, or in the amylopectin content. The non-dialysable P of starch does not correspond with the amylophosphoric acid content, nor is the smaller  $\eta$  of solutions of aged starch due to hydrolysis of this ester. The ageing of starch is associated with diminution in the capacity of its colloids to imbibe H<sub>2</sub>O. The view that starch is a mixture of amylopectin and amylose is not supported by the above results. R. T.

Honey.—See XIX.

PATENTS.

[Paper sacks for] packing invert sugar and like substances for transport. MANBRÉ & GARTON, LTD., and E. A. BERRY (B.P. 377,330, 8.5.31).

Sugar-cane sprouts.—See V.

# XVIII.—FERMENTATION INDUSTRIES.

Butyl-acetone fermentation of corn meal. Interrelations of substrate and products. W. H. PETER-SON and E. B. FRED (Ind. Eng. Chem., 1932, 24, 237— 242).—The bacteriological and chemical changes occurring during the fermentation of maize mash by *Clostridium acetobutylicum* (Weiz.) have been determined at frequent intervals. The vegetative cells were found to be the active agents and during the period of most rapid change (18—30 hr.) 1 g. of bacteria metabolised 0.7 g. of starch per hr. Considerable proteolysis occurs concurrently with the fermentation and at the end of 75 hr. about 60% of the total N (0.8 g./litre) was in the sol. condition. AcOH and PrCO<sub>2</sub>H form the bulk of the volatile acids and it is probable that the period of greatest production is during 18-30 hr. from the start. In this period there is a sharp fall in the acid curve, but a sharp rise in the solvent-production curve. The average figures for the solvent are COMe, 31, EtOH 9, and BuOH 60%, but the proportions appear to vary according to the  $p_{\rm H}$ , high acidity favouring high COMe2 and low BuOH production and vice versa. The mol. proportions of EtOH, COMe<sub>2</sub>, and BuOH are approx. 1, 2, and 3, respectively. Up to the 18th hr. H<sub>2</sub> production is slightly in excess of the CO<sub>2</sub>, but thereafter a sharp rise in the latter occurs. The total gas consisted of 60% CO<sub>2</sub> and 40% H<sub>2</sub>, the vol. being about 20 times that of the mash. A theoretical mechanism of the fermentation is traced from the data. H. J. D.

"Sinker" test for malts. K. SILBEREISEN (Woch. Brau., 1932, 49, 193—194).—Comparison of the results of "sinker" tests of 42 samples with other physical and analytical data shows that it is not a trustworthy index of modification. F. E. D.

Determination of the source of wort or vinasse by chemical analysis. M. V. IONESCU and L. GAAL (Bull. Soc. chim., 1932, [iv], 51, 616–618).—The % of  $P_2O_5$  and total sol. alkalis in the incinerated extract from the completely fermented vinasse or wort may be used to distinguish the following groups : (a) cereals, (b) fruit, beet, and potatoes, and (c) beet molasses.

A. A. L.

Analysis of [brewery] cooler deposit. L. HEINTZ (Woch. Brau., 1932, 49, 177–180, 187–191, 195–198). —The extraction methods previously applied to hops (B., 1931, 941) and barley husks (B., 1932, 42) are applied to cooler deposit. Et<sub>2</sub>O extracts 9–11% and subsequent treatment with EtOH a further 12–14%. The separated fractions resemble the corresponding products from hops and barley husks. No true tannin could be detected, and it is considered that such is not present in hops or malt. F. E. D.

Effect of the distillation method on the composition and quality of wine distillates. H. WÜSTENFELD and C. LUCKOW (Z. Unters. Lebensm., 1932, 63, 542— 555).—The actual quality of the distillate depends to a large extent on the method of distillation but the analytical results are less affected. The first fractions give the best flavoured portions but the latter, though increasing the yield, are less fine. Data obtained in numerous small-scale distillations are given. E. B. H.

