# BRITISH CHEMICAL ABSTRACTS

# B.—APPLIED CHEMISTRY

MAY 20 and 27, 1932.\*

# I.-GENERAL; PLANT; MACHINERY.

Application of theoretical diffusion equations to the drying of solids. T. K. SHERWOOD (Amer. Inst. Chem. Eng., Preprint, 1931, 11 pp.) .- The drying process consists of a const.-rate period followed by a falling-rate period; the latter frequently consists of a period in which the deceleration is due to decrease in wetted surface, followed by a period in which the rate of drying is controlled by the rate of diffusion of H<sub>2</sub>O through the solid. With certain slow-drying materials no const.rate period occurs ; the case is examined mathematically. With many common materials a short const.-rate period is followed by a falling-rate period in which internal liquid diffusion controls the process throughout. Equations are derived for the H<sub>2</sub>O gradient approached in a slab drying at a const. rate. On the assumption that the results apply at the crit. (inflexion) point, the relation between H<sub>2</sub>O content and time is given for the subsequent CHEMICAL ABSTRACTS. falling-rate period.

Drying of porous solids. Diffusion and surfaceemission equations. A. B. NEWMAN (Amer. Inst. Chem. Eng., Preprint, 1931, 9 pp.).—Two stages are postulated: (1) a period of const.-drying rate, when the diffusion of liquid from the interior is sufficiently rapid to keep the surface completely wet, and (2) a period during which the drying rate is supposed to be directly proportional to the free-liquid concn. at the surface at any instant. Mathematical expressions for plane, spherical, and cylindrical solids are given.

CHEMICAL ABSTRACTS.

Heat transmission to oil flowing in pipes. Effect of tube length. T. K. SHERWOOD, D. D. KILEY, and G. E. MANGSEN (Amer. Inst. Chem. Eng., Preprint, 1931, 15 pp.).—A discussion. Experiments on heating oil in both the viscous and the turbulent regions with various lengths of Cu tube are described.

CHEMICAL ABSTRACTS. Micro-analytical methods in the industrial laboratory. III. Micro-apparatus for gravimetric determination of water in coal and other solid materials. F. VETTER (Mikrochem, 1932, 10, 407-408; cf. A., 1932, 72).—The sample is placed in a boat in an 8—10 mm. diam. tube surrounded by a jacket through which PhMe vapour is passed. The H<sub>2</sub>O evolved is carried into an absorption vessel by a stream of dry N<sub>2</sub>. The apparatus may be used also for vac. drying. H. F. GILLBE.

Determination of the hygroscopic equilibria of some Japanese materials. S. KAMEI and T. SEDOHARA (J. Soc. Chem. Ind., Japan, 1932, 35, 57-64 B). --The equilibrium H<sub>2</sub>O contents of various specimens of soap, clay, and wood at 20°, 30°, and 40° have been determined as a function of the R.H. of the atm. In all cases a characteristic S-shaped curve is obtained which shows evidence of adsorption effects at both very low and very high R.H. vals. The influence of humidity on the  $H_2O$  content of the material is greater than is that of temp.; if the temp. is raised at const. humidity, the  $H_2O$  content falls only slightly as compared with the change effected by lowering somewhat the humidity. H. F. GILLBE.

Measurement of smoke. J. F. SHAW (J. Inst. Fuel, 1932, 5, 167-174).-The physical and chemical properties of smoke of different intensities have been correlated with its optical intensity as measured by a modified Féry pyrometer. Screens of various intensities, previously adopted as standard by the Heat Engines Trials Committee of the Institution of Civil Engineers ("Civil" screens), were calibrated with smoke, making use of the optical densities recorded by the Féry pyrometer. Tests made at the Fuel Research Station on the smoke emission of a hand-fired Babcock boiler showed that (1) either the absorption of light passed through the chimney, or a set of "Civil" screens is a satisfactory method of measuring smoke concn., (2) the concn. of the smoke at any fixed load is proportional to the optical density, (3) if the boiler is worked at varying loads, the same optical intensity corresponds to a greater concn. of solid matter at light loads than at full loads, due mainly to the change in the nature of the particles, (4) the amount of light transmitted through the smoke, except with very slight smokes, is very small; the thickness of the smoke column (or diam. of the chimney) has therefore little effect on screen readings with other than light smokes. Preliminary tests made at certain industrial plants are described and the measurement of particle size in the light of recent research is discussed. C. B. MARSON.

Volume and temperature of gas mixtures. E. ANDERSON (Chem. and Met. Eng., 1932, 39, 80).—If vols. of gases at different temp. are mixed the final gas vol. is the sum of the original vols. so long as the pressure and sp. heat remain unchanged. Proof of this is given. Consequently when chimney gases are diluted with cold air, for example, the final vol. is simply the sum of the original vols. The temp.  $T_3 = (V_1 + V_2)T_1T_2/(V_1T_2 + V_2T_1)$ , temp. being expressed in degrees abs.

C. IRWIN.

a

Relative-absolute humidity conversions. A. J. MONACK (Chem. and Met. Eng., 1931, 38, 718-719). A conversion chart for relative (ratio of wt. of  $H_2O$ vapour per cu. ft. of air to that per cu. ft. of saturated air at same temp.) to absolute (ratio of wt. of  $H_2O$ 

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

vapour per lb. of dry air to that per lb. of dry air when air is saturated at the same temp.) humidities is given. D. K. MOORE.

Humidity chart for steam-air mixtures. S. KAMEI (J. Soc. Chem. Ind., Japan, 1932, 35, 51—57 в).— The chart reproduced gives : (1) humidity as a function of temp.; (2) the sp. vol. of dry air and of air containing from 1% to 15% of H<sub>2</sub>O, as a function of temp.; (3) the sp. heat of air as a function of the humidity; and (4) the adiabatic cooling lines. The application of the chart to the rapid solution of drying problems is illustrated by sp. examples. H. F. GILLEE.