[Determination of] potassium and tartrate in wines. J. DUBAQUIÉ (Ann. Falsif., 1932, 25, 280–285). —K is pptd. as a mixed K and  $NH_4$  H tartrate, which is titrated with alkali (litmus); the  $NH_3$  is then driven off, and the solution retitrated. Tartrate is pptd. as K H tartrate by means of KOAc and KClO<sub>3</sub> and titrated with alkali (phenolphthalein). T. McL.

#### PATENT.

Dealcoholising beer and the like. C. B. SCHNEIBLE, Assr. to C. B. SCHNEIBLE, I. S. CLARK, and B. B. SCHNEIDER (U.S.P. 1,832,587, 17.11.31. Appl., 30.7.26).— Beer which has been decarbonated in a chamber fitted with baffles by heating at approx.  $50^{\circ}$  in a vac. is distilled to remove EtOH. The CO<sub>2</sub> and traces of EtOH from the decarbonating operation are injected under pressure into the dealcoholised beer which has been cooled by being passed in heat-exchange relationship with fresh beer, whereby the latter is heated to decarbonation temp. C. R.

# XIX.—FOODS.

Determination of rye flour in milling products and bakery goods. R. STROHECKER (Z. Unters. Lebensm., 1932, 63, 514–522).—Tillmans' method (B., 1929, 70) for extraction of trifructosan (I) has been adapted for use as a gravimetric method with an accuracy of 5–10% for flour and 10% for bread. 60% milled rye flour contains  $2 \cdot 4\%$  of (I) and 60% wheat flour  $0 \cdot 32\%$ . About 30% is destroyed in baking. The content of (I) increases in rye flour with the milling grade but changes little in wheat. E. B. H.

Tenderness of meat. I. Determination of relative tenderness of chilled and quick-frozen beef. D. K. TRESSLER, C. BIRDSEYE, and W. T. MURRAY (Ind. Eng. Chem., 1932, 24, 242—245).—Two instruments have been evolved for measuring the tenderness of meat: one a cutting gauge recording the force required to puncture with a blunt point a piece of meat of a certain thickness, and the other a penetrometer recording the distance penetrated by a blunt needle under a certain pressure in a definite time. The relative tenderness of various cuts of steak have been determined before and after quick-freezing. Quick-freezing and storage at  $-28\cdot9^{\circ}$  for a week increased the tenderness by about 20%. A preliminary test indicated that there is a progressive increase in tenderness during storage.

#### H. J. D.

Detection of commencement of decomposition in flesh. G. BROTZU (Z. Unters. Lebensm., 1932, 63, 503—514).—The methods suggested are rapid and of easy technique : (1) determination of  $NH_2$ -acids by the method of Henriques and Gjaldbäk, of Grünhut, or of Van Slyke (limit 500—600 mg.  $NH_2$ -acid-N per 100 g. meat); (2)  $p_{\rm H}$  determination of juice of meat: at 6·3, or above, meat commenced to decompose; (3) bacteriological method : the meat is decomposing if there are large nos. of bacteria in 0·1 c.c. of juice expressed from minced meat. E. B. H.

Chemical composition of certain kinds of sausage and other meat food products. R. HOAGLAND (U.S. Dept. Agric. Circ., 1932, No. 230. 9 pp.).—Records of 20 meat products are given. A. C. P.

Comparative study of juices from frozen fruits. T. A. PICKETT (Ind. Eng. Chem., 1932, 24, 353—354).— The juices expressed from blackberries, cherries, dewberries, peaches, pears, plums, raspberries, and strawberries, (a) slow-frozen and stored at  $-12 \cdot 2^{\circ}$ , (b) quickfrozen at  $-73 \cdot 3^{\circ}$  and stored at  $-12 \cdot 2^{\circ}$ , were examined at intervals up to 3 months. In each case, the acidity of fruit frozen and tested immediately was > that of fresh fruit and it increased with the length of the storage period. In the (a) samples the acidity was > in the (b) and hence the fruits frozen at the lower temp. and stored at  $-12 \cdot 2^{\circ}$  resembled more closely the fresh fruit than did those frozen at  $-12 \cdot 2^{\circ}$ . The increase in acidity was due to the concn. of the same type of acid as was present in the fresh juice. H. J. D.

Vitamin-C content of orange-crush beverage. E. M. and F. C. KOCH (Ind. Eng. Chem., 1932, 24, 351-352).—The syrup, made from mechanically expressed orange juice by the addition of sugar, citric acid, NaOBz, and colouring matter, and refrigerated until bottled, has been found to retain almost undiminished for 3 months its antiscorbutic potency, which is originally equal to that of freshly expressed juice. Unstrained orange juice, however, either frozen or refrigerated, loses its potency almost completely in 3 months. The final beverage made by the dilution of the syrup with 12 vols. of carbonated  $H_2O$  gradually loses its vitamin-C potency, but a sample diluted with min. exposure to air was stored in a refrigerator for 10 days without loss of activity. H. J. D.

Volumetric determination of formic acid in fruit juices and syrups. G. VON SZELÉNVI (Z. Unters. Lebensm., 1932, 63, 534—541).—The best conditions for Fincke's HgCl<sub>2</sub> method (B., 1911, 235) have been worked out, as the results vary with the concn. of reagent etc.; the HgCl<sub>2</sub> concn. in the mixture should be  $\lt 0.01\%$ . More rapid and as accurate results are obtained by oxidation of a distillate from the sample with CH<sub>2</sub>Br·CO<sub>2</sub>H, followed by iodometric titration of excess Br, or with KMnO<sub>4</sub> in Na<sub>2</sub>CO<sub>3</sub> solution with addition of ZnSO<sub>4</sub> followed by titration of excess of KMnO<sub>4</sub> with As<sub>2</sub>O<sub>3</sub>. Raspberry juice contains small amounts of HCO<sub>2</sub>H naturally. E. B. H.

Honey. K. BRAUNSDORF (Z. Unters. Lebensm., 1932, 63, 526-534).—Honey stored 10 months at 20° gave rather more intensely red resorcinol tests than originally. Honey heated at 81° lost much diastatic activity, although samples differed considerably, and little effect was produced on the resorcinol and phloroglucinol tests unless the temp. was higher than this. The resorcinol test is the more sensitive (cf. B., 1931, 740). E. B. H.

Comb honey. II. K. BRAUNSDORF (Z. Unters. Lebensm., 1932, 63, 522—526; cf. B., 1932, 46).— "Worker" cells have a diam. of 20—22 mm. for 4 adjacent cells; "drone" cells are 20 mm. for 3 cells. The bottoms of the cells in natural combs have a 3-sided pyramid shape but frequently the bases of the cells in commercial comb honey with artificial cell walls are depressed hemispheres. E. B. H.

Changes in nectar concentration produced by the honey bee. I. Changes occurring between the flower and the hive. O. W. PARK (Iowa Agric. Exp. Sta. Res. Bull., 1932, No. 151, 210—244).—The sugar concn. of nectar is better examined in samples from the bee than from the flower. For ordinary purposes refractometric measurements are adequate. A slight decrease in sugar concn. (1%) occurs during transport from flower to hive. A. G. P.

Relation of temperature to the deterioration of honey in storage. H. F. WILSON and G. E. MARVIN (J. Econ. Entom., 1932, 25, 525-528).—Below 13°

honey may be stored without serious risk of fermentation. Between 13° and 25° most honeys will ferment. Slightly below 27° fermentation develops slightly in unripe but rarely in well-ripened samples. A. G. P.

Moisture in top and bottom layers of honey after one year's storage as indicated by the vacuum drying oven and the refractometer. G. E. MARVIN and H. F. WILSON (J. Econ. Entom., 1932, 25, 514–520). —Crystallisation of honey causes a redistribution of the  $H_2O$  content, that of the top layer being > that of the bottom. Differences may amount to 3.7%.