Elimination of sulphur compounds from boilerfurnace gases. H. F. JOHNSTONE (Fuel, 1932, 11, 84-90; cf. B., 1931, 613, 701).-70-95% of the S in the coal burned under the boiler appears as gaseous compounds (97-98% SO2, 3-2% SO3) in the furnace gases. It is proposed to remove these by washing the gases with H<sub>2</sub>O and continuously oxidising the dissolved SO<sub>2</sub> by adding a suitable catalyst. Laboratory tests show that Mn and Fe salts are effective catalysts for this reaction, the optimum concn. being approx. 0.025% of Mn or 1.2% of Fe. In the case of the Mn as much as 5400 cu. ft. of gas per gal. of H<sub>2</sub>O can be washed with an average efficiency of  $92 \cdot 2\%$  before the differential efficiency falls to 30%; at this time the concn. of  $H_2SO_4$  in the liquor is  $45 \cdot 5\%$ . Traces of phenols inhibit the catalytic action, as does also H<sub>2</sub>S in the gas in concns. >0.2%. Larger-scale experiments were carried out with a rotary scrubber having a capacity of 100 cu. ft./min. The flue gases had the composition : CO2 5%, O2 15%, SO2 0.1%, and the time of contact was 0.05 sec. The efficiency was lower than that of the laboratory scrubber, but compared favourably with that obtained by other large-scale methods. The vol. of H<sub>2</sub>O required was approx. 270 gals. per ton of coal.

A. B. MANNING.

Microscopical appearance of flue dusts. A. ECCLES and A. McCULLOCH (Fuel, 1932, 11, 102—113).— Photomicrographs of 22 samples of flue dust are reproduced, and an attempt is made to classify the different types of particle observed. A. B. MANNING.

Generalisation concerning plasticisation. O. MANFRED (Z. physikal. Chem., 1932, B, 15, 383—387).— When a substance is caused to undergo plastic deformation by being subjected to a largely unidirectional strain, such as stretching or rolling, its physical characteristics change in such a way with increasing degree of plasticisation as to tend to compensate for the treatment undergone; e.g., the hardness of metals is increased by rolling. R. CUTHILL.

Pivot mounting of pyrometers. Anon. (Chem. and Ind., 1932, 316-317).

NaCl works design.—See VII. Rotary cement kilns.—See IX. Determination of  $p_{\Pi}$ .—See XI. Determining hardness of  $H_2O$ .—See XXIII.

See also A., April, 357, Thermostat. Thermoregulators. Radiation thermocouples.

#### PATENTS.

Rotary kiln and cooler. P. T. LINHARD, Assr. to F. L. SMIDTH & Co. (U.S.P. 1,813,061, 7.7.31. Appl., 18.12.28).—In a kiln having a number of cooling drums surrounding the discharge end, those parts of the cooling drums that receive the clinker from the main kiln are formed with a spiral enlargement so that in less than half a revolution the material is moved away from the ports and does not drop back into the kiln. B. M. VENABLES.

Drying apparatus. H. ANHALTZER (U.S.P. 1,813,133, 7.7.31. Appl., 13.3.28).—Viscous material is spread between two belt conveyors which separate and take a sinuous course in a drying chamber. The guide rollers that touch the belts on the same side as the material are deeply fluted so that they make line contact only with the belts. B. M. VENABLES.

Disposal of [fly-]ash in furnaces. INTERNAT. COMBUSTION ENG. CORP., Assees. of W. R. WOOD (B.P. 368,031, 10.2.31. U.S., 7.7.30).—The light ash deposited in parts other than in the combustion chamber is collected and returned to any furnace having a slagging bottom, through ports which are below those for the main fuel supply. B. M. VENABLES.

Removal of scale-forming material from boilers or hot-water cisterns. C. FUHRY and J. MøLLMANN (B.P. 368,143, 15.5.31).—Folded or rolled wire netting is placed close to the cold-water inlet and at other places where scale tends to deposit. Guides may be provided to cause the convection flows to pass through the netting. B. M. VENABLES.

Autoclave. J. J. GREBE, Assr. to Dow CHEM. Co. (U.S.P. 1,814,796, 14.7.31. Appl., 23.4.30).—In an autoclave for treatment of a liquid which is an electrolyte with the aid of a metallic catalyst, the latter is electrically insulated from the wall of the vessel, which may then be of metal adapted to resist corrosion and pressure irrespective of the electrical potential between it and the catalyst. B. M. VENABLES.

Arrangement for heat-treating materials by means of air or other gases. J. M. PETERSON (B.P. 366,912, 4.10.30).—The apparatus comprises a double co-axial drum rotating between fixed end-members. The annular space is divided by radial partitions, and only one or two at a time of these divisions are open to the inlet and outlet ports for gases in the end-members. After passing through the annular space, the gases return in contact with the material in the central space. B. M. VENABLES.

Catalytic apparatus. J. M. SELDEN, ASST. to SELDEN Co. (U.S.P. 1,810,147, 16.6.31. Appl., 26.9.27).—An apparatus as described in U.S.P. 1,647,317 (B., 1928, 42) is provided with heating means. B. M. VENABLES.

Effecting [exothermic] reactions between gases with the aid of a catalyst. Soc. NAT. DE RECHERCHES SUR LE TRAITEMENT DES COMBUSTIBLES (B.P. 367,772, 18.10.30. Fr., 21.10.29).—An apparatus in which the gas passes in succession through a heat exchanger, outside the catalyst tubes, through a heater, inside the catalyst tubes, and the reverse way through the heat exchanger is provided with one or more by-pass valves and passages by which a proportion of the gas may avoid passing through the heat exchanger and/or the outside of the catalyst tubes. B. M. VENABLES.

Treatment of materials for employment in chemical reactions as catalysts, catalyst supports,

404

fillers, constructional materials for reaction vessels or linings thereof, and employment of such materials in chemical reactions. BRIT. CELANESE, LTD., and W. BADER (B.P. 366,973, 7.11.30).—Materials (graphite, fireclay, bauxite, pumice, etc.) for use, e.g., in the thermal decomp. of aliphatic acids into their anhydrides or for the production of oxygenated org. compounds from oxides of C and  $H_2$ , are treated with an aq. solution of a hydroxycarboxylic acid (tartaric, malic, or lactic acid) and extracted with a volatile solvent (AcOH) to remove Fe and other metallic impurities.

L. A. Coles.

Apparatus for exchanging heat [between two liquids]. G. D. DODD (U.S.P. 1,813,234, 7.7.31. Appl., 24.3.28).—A tubular heat exchanger is divided into two sections in both of which the cooler liquid takes a sinuous course; in the hotter of the two sections the heating liquid is flashed into vapour by reduced pressure and condensed in the cooler section. B. M. VENABLES.