A. G. P.

Parchment paper as a source of mould spores. J. GREGER (Z. Unters. Lebensm., 1932, 63, 560—564).— Butter wrapped in parchment paper is liable to mould spoilage from the paper. The trouble may be obviated by the use of paper free from sugar, dextrin, etc., and storage in dry rooms. E. B. H.

Fats in frying.—See XII. Insecticides.—See XVI. Saffron. CHCl<sub>a</sub> extraction.—See XX.

## PATENTS.

Manufacture of bread. B. JUKES (B.P. 375,248, 29.3.32).—A dough is made from ingredients which include a high proportion (e.g., 30%) of malt flour, by the addition of a mixture of cottonseed oil and H<sub>2</sub>O, the yeast being mixed in subsequently. The bread is said to be of uniform texture and good crust colour.

E. B. H.

Making of bread. BRIT. ARKADY Co., LTD., and H. HEWITT (B.P. 375,342, 24.2.31).—The fermentation period of a dough may be considerably shortened by the addition of papain as a gluten-softening agent, up to 1 oz of "strength 1:200" being used for 280 lb. of flour. The rate of gas production of the yeast may also be accelerated by the addition of an  $NH_4$  salt.

E. B. H.

Stimulation of sugar production in bread making. L. and E. ELION (B.P. 375,679, 12.11.31).—A persulphate, alone or in combination with a diastatic prep., is added to flour deficient in diastatic power to improve the sugar production during fermentation. E. B. H.

Preserving and maintaining the fresh condition of animal and vegetable substances. H. S. EGEBERG (B.P. 374,927, 14.3.31. Norw., 8.4.30).—Fish and meat are impregnated by immersion in a solution containing 0.1-0.15% of BzOH, *p*-hydroxybenzoic acid, or one of their esters and 4-5% NaCl. The temp. of the solution should differ by  $< 10^\circ$  from that of the substance to be preserved. E. B. H.

Preparation of gelatin food product. SWIFT & Co., Assees. of C. C. ZEIGLER (B.P. 374,894, 17.3.31. U.S., 28.5.30).—Tablets which dissolve rapidly in hot  $H_2O$  to form a gelatin dessert are prepared by drymixing granulated gelatin and sugar, powdered fruit acid, colouring and flavouring matter and then adding sufficient liquid to cause the mass to cohere when compressed. E. B. H.

Treatment of waste materials, fish offal, waste fish, etc. R. A. HARTMANN A.-G., G. N. DAVIDSON, and J. S. WALKER (B.P. 374,166, 11.3.31).—Offal is continuously supplied to a perforated casing wherein it is subjected to action of steam or a solvent and passed into an outer casing where the mixture is discharged, the solid being retained on the screen and removed by means of a helical brush. E. B. H.

Preventing formation of mildew in articles of food. M. RIEGEL (B.P. 375,686, 23.11.31).—The surface of the food is coated with an antiseptic glycerin fluid, *e.g.*, a glycerol formate. H. R.-D.

Treatment [moulding] of bread and like dough. J. E. POINTON, L. S. HABBER, and BAKER PERKINS, LTD. (B.P. 377,264, 16.4.31).

Apparatus for aërating liquids [beverages]. G. GERBER (B.P. 376,842, 14.12.31).

Treating liquids.—See I. Org. Ca phosphate. Theobromine.—See XX.

# XX.-MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Detection of antipyrine in pyramidone. P. DUQUÉNOIS (J. Pharm. Chim., 1932, [viii], 16, 28-31).— When nitrous  $HNO_3$  is added to pyramidone solution a violet ring is formed between the two layers and the acid is coloured greenish-yellow. In the presence of antipyrine a brownish-yellow zone appears under the violet ring and after about 5 min. (2% antipyrine) a green ring due to nitroscantipyrine is formed between the violet and the brown. The reaction is sensitive to 1% of antipyrine. E. H. S.

Pecker's reaction and its relation to the presence of copper in distilled cherry-laurel water. J. GOLSE and J. HUGOT (J. Pharm. Chim., 1932, [viii], 16, 20-26).—The reactions indicating the presence of a reducing substance in cherry-laurel water, *i.e.*, Pecker's  $(NH_4 \text{ molybdate-}H_2SO_4)$  and Meillère's (nitromolybdate- $H_2SO_4$ ) reactions, are due solely to the presence of a cuprocyanic acid formed from HCN and  $Cu_2(CN)_2$ which is derived from the Cu vessels used in distillation. Sol. Sn compounds are not formed by the action of the water on Sn. Methods for the detection of HCN and Sn are given. E. H. S.

Reduction of molybdate reagents by distilled cherry-laurel water. F. MORVILLEZ and (MLLE.) DEFOSSEZ (J. Pharm. Chim., 1932, [viii], 16, 27-28).— Pecker's reaction is due to the presence of Cu, which is present in all commercial cherry-laurel waters. Certain Cu-free samples, after contact with Sn, also give the reaction but the presence of Sn in normal samples is only accidental. E. H. S.

Analysis of saffron. A. BONIS (Ann. Falsif., 1932, 25, 268-271).—Extraction of 1 g. of saffron with 100 c.c. of  $H_2O$  in 10-c.c. portions gives  $59 \cdot 5 - 60 \cdot 50'_{0}$  extract on the original material, calc. to dry wt. Lower figures indicate adulteration with extracted saffron or other material; high vals. are due to sugar or honey, which may be identified by Cu-reducing power, before and after inversion. T. McL.

Relation between physiological activity and content of anthraquinone derivatives of the bark of *Rhamnus purshiana* and of extracts obtained therefrom. J. D. VAN DER GRAAFF (Pharm. Weekblad, 1932, 69, 753-768).—The determination of anthraquinone derivatives (emodins) in plants and plant extracts

is discussed, and the following method is recommended. The sample (100 g.) is dried at 100° for 1 hr. and extracted with dry Et<sub>2</sub>O for 30 min.; the extract is evaporated, the residue dissolved in 6.5 c.c. of 0.067N-Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>, and 3.5 c.c. of 0.1N-HCl are added ( $p_{\rm H} 8.5$ ). The solution is extracted with Et<sub>2</sub>O, and after evaporation of the Et.O the residue is dissolved in 10 c.c. of aq.  $NH_3$ ; the content of emodin-A is then determined by the extinction method with  $NiSO_4$  solution. Emodin-B is determined by extracting the solution in NH<sub>2</sub> with Et<sub>2</sub>O, dissolving the residue in Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>-HCl, and repeating the procedure. Emodin-C is determined by extracting the residue from the original Et.O extraction with 4N-HCl, extracting this solution with Et<sub>2</sub>O, and proceeding as before. Details are given of the emodin contents of extracts of cascara prepared in various ways, and of the quantities retained by the residues. Tests with mice show that the physiological action of such extracts is not due to the emodin present. The fresh bark is extremely active, but the active material H. F. G. has so far not been isolated.

Detection of narcotic drugs. Non-specificity of chemical reactions for *Cannabis indica* and its derivatives. H. TROLLE (Ann. Falsif., 1932, 25, 273-280).—The reactions of Beam and Caspari are not sp. for *C. indica* and its derivatives. Extracts of *Anchusa tinctoria* and mixed extracts prepared from thyme, pyrethrum, cloves, nutmeg, fennel, coriander, alkanet, cinnamon, liquorice, absinthe, ginger, and cassia bark give all the colour reactions described by these authors. T. McL.

Percolation apparatus for chloroform extractions (caffeine determinations). J. GROSSFELD (Z. Unters. Lebensm., 1932, 63, 555-557).—An apparatus for the extraction of aq. solutions with CHCl<sub>3</sub> is on the Soxhlet principle, with a device for causing the solvent in form of dry small drops to pass through the solution. E. B. H.

#### PATENTS.

Manufacture of preparations adapted for producing iodine on surfaces. P. SCHESTAKOFF (B.P. 374,353, 9.10.31. Ger., 10.10.30).—A sol. non-hygroscopic iodide, a neutral salt, and a salt which in the presence of acid liberates I from iodides are melted together and cast into sticks. Similar sticks are prepared by melting  $Al_2(SO_4)_3$ ,18H<sub>2</sub>O with tartaric acid or an alum. By rubbing a wound with an iodide and then with an acid stick free I is liberated in the nascent state.