Heat-generating compound. J. R. DULANY, Assr. to CHAMBERLAIN Co. (U.S.P. 1,814,741, 14.7.31. Appl., 10.9.27).—The composition comprises dry NaOH or KOH and the dross from the melting of Al.

L. A. COLES.

**Refrigerants.** L. K. WRIGHT (U.S.P. 1,813,335—6, 7.7.31. Appl., [A] 7.10.27, [B] 8.11.27).—The use is claimed of (A) allylene [either  $C(CH_2)_2$  or CH:CMe], or (B) butylene (preferably  $\Delta^{\beta}$ -butylene); these may be used in a simple-compression apparatus or in conjunction with an absorbent (EtOH) or an adsorbent (SiO<sub>2</sub> gel, activated C, etc.). L. A. COLES.

Fire extinguisher. C. S. THOMPSON, ASST. to THOMPSON MANUFG. Co. (U.S.P. 1,813,367, 7.7.31. Appl., 24.11.25).—The composition comprises, e.g., 3 lb. of  $As_2O_3$ , 7 lb. of borax, 6 lb. of NaOH, and sufficient  $H_2O$  to yield 5 gals. of solution. L. A. COLES.

Pulverising mills. C. R. TAYLOR (B.P. 367,124, 16.1.31).—The apparatus has a vertical shaft, and a fan chamber which is situated above the beater chamber  $(\mathcal{A})$  with a narrow neck between. The fineness of the material drawn away is regulated by the amount of air admitted through the bottom of  $\mathcal{A}$ , a ring-shaped valve being provided for the purpose. B. M. VENABLES.

[Cooling of] grinding mill. V. SAHMEL, Assr. to F. L. SMIDTH & Co. (U.S.P. 1,813,086, 7.7.31. Appl., 14.11.29).—The temp. of a mill grinding Portland cement is regulated by external cooling  $H_2O$  or other means the flow of which is started when the motor starts and is controlled by a thermostat in the trunnion. Another thermostat operates a warning device should a certain temp. be exceeded. B. M. VENABLES.

Stone-breaking and other crushing machines. H. R. MARSDEN, LTD., and N. TAYLOR (B.P. 367,631, 24.4.31).—The liner of the fixed jaw is held in place merely by downward pressure of wedge bolts.

B. M. VENABLES.

Pulverisers. ERIE CITY IRON WORKS, Assees. of G. H. KAEMMERLING (B.P. 366,529, 1.11.30. U.S., 2.9.30).—Forms of renewable paddles are described. B. M. VENABLES.

Fine grinding of coal, pigments, raw materials for manufacture of cement, and the like. K. MIDDELBOE (B.P. 367,548, 9.2.31).—In an apparatus in which a number of smaller rolls or grinding bodies rotate in contact with either a large central roll or an outer ring, the smaller rolls are coupled together in pairs or threes so that the distances between the axes of a set remains const. B. M. VENABLES.

Apparatus for cooling and screening granular substances [e.g., poultry and cattle foods]. W. DRYDEN (B.P. 366,922, 8.11.30).—The apparatus comprises a cylindrical, rotating screen divided into zones of different mesh and provided with a hollow core which may or may not be used for the passage of cooling medium; the main part of the cooling is effected by admission of air or gas through the wall of the hopper which collects fines, through the screen, and out at the end where the material enters. B. M. VENABLES.

Production of finely-divided material. A. B. CUMMINS, ASST. to CELITE CORP. (U.S.P. 1,813,916, 14.7.31. Appl., 29.4.29).—Diatomaceous earth is separated by a pneumatic system into grades of the desired fineness and of excessive fineness; the latter is then fritted in a furnace and returned to the grading circuit. B. M. VENABLES.

Mixer. A. J. SACKETT, Assr. to A. J. SACKETT & Sons Co. (U.S.P. 1,812,450, 30.6.31. Appl., 14.7.28).— A mixer of large diam. is provided with a sliding joint between the bottom and side, the former being heated. The stirring gear is of planetary type and a central bottom discharge is provided. B. M. VENABLES.

Mixing machine. F. JOHNSON (U.S.P. 1,811,719, 23.6.31. Appl., 1.3.29).—A trough-shaped mixer with renewable sheet-metal bottom is described.

B. M. VENABLES. Machines adapted for use for mixing, emulsifying, homogenising, comminuting, and like purposes. W. R. SERGEANT, and BURT, BOULTON & HAYWOOD, LTD. (B.P. 567,368, 16.10.30).—Beaters of egg-shaped or other section are rotated in annular spaces between fixed (or relatively rotating) annular passages provided with projections having small and adjustable clearance from the beaters so that the latter sometimes have a shearing action and sometimes are moving in free spaces, producing turbulence. Ports are provided in the webs so that the fluid passes through the annuli in succession, either inwardly or outwardly.

B. M. VENABLES. Mixing of powdered solid materials with liquids particularly in the production of dough. W. W. TRIGGS. From AMER. MACHINE & FOUNDRY Co. (B.P. 367,090, 19.12.30).—The solid material, e.g., flour, is allowed to fall in a hollow cylindrical stream into sprays of the liquid which are horizontal or inclined and are tangential to a circle of smaller radius than that of the falling flour. The instantaneously made dough is collected by a helical scraper from the wall of the spraying chamber and delivered by means such as a screw conveyor to extrusion or dividing devices.

B. M. VENABLES.

Separation of materials from one another. B. NORTON and J. N. COLLINS (B.P. 367,170, 12.2.31).— An apparatus, *e.g.*, for the de-dusting of coal, comprises a series of steps over which the material is caused to fall

by vibration and/or gravity and across which currents of air are directed; the dust-laden air passes through a cyclone separator and is returned by a fan for re-use. The surface of the steps is preferably corrugated and the air arrives through louvres, but leaves through nozzles at a greater velocity. B. M. VENABLES.

Vibratory sifting apparatus for treating potters' slip, powdered substances, and the like. W. Pop-MORE and O. G. COLLIER (B.P. 367,047, 20.11.30).—A lawn frame or other screen is vibrated in the longitudinal direction by means of an a.c. electromagnet.