A. R. P.

Organic calcium phosphate. T. B. WAGNER (U.S.P. 1,833,506, 24.11.31. Appl., 19.5.28).—A solution of a Ca salt of an acid which has no appreciable solvent action on the desired Ca salt, e.g., AcOH, is added to the steep water of maize and the resultant ppt. is collected. Free acid generated during ppta. may be neutralised. E. H. S.

Preparation of stable, supersaturated calcium gluconate solutions. CHEM. FABR. VORM. SANDOZ (B.P. 372,225, 14.10.31. Ger., 14.10.30. Addn. to B.P. 314,460; B., 1929, 698).—The crystallisation of Ca gluconate is retarded by addition of a Ca salt of a monocarboxylic acid derived from aldodisaccharoses; e.g., in 5% Ca lactobionate solution 8.8% of Ca gluconate remains dissolved after 25 days. C. H.

Extracting theobromine from natural products containing theobromine. F. SEITZ, Assr. to C. H. BOEHRINGER SOHN (U.S.P. 1,833,597, 24.11.31. Appl., 23.7.29. Ger., 18.5.29).—Ground products, particularly cocoa and cocoa waste, containing theobromine, moistened with an aq. liquid, e.g.,  $H_2O$ , aq. Ca(OH)<sub>2</sub>, etc., are mixed with excess of powdered CaO or Ca(OH)<sub>2</sub> and the mass is extracted with  $H_2O$ . The extract, with or without addition of an electrolyte, is acidified to obtain theobromine. E. H. S.

Manufacture of 3:5-di-iodo-4-pyridone. SCHER-ING-KAHLBAUM A.-G. (B.P. 372,255, 12.1.32. Ger., 11.2.31).—Di-iodochelidamic (3:5-di-iodo-4-pyridone-2:6-dicarboxylic) acid is decarboxylated by boiling with Ac<sub>2</sub>O, Bz<sub>2</sub>O, or other anhydride, and the product is hydrolysed with H<sub>2</sub>O or aq. NaOH to give 3:5-diiodo-4-pyridone, decomp. >300°. C. H.

Manufacture of N-alkyldi-iodochelidamic acids. SCHERING-KAHLBAUM A.-G. (B.P. 372,245, 30.11.31. Ger., 18.12.30).—Di-iodochelidamic acid is alkylated, e.g., with Me<sub>2</sub>SO<sub>4</sub> and KOH, to give the N-Me compound, decomp. 174°, which can be used in pyclography by intravenous injection without causing rise in bloodpressure and stimulation of the respiratory centre. C. H.

Manufacture of products from [stabilisation of aqueous solutions of] 7-iodo-8-hydroxyquinoline-5-sulphonic acid or its salts. F. KROLLPFEIFFER (B.P. 371,894, 29.1.31. Ger., 30.1.30).—Aq. solutions of 7-iodo-8-hydroxyquinoline-5-sulphonates are stable to boiling in presence of Na 8-hydroxyquinoline-5sulphonate, p-phenolsulphonate,  $\beta$ -naphthol-3:6disulphonate, gallate, gallocarboxylate, pyrocatechol-3:5-disulphonate, or salts of other aromatic or heterocyclic sulphonic acids or hydroxybenzoic acids; alkali, especially NaHCO<sub>3</sub>, may be present. C. H.

Manufacture of amino-substituted organic arsenic compounds. I. G. FARBENIND. A.-G. (B.P. 373,085, 11.1.32. Ger., 10.1.31).—A nitro-1-hydroxy-2-methoxybenzenearsinic acid is reduced, preferably as Na salt with Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>, the Na amino-1-hydroxy-2methoxybenzenearsinate being pptd. by AcOH. The chemotherapeutic index of the products is 1:20 or more. The prep. of 3-amino-4-hydroxy-5-methoxybenzene-1-arsinic acid (reduced further to the arsenoxide, arseno-compound, and arsine) and the 2:3:4:1isomeride is described. C. H.