B. M. VENABLES.

Centrifugal separating machines. RAMESOHL & SCHMIDT A.-G. (B.P. 366,873, 14.8.31. Ger., 14.8.30).— A centrifuge for the separation of liquid from solids and heavy matter is opened at intervals to discharge the latter by cutting off the supply of a hydraulic liquid normally acting on the top of a conical inner wall of the bowl and holding the peripheral outlet closed. B. M. VENABLES.

Centrifugal separators. T. A. BRYSON (B.P. 367,301, 14.8.31. U.S., 26.8.30).—A centrifugal separator for materials that must be treated in thin layers, *e.g.*, paper pulp, which has both light and heavy impurities, is provided with several concentric drums which are supplied with separate portions of the original material. The drums are provided with internal annular baffles pierced at an intermediate radius, so that the pure middle layer can flow to the outlet while the inner and outer layers of impurities are retained and removed after stopping the apparatus. B. M. VENABLES.

Separator. W. T. POWERS, ASST. to DIRECT SEPARA-TOR CO., INC. (U.S.P. 1,813,637, 7.7.31. Appl., 20.9.27).— A separator of the deflection type for, *e.g.*, removing water from compressed air is described.

B. M. VENABLES.

Regulating device for the supply of liquid to centrifugal separators. AKTIEB. SEPARATOR (B.P. 368,167, 12.6.32. Swed., 21.6.30).—In a separator of the type in which the solid matter is removed by a carrier liquid, the separated and carrier liquids are continuously weighed and the motion of the balance beam is caused to decrease the supply of carrier liquid when the flow of separated liquid increases, and vice versa. Alternatively, the regulation of the carrier liquid may be effected by the difference or proportion between the unseparated feed and the separated liquid.

#### B. M. VENABLES.

Multilayer filter. R. L. SEBASTIAN and L. FINKEL-STEIN, ASSTS. to H. A. KUHN (U.S.P. 1,814,190, 14.7.31. Appl., 28.10.26).—The efficiency of a filter, especially of the type used in gas masks, is increased by the closing of all the pores below a certain size by passing a solid smoke through the layers separately. Since the flow of a gas through a capillary tube is proportional to the 4th power of the diam., the resistance to breathing is not much increased by closing the smaller apertures ; the use of a few large apertures widely spaced causes deposition of the foreign matter on the surface of the filter rather than in the pores, because the particles have a considerable distance to flow parallel to the surface before reaching an aperture. B. M. VENABLES. Filtering means. J. J. NAUGLE (U.S.P. 1,813,073, 7.7.31. Appl., 25.5.28).—Means for withdrawing filtrate from filter leaves are described. B. M. VENABLES.

Liquid-purifying device. J. H., Assr. to M., STOCK-HOLDER (U.S.P. 1,813,687, 7.7.31. Appl., 8.7.30).—A method of rendering mineral oil-contaminated  $H_2O$ potable comprises the stages of heating to remove odours, settling, and filtering. The  $H_2O$  is preferably sprayed in the first two stages. B. M. VENABLES.

[Preparation of] emulsion of immiscible ingredients. N. BENDIXEN and J. C. VREDENBURG (B.P. 367,890, 27.11.30 and 28.2.31).—The constituents are passed centrifugally through a number of concentric rings having suitable apertures. B. M. VENABLES.

Manufacture of articles in which volatile substances are evaporated. CARRIER ENG. Co., LTD., and J. S. JONES (B.P. 366,968, 5.11.30).—A volatile substance is removed from manufactured articles by the circulation over them of a substantially unchanged body of gas which is reconditioned both as regards temp. and content of volatile substance by means of sprays of a scrubbing liquid which is a solvent for the volatile substance. The scrubbing liquid and the substance are separated by refrigeration, which is effected on a variable fraction of the flow so that the scrubbing liquid is cooled to the correct temp. for re-use, under the control of a thermostat situated in the current of gas leaving the scrubber. B. M. VENABLES.

Recovery of gaseous or vaporous substances from gases or vapours, or mixtures of both, by fixation with adsorption media and expulsion by means of steam. METALLGES. A.-G. (B.P. 367,209, 16.3.31. Ger., 1.5.30) .- After the expulsion of previously adsorbed constituents by means of steam, the treatment of a fresh batch of gases immediately commences; the gases are supplied at raised temp. during the whole, or the commencement only, of the adsorption period. The adsorption proceeds at once because although the adsorbent near the inlet may be too hot, and near the outlet too wet, some intermediate portion is sufficiently dry and cool (by evaporation) to be effective. Temp. of 40-70°, in some cases up to 120°, are claimed as suitable. B. M. VENABLES.

Preventing gas absorption in liquids and rusting of submerged iron [in, e.g., water-seal gasometers]. B. W. MUELLER, Assr. to P. LECHLER (U.S.P. 1,814,053, 14.7.31. Appl., 17.7.28. Ger., 8.8.27).— The H<sub>2</sub>O is completely covered by a layer  $< \frac{1}{2}$  in. thick of a viscous mixture, d < 1, of hydrocarbon oil and bituminous material, e.g., a mixture of 20 pts. of molten paraffin pitch and 80 pts. of mineral oil of high flash point. L. A. COLES.

Separation of gaseous mixtures into their constituents. I. H. LEVIN, and GAS INDUSTRIES Co. (B.P. 366,934, 8.11.30).—A rectification system is described in which the gaseous mixture leaves the expansion engine in a dry state and, before entering the rectifier, is cooled by heat exchange with the separated products so that it becomes wet; the products then pass to another heat exchanger between the compressor and expansion engine. The rectifier is divided into two stages at different pressures. B. M. VENABLES. Separation of substances from a gaseous medium. W. W. TRIGGS. From ELEKTRISKA SVETS-NINGS AKTIEBOLAGET (B.P. 366,926, 7.8.30).—A cyclone separator with horizontal axis is described. Layers of conc. dust are skimmed off at the lower part of the circumference (which causes a gradual decrease in centrifugal force) and towards the clean end the angular velocity of whirling is increased by tapering the shell. B. M. VENABLES.

Apparatus for extracting or separating dust or other matter from air or gases. HEENAN & FROUDE, LTD., and G. H. WALKER (B.P. 367,120, 14.1.31).— The apparatus comprises a fan and turbine adjacent on the same shaft, but separated by an imperforate diaphragm. The dirty air enters near the axis of the fan, and the clean air leaves near the axis of the turbine; intermediately the heavy material is removed by centrifugal force through perforations in a cylindrical screen surrounding the combined rotor.

B. M. VENABLES.

[Air-]separating apparatus. E. C. ST.-JACQUES (B.P. 367,194, 3.3.31. Fr., 5.3.30).—The apparatus comprises a cylindrical casing surmounted by a cone through the apex of which the fine material is withdrawn in a current of air which is admitted through the base of the apparatus and through a series of adjustable apertures in the cylindrical wall. The material is projected in through a tangential inlet above the air apertures and inclined horizontally or downwards.

B. M. VENABLES.

Gas-scrubbing device. A. L. GENTER, Assr. to GENTER THICKENER Co. (U.S.P. 1,814,377, 14.7.31. Appl., 23.8.27).—A scrubber is superposed upon a thickener filter. B. M. VENABLES.

Friction material [brake linings]. WORLD BESTOS CORP., Assees. of W. NANFELDT (B.P. 367,176, 17.2.31. U.S., 20.2.30).—The material contains (preferably) 45% of asbestos fibre, 3.6% of MgO, 18% of smoked rubber, 9% of S, 6% of graphite, 6% of PbO, 3% of Fe<sub>2</sub>O<sub>3</sub>, and 9.1% of kaolin; a mixture of the rubber, previously softened with a volatile solvent, with the other constituents is partly cured in band form and curing is completed after the final shaping.

L. A. Coles.

Manufacture of hydrometer floats. H. F. CHRISTIE and K. W. COUSE (U.S.P. 1,813,384, 7.7.31. Appl., 7.6.27).—The hydrometer is loaded with Pb shot embedded in fusible material such as wax. It is heated in stages in a liquid of higher sp. gr. than it is capable of measuring, so as to exaggerate any lack of uprightness, which fault is corrected by tilting by hand in the opposite direction until the hydrometer floats upright; then the wax is allowed to set. B. M. VENABLES.

Kilns and the like. C. A. JENSEN. From BESSEMER CEMENT CORP. (B.P. 368,015, 26.1.31).—See U.S.P. 1,793,408; B., 1931, 951.

[Shield for carbon dioxide] fire-extinguishing apparatus. W. KIDDE & Co., INC., Assees. of D. MAPES (B.P. 368,837, 12.12.30. U.S., 3.1.30).

Apparatus for heating liquids. [Spray-type geyser.] L. F. THOMPSON (B.P. 368,175, 25.6.31).

Means for repairing retorts and other structures. T. FRASER (B.P. 369,365, 3.3.31).

[Means for stretching the screens of] vibrating sieves. SALT UNION, LTD., and R. BENNET (B.P. 368,026, 5.2.31).

[Screen plate for] screening and filtering apparatus. F. W. BRACKETT (B.P. 367,144, 28.1.31).

[Illuminator for] thermometers, pressure gauges, and like indicators for industrial purposes. D. GILLIES (B.P. 367,906, 28.11.30).

Electrostatic apparatus for gases. Diaphragms for filters etc.—See XI. Coated containers.—See XIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Chemical constitution of coal. Action of chlorine on coal. III. Action of solvents. A. Eccles, H. KAY, and A. MCCULLOCH (J.S.C.I., 1932, 51, 49-59 T).-The constitution of the ulmic portion of bituminous coal is discussed on a basis of the products of oxidation of coal. C6H6 extracts 10.20% of chlorinated Arley coal and 21.75% of chlorinated Lower Mountain Mine coal. Et<sub>2</sub>O, light petroleum, and EtOH extract fusible constituents from the  $C_6H_6$  extracts in varying amount. The residue from the  $C_6H_6$  extraction and portions of the C<sub>6</sub>H<sub>6</sub> extract are non-fusible. COMe<sub>2</sub>-sol. products from the C6H6 residue are contaminated with COMe2 condensation products. CHCl<sub>3</sub>, as well as dissolving all the above extracts, dissolves further quantities of the COMe<sub>2</sub>-insol. residue. The use of pyridine following that of CHCl<sub>3</sub> extracts additional small quantities of material. All the extracts and the residues contain a large proportion of Cl.

Italian lignites and peats. I. UBALDINI and F. MAGALDI (Annali Chim. Appl., 1932, 22, 99—131).— Analytical determinations on 3 peats and 13 lignites show that the lignites from Sala Bossi and Valdarno are of xyloid nature and contain large quantities of resinous bitumen, and those from Gualdo Cattaneo and Mercure are rich in humic substances. O. F. LUBATTI.

Gasification of coals with oxygen at low temperature. G. NATTA and M. STRADA (Giorn. Chim. Ind. Appl., 1932, 14, 76-86).—Gasification of coal poor in volatile matter at low temp. (700-800°) with O2 and excess of steam yields directly a gas which is rich in H<sub>2</sub> and very poor in inert components; the mixture, after elimination of CO2, may be used in such org. syntheses as that of alcohols from water-gas. The conditions have been determined for obtaining, in the resultant gas, vals. of the ratio H<sub>2</sub>: CO varying from 1.5 to 4. The val. 2-2.5, which is the optimum for the synthesis of MeOH, requires the consumption of 0.28 kg. of C and 0.18 cub. m. of  $O_2$  per cub. m. of gas obtained, and a temp. of about 750° in the lower principal zone of gasification. In an industrial generator in which O<sub>2</sub> is used, it is not possible, even with highly reactive coals, to realise conditions of chemical equilibrium between the reacting gases, the divergence from such conditions increasing at low working temp. On the basis of the prices of coal, electrical energy, and O, (produced either by distillation or electrolytically), the cost of the 1:2-2.5 mixture of CO and  $H_2$  is calc.

The costs of H, obtained from ordinary water-gas and from the gas formed in a generator using O<sub>2</sub> are also compared. Even allowing for the cost of O2, gasification with O2 appears to be economical for units producing at least 1000 cu. m. of gas per hr. T. H. POPE.