Manufacture of condensation products of aminosubstituted arseno-compounds. I. G. FARBENIND. A.-G. (B.P. 372,213, 18.9.31. Ger., 20.9.30).—The  $\mathrm{NH}_2$ -groups in a diaminoarsenobenzene are caused to react severally (a) with  $\mathrm{CH}_2\mathrm{O}$ -bisulphite, and (b) with an olefine oxide to give easily tolerated arsenicals valuable against amœbic dysentery. Examples are : 4 : 4'-bis-(4-amino-2 : 3-dimethyl-5-pyrazolon-1-yl)arsenobenzene with glycide and  $\mathrm{CH}_2\mathrm{O}$ -bisulphite ; 3 : 3'-diamino-4 : 4'dihydroxyarsenobenzene with glycide or propylene oxide and  $\mathrm{CH}_2\mathrm{O}$ -bisulphite ; 3 : 3' : 4'-triamino-4-hydroxyarsenobenzene with glycide (2 mols.) and  $\mathrm{CH}_2\mathrm{O}$ bisulphite (1 mol.). C. H. 818 CL. XXI.—Photographic Materials. CL. XXII.—Explosives; Matches. CL. XXIII.—Sanitation etc.

Manufacture of 1-alkoxy- and 1-substituted alkoxy-2-acylamino-benzenearsinic acids. I. G. FARBENIND. A.-G., Assees. of L. CASSELLA & Co. G.м.в.Н. (В.Р. 373,071, 30.11.31. Ger., 29.11.30).-A nitro-o-acylaminophenol is alkylated and reduced. and the NH<sub>2</sub> replaced by AsO<sub>3</sub>H<sub>2</sub>, whereby pure products are obtained without having to separate byproducts (cf. B.P. 295,744 ; B., 1928, 799). Examples 4-nitro-2-acetamidophenol, condensed with are : CH<sub>2</sub>Cl·CO<sub>2</sub>H or CH<sub>2</sub>Cl·CH<sub>2</sub>·OH and reduced ; 5-nitro-2-acetamidophenol, methylated, and reduced; the amine in each case is converted by Bart's reaction into the arsinic acid. C. H.

Manufacture of [organic] antimony compounds. CHEM. FABR. VORM. SANDOZ (B.P. 372,143, 26.6.31. Ger., 8.7.30. Addn. to B.P. 343,898; B., 1931, 654).— A hydrated  $Sb_2O_5$  or  $Sb_2O_3$  is dissolved in aq. solutions of multivalent metal salts of acids of the gluconic type, and the products are isolated, *e.g.*, by pptn. with MeOH. Mg antimonyl gluconate is obtained from Mg gluconate and the Sb hydroxide pptd. from tartar emetic by HCl; Ca, Cu, and Ni antimonyl gluconates and Ca antimonyl mannonate are similarly prepared. C. H.

**Preparation of specific antigen coctoimmunogen.** R. TORIKATA (U.S.P. 1,833,384, 24.11.31. Appl., 29.7.22).—Antigens which are free or nearly free from secondary reactions are prepared from bacterial materials which normally have a tendency to impede the action of the antigen (due to the presence of a substance, "Impedin," which interrupts the mutual action of the antigen with the antiserum) by subjecting the pure culture of human or animal pathological tissues to X- or ultra-violet rays or to 100° for 30 min.

E. H. S.

Aldehydes etc.-See III.

# XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

# PATENTS.

Ink for photographic films. E. B. MIDDLETON, Assr. to DU PONT FILM MANUFG. CORP. (U.S.P. 1,833,086, 24.11.31. Appl., 24.4.28).—Such inks (insol. in EtOH after application to unexposed and undeveloped cellulose ester motion-picture films) comprise cellulose solvents, *e.g.*, "cellosolve," diacetone alcohol, colouring material, *e.g.*, nigrosine base, lampblack, and a thickener, *e.g.*, shellac, sandarac, or preferably nitrocellulose.