Influence of catalysts in the liquefaction of Fushun coal by berginisation. IV. X-Ray spectroscopic analysis of Fe<sub>3</sub>O<sub>4</sub> catalyst. V. Influence of FeO catalyst. VI. X-Ray spectroscopic analysis of FeO catalyst. R. ABE (J. Soc. Chem. Ind., Japan, 1932, 35, 17-19 в, 19-22 в, 23-25 в).-IV. X-Ray spectroscopic analysis of the Fe<sub>3</sub>O<sub>4</sub> catalyst gave a spectrum similar to that of the  $\text{Fe}_2O_3$  catalyst and indicated the presence of  $\alpha$ -Fe and  $\text{Fe}_3O_4$  at the beginning of the reaction. The reduction of the Fe<sub>3</sub>O<sub>4</sub> was much slower than that of Fe<sub>2</sub>O<sub>3</sub>; pronounced Fe<sub>3</sub>O<sub>4</sub> spectra were obtained after 60 min. reaction, but after 90 min. the spectra showed the presence of  $\alpha$ -Fe and very small amounts of Fe<sub>3</sub>O<sub>4</sub>.

V. The yields and analyses of products obtained by treatment of paraffin oil and Kojoshi-Sakuraso coal with H<sub>2</sub> (75 kg./sq. cm. initial pressure) in the presence of pure FeO catalyst are given. The gas pressure with FeO was lower than with Fe<sub>3</sub>O<sub>4</sub> and Fe<sub>2</sub>O<sub>3</sub> and gave different pressure/temp. curves than the last-named. The gaseous products contained no detectable H<sub>2</sub>S and traces of  $NH_3$ ; the amounts of  $H_2O$  and  $CO_2$  were slightly less, and of CO slightly greater, than those obtained with Fe<sub>2</sub>O<sub>3</sub> and Fe<sub>3</sub>O<sub>4</sub> catalysts. Yields of hydrocarbons capable of absorption with active C were small. 34.79% liquefaction of the coal was obtained after 30 min. at 400°. FeO did not catalyse hydrogenation as readily as did  $Fe_2O_3$  and  $Fe_3O_4$ , but it induced greater liquefaction. The yield of oils decreased with increased amounts of absorbed H<sub>2</sub> and of gaseous hydrocarbons.

VI. X-Ray spectroscopic analysis of the FeO catalyst indicated that after the reaction  $\alpha$ -Fe is always present mixed with Fe<sub>3</sub>O<sub>4</sub> (probably formed from the O<sub>2</sub> impurity in the H2) and some unchanged FeO. When the catalyst was completely reduced to pure Fe no increased liquefaction of the coal was observed.

H. E. BLAYDEN.

Tarry water from the lignite of Valdarno [Tuscany]. I. UBALDINI and A. MARIOTTI (Annali Chim. Appl., 1932, 22, 131-137).-The tarry water obtained from the dry distillation at low temp. of the white variety of these lignites is strongly acid as these materials are rich in O and give a tar containing large amounts of phenols and carboxyl compounds. The acid fraction of these waters contains only a small amount of lower fatty acids and is unimportant from the practical point of view. Sol. aldehydic substances are also present. O. F. LUBATTI.

Chemistry of low-temperature tar. G. T. Mor-GAN (J.S.C.I., 1932, 51, 67-80 T).-Jubilee Memorial lecture.

Hydrogenation of low-temperature tar products. G. T. MORGAN and J. T. VERYARD (J.S.C.I., 1932, 51, 79-81 T).-Waxes from low-temp. tar are converted by hydrogenation under pressure into mobile colourless oil to the extent of 85%. The distillates from this crude oil, which are water-white and stable on exposure, are of low sp. gr. and low I val. When heated for 2 hr. at 420° with H, under 120 atm. in presence of MoO3 and S, resinols, resinenes, and resinamines from low-temp. tar yielded 66%, 71%, and 66%, respectively, of mobile fluorescent oil. These oils contained 33, 43, and 33% respectively of neutral oils. The resinol product contains petrol-sol. bases and the resinene product both phenols and bases, whereas the resinamine product contained no phenols.  $C_6H_6$ , PhMe, and  $C_{10}\dot{H}_8$  were also detected in the neutral oils from hydrogenated resinols, whereas the phenolic fraction of this hydrogenation gave a trace of PhOH. The hydrogenated oils from resinenes and resinamines also contained C<sub>10</sub>H<sub>8</sub>.

Extraction of phenols in tar oils with liquid ammonia. M. JARRY (Compt. rend., 1932, 194, 614-615).-The phenols pass almost completely into the lighter NH<sub>3</sub> layer when the oil is rapidly agitated with liquid NH3 at room temp. After siphoning off the heavy neutral layer, evaporation gives the phenols and an almost complete recovery of the NH<sub>3</sub>. J. W. BAKER.

Benzol purification by electrolytic oxidation. W. GLUUD, B. LÖPMANN, and K. KELLER (Ber. Ges. Kohlentech., 1932, 4, 82-95).-A mixture of 1-2 pts. of benzol and 1 pt. of 15-20% H<sub>2</sub>SO<sub>4</sub> was electrolysed between Pb plates, using a diaphragm, at 8 volts and 0.03 amp./sq. cm. with agitation. Brown flakes separated and for the most part settled with the acid. The remainder were removed from the benzol by a dil. soda wash. For benzol treated in this way the  $H_2SO_4$  coloration test is not suitable. The  $O_2$  or resinformation test (B., 1930, 1137) is recommended. Too low c.d. gives imperfect rectification, too high perhaps causes benzol losses. This may be associated with the formation of  $PbO_2$ . The best anode potential is obtained with an anode polished with sand ; a wire brush is not so effective. Washing is rather more efficient if the crude benzol contains a fraction of b.p.  $> 145^{\circ}$ . The experiments described are only on the laboratory scale, but gave a product which remained stable for 5 months. Larger-scale tests are to be made. C. IRWIN.

Relation between the composition of retortable carbonaceous materials and their yield of crude oil. H. BRIGGS (Proc. Roy. Soc. Edinburgh, 1930-31, 51, 142-147).-An attempt is made to correlate graphically the yield of crude oil (gals./ton) and the % of volatile hydrocarbons (ash-free dry basis), C/H ratio (dry ash-, S-, and N-free basis), and the vals. of (% of volatile hydrocarbons —  $[2 \times \% 0]$ ). When retorted under very similar conditions, Scottish oil shales show irregularities which are attributed to differences in chemical nature of the org. constituents. For minerals of higher rank than lignite, the following approx. equation applies:  $Y = 0.47 [V - (2 \times O)]^{4/3}$ , where Y is the yield of oil in gals./ton and V and Oare the % volatile hydrocarbons and % O, respectively, on dry ash- and S-free basis. H. E. BLAYDEN.

Action of volcanic ash and pumice on oils. T. TERADA, M. HIRATA, and R. YAMAMOTO (Proc. Imp. Acad. Tokyo, 1931, 7, 348-350).-Certain relationships between the densities, viscosities, mol. spacings, and  $n_{\rm D}$  of the distillates obtained by heating different animal and vegetable oils with volcanic ash or pumice are very similar to the corresponding relationships for mineral oils from U.S.A., Russia, and Japan, in spite of marked chemical differences between the two classes. This suggests a possible explanation of the origin of the Japanese Tertiary oil fields. N. H. HARTSHORNE.

Properties and compositions of the gasoline fractions of representative Japanese crude petroleums. VIII. M. MIZUTA (J. Soc. Chem. Ind., Japan, 1932, 35, 80 B; cf. B., 1932, 375).—68 gasoline fractions from Japanese crude oils were analysed for aromatic, paraffin, and unsaturated hydrocarbons. The composition of a mixture of these can be calc. approx. from the observed sp. gr. of the mixture. J. L. D'SILVA.

Total heats and specific heats of synthetic paraffinic distillates and some petroleum fractions. A. J. E. SWANN (Fuel, 1932, 11, 113-118).—The total heats of five light oils have been determined over the range from 0° to their initial b.p. by the continuous-mixture method (cf. Lang, Jessel, and Steed, B., 1931, 143). Three of the oils were obtained by cracking paraffin wax in the presence of anhyd. AlCl<sub>2</sub> and subsequently refining and fractionating the product. These oils contained <1.5% of unsaturated hydrocarbons and naphthenes; their boiling ranges were 100-130°, 130-160°, and 160-190°, respectively. The other two oils were fractions of a straight-run Persian distillate and had boiling ranges 100-125° and 125-150°, respectively. The total heats from 0° were parabolic functions of the temp. (t); e.g., that of the first of the synthetic paraffin distillates (b.p. 100-130°) could be represented by  $H = 0.524t + 0.0004t^2$ , and that of the Persian dis-tillate (b.p. 100-125°) by  $H = 0.450t + 0.00068t^2$ . The sp. heat of the paraffin distillates were linear functions of their sp. gr. and could be represented by the equation: sp. heat = 1.65 - 0.913d + 0.0008t. The temp, coeff. of sp. heat was independent of the sp. gr. The mean mol. wt. and the n were also linear functions of the sp. gr. A. B. MANNING.

Heat-insulation of the Engler flask during the distillation test [for fuel oils]. M. MIZUTA (J. Soc. Chem. Ind., Japan, 1932, 35, 51 B).—The best method for insulating the distilling flask (for the Engler test of petrol, kerosene, and gas oil) is to coil asbestos cord around the neck; there is no advantage in covering the shield with asbestos sheet except when testing kerosene and gas oil, whilst for petrol alone it is not necessary to use either the sheet or the coil insulation. H. F. GILLBE.

Recent advances in gasoline extraction by the active-carbon process. E. REISEMANN (Petroleum, 1932, 28, No. 6, 1—13).—Modern methods of gasoline extraction from natural gas are described with particular reference to the Bayer active-C process in which efficiency is increased by utilising the residual gas in a circulatory system for drying and cooling the adsorbent C after steaming. Typical analyses of natural gases are given and the factors determining adsorption efficiency discussed. The composition of the extracted gasoline is markedly influenced by the moisture content of the adsorption C, and may be varied by variation of the presteaming temp. and the height of C charge. The extraction of products rich in  $C_2H_6$ ,  $C_3H_6$ , and  $C_4H_{10}$  is discussed and their utilisation briefly considered.

H. E. BLAYDEN.

Oxidation of petroleum oils in presence of analytically inappreciable catalysts. K. I. IVANOV and N. N. PETIN (J. Gen. Chem. Russ., 1931, 1, 704— 716).—Washing with tap-H<sub>2</sub>O greatly increases the oxidisability (by O<sub>2</sub> under pressure) of two samples of Caucasian petroleum, one consisting mainly of paraffins and the other of naphthens, but both containing naphthenic acids. Boiling with H<sub>2</sub>SO<sub>4</sub> and subsequent neutralisation with Ca(OH)<sub>2</sub> destroys the activity of the H<sub>2</sub>O; distilled H<sub>2</sub>O and "synthetic tap-H<sub>2</sub>O" are inactive, and addition of Fe, both in colloidal and in salt form, does not activate them. G. A. R. KON.

Heat transmission to oil. Determining  $H_2O$  in coal.—See I. Concn. of  $K_4Fe(CN)_8$ .—See VII.

See also A., April, 354, Determining CO. 360, Formation and bacteria of coal. Peat soils.

# PATENTS.

Coke ovens. C. STILL (B.P. 368,180, 7.7.31. Ger., 12.7.30).—The smoke formed during the charging and emptying of coke-oven chambers is burned in a suction tube constructed like a chimney which can be fitted over an opening in the oven roof. Compressed air is introduced into the lower part of the tube through a jet injector, and additional air for combustion is supplied through an annular space in the upper part of the tube. A. B. MANNING.

Coke ovens. AKZIONERNOJE OBSCHTSCHESTWO PO PATENTOWANIJU I REALISATZIJ ISOBRETENIJ "PRIS" (B.P. 368,194, 17.8.31. Ger., 29.8.30).—A coke-oven battery comprises alternate coking and heating chambers. Each heating chamber is connected at each end by floor openings with combustion chambers in which combustion takes place alternately so that the heating gases traverse the entire length of each heating chamber in alternately opposed directions. The air for combustion is preheated in regenerators or recuperators heated by the waste gases. A. B. MANNING.

**Regenerative coke ovens.** W. MUELLER (B.P. 368,095, 30.3.31).—Between the oven chambers are heating walls each comprising a single bank of vertical flues. Below the chambers are regenerators, each of which is connected to the lower ends and the upper ends, respectively, of the flues of two consecutive heating walls. The flues of alternate heating walls are heated with upwardly flowing flames, whilst the flues of the intermediate heating walls are heated by downwardly flowing flames, the flow in all flues being reversed at suitable intervals. A. B. MANNING.

[Production of active carbon by] treatment of pitches and tars. O. D. CUNNINGHAM, Assr. to P. C. REILLY (U.S.P. 1,813,341, 7.7.31. Appl., 30.4.25).— Pitch or tar is emulsified, preferably with NaOH or KOH, the emulsion is carbonised with stirring at a low temp. (400°), and any residual volatile matter is then removed either by extraction with light tar oils, COMe<sub>2</sub>, etc., or by further heating of the semi-coke in admixture with an inert powdered spacing material. The product is washed with H<sub>2</sub>O and dil. HCl, calcined at 900°, activated by partly burning it in combustion gases or steam, and finally again washed. Other substances that may be used for making the initial emulsion in order to give the desired cellular structure to the C produced are MgO, Ca(OH)<sub>o</sub>, Na cresylate, soaps, etc.

A. B. MANNING.

Grinding graphite. G. HANEKOF (U.S.P. 1,814,215, 14.7.31. Appl., 31.3.28. Ger., 16.3.25).—Finely-divided graphite, suitable for use in dry-cell elements, is prepared by grinding a mixture of the graphite with rock salt or a similar sol. salt, and extracting the product with  $H_2O$ . A. B. MANNING.

Destructive hydrogenation of carbonaceous materials. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 367,776, 14.11.30).—The products of the destructive hydrogenation of coal etc. are freed from unconverted solid materials, *e.g.*, by filtering or centrifuging, after the addition of a solvent, if desired, the lower-boiling oils are removed by distillation, and the high-boiling oils are then freed from asphaltic materials by hydrogenation under pressure in the presence of catalysts containing an oxide of a metal of group VI, *e.g.*, Mo. The oils freed from asphalt are used for "pasting" fresh amounts of coal. A. B. MANNING.

Gas producer. R. DAAE (U.S.P. 1,814,787, 14.7.31. Appl., 20.5.25).—The rate of supply of fuel, steam, and air to the producer and the rate of discharge of ash therefrom are all automatically regulated by a mechanism which is actuated by fluid pressure and is controlled by the variation of gas pressure in the offtake of the producer. A. B. MANNING.

Water-gas carburettor. G. W. DAVEY and F. H. WAITE (U.S.P. 1,813,386, 7.7.31. Appl., 2.2.26).— Passages are provided in the upper half of the refractory lining of the carburettor and are used for preheating steam, which is then admitted to the top of the carburettor simultaneously with the preheated gas oil in order to prevent absorption of oil and C by the chequer-brick. Similar passages in the lower half of the lining serve to preheat the air used for the combustion of the blast gases in the carburettor. A. B. MANNING.

Carburetted water-gas sets. G. W. PARKER (B.P. 367,970, 22.12.30).—The set comprises a generator, carburettor (A), and superheater (B) combined in known manner. An exhauster is attached to the outlet of B in order to withdraw the gases from A and B and so draw the air and steam through the fuel bed. Hollow chequer-bricks are arranged within A and B in such a manner as to provide vertical passages for the gases and reduce the back-pressure to a min. The gases are passed through the system at a relatively low speed.

A. B. MANNING.

Manufacture of water-gas or producer gas with simultaneous cracking of hydrocarbon oils. PE-TROLEUM CONVERSION CORP. (B.P. 367,966, 19.12.30. U.S., 28.12.29).—Producer gas direct from the producer or water-gas heated by passage through chequer-brick preheated by the products of combustion of the airblasting operation is mixed with oil vapour which is thereby cracked. After scrubbing with the raw oil and cooling to condense the light hydrocarbons, the gas enriched by hydrocarbons not removed by the scrubbing and cooling operations is used as town's gas. D. K. MOORE.

Manufacture of oil gas. L. B. Jones (B.P. 367,812, 15.8.30).-The apparatus consists of two grates between which are two beds of chequerwork each connected to the upper part of the grate chamber next to it and communicating with each other by a passage underneath. Air is passed through one of the fuel beds, thereby heating it to incandescence, and then through both beds of chequerwork to the atm., secondary air being introduced to burn any producer gas. Steam is now passed through the hot fuel, and both the watergas produced and the oil fed in pass first through the hot chequerwork to decompose the oil and then downwards through the other fuel bed to remove C from the gas. After a time this latter fuel bed is air-blasted and the direction of flow reversed. If desired, tar removed from the gas may be returned as fuel by pumping it on to the top of the fuel bed acting as a filter. D. K. MOORE.

Manufacture of acetylene from hydrocarbons by means of an electric arc. Soc. CHEM. IND. IN BASLE (B.P. 368,164, 8.6.31. Ger., 6.6.30).—The utilisation of energy in the manufacture of  $C_2H_2$  by passing hydrocarbon vapours through an electric arc is increased by raising the c.d. of at least one of the electrodes to the point at which loss of substance from the electrode can be detected. The gases are continuously circulated and a part is continuously withdrawn from the system through one of the electrodes, used in the form of a tube. D. K. MOORE.

**Distillation of tar.** BARRETT Co., Assees. of S. P. MILLER (B.P. 367,842, 22.8.30. U.S., 30.8.29).—The hot gases from a coke-oven battery are fractionally cooled in such a manner as to condense in the collector main a heavy tar suitable for use as a road tar without further distillation, and in indirect or direct condensers one or more lighter tars. The cooling of the gases in the collector main is effected by spraying them with gas liquor and/or tar, or by passing a stream of the cooling medium along the bottom of the main. The lighter tars are distilled in a pipe-coil or other type of still, preferably by utilising the heat of the gases from selected ovens of the battery, and tar acids,  $C_{10}H_8$ , etc. are recovered from the products. A. B. MANNING.

Apparatus for treating, distilling, and decomposing hydrocarbon oils. H. T. DARLINGTON, ASST. to M. B. SCHUSTER (U.S.P. 1,813,117, 7.7.31. Appl., 24.6.29).—The apparatus consists of a horizontal drum with a reflux tower attached to the top and a tube extending downwards attached to the bottom. From this tube upper and lower banks of heating pipes extend outwards to a vertical chamber. Oil is fed into the tube either at the bottom or about halfway up and is circulated through the lower heating