S. S. W.

Preparing subtractive multi-colour pictures. I. G. FARBENIND. A.-G. (B.P. 375,338, 24.12.30. Ger., 24.12.29).

# XXII.—EXPLOSIVES; MATCHES.

#### PATENTS.

Production of solid bodies of explosive substances. O. MATTER (B.P. 374,871, 9.3.31. Ger., 4.4.30).—The substance (e.g., trinitrotoluene) is dissolved in a solvent (COMe<sub>2</sub>) and poured, while stirring, into a second miscible liquid (H<sub>2</sub>O) without dissolving the substance; the ppt. is washed, dried, and pressed. H. R.-D. Detonating caps. W. ESCHBACH and H. WIPPEN-HOHN (B.P. 374,060, 20.1.31. Ger., 7.7.30).—A detonating device for projectiles or bombs combines a percussion cap and a detonator, the former containing an igniting composition, e.g.,  $PbN_6$ , which may be mixed with powdered quartz or glass, tetrazene, and/or silicides. Alternatively, the ignition mixture may consist of a friction composition in which KCIO<sub>3</sub> and Sb are replaced by an explosive, such as nitropentaerythritol. W. J. W.

# XXIII.—SANITATION; WATER PURIFICATION.

Microdetermination of carbon in the organic state in water. M. PICON (J. Pharm. Chim., 1932, [viii], 16, 5—20).—A modified Nicloux micro-method (A., 1929, 204) is employed and the analyses of many potable waters are discussed from the viewpoint of the influence of various methods of determination. The results of different methods of evaporation in the hot, the cold, and in presence of HCl or Ba(OH)<sub>2</sub> furnish information on the character of the C compounds present. The quantity of C in the H<sub>2</sub>O diminishes with time and most waters contain recognisable amounts of C even when not indicated by KMnO<sub>4</sub>, which gives only relative vals. Some volatile acids are only slightly affected by KMnO<sub>4</sub>. E. H. S.

Testing of tap water for small quantities of lead and copper. N. SCHOORL (Z. anal. Chem., 1932, 88, 325-336).-Both metals are determined together colorimetrically with Na2S, after addition of AcOH to 0.02N, the colour due to Cu being 1.5 times as intense as that due to Pb. The Pb is determined in a second portion in the same way after addition of aq. NH<sub>3</sub>, NH<sub>4</sub>Cl, and KCN; and finally the Cu alone is determined colorimetrically with K4Fe(CN)6 after addition of NaHCO3. When only minute amounts of Pb are present 1 litre of H<sub>2</sub>O is treated with NaOH to alkalinity, then with KCN and 0.5 g. of CaCO<sub>3</sub>. After shaking for 1 hr., the CaCO<sub>3</sub> containing all the Pb is collected and dissolved in AcOH and the solution tested for Pb as above. Minute amounts of Cu may similarly be collected, using NaOH and MgCl<sub>2</sub>. The pptd. Mg(OH)<sub>2</sub> is dissolved in  $H_2SO_4$  and the filtered solution tested colorimetrically with K4Fe(CN)6. A. R. P.

Insecticides.-See XVI.

#### PATENTS.

**Purification of potable water.** LURGI GES. WÄRME-TECHNIK M.B.H., and F. SIERP (B.P. 374,125, 4.3.31).— Fine-grained C is treated with a suitable nutrient medium containing small quantities of PhOH etc., under aërobic conditions; the surface of the C is thereby charged with organisms capable of absorbing PhOH etc. The material may be used for water purification in conjunction with the main filter and its activity may be maintained by periodic re-aëration.

C. J.

Gas or air filter, particularly respiratory filters. Respiratory filters. B. DRÄGER, Assee. of DRÄGER-WERKE, H. U. B. DRÄGER (B.P. 377,541-2, 23.11.31. Ger., [A] 6.10.31, [B] 26.1.31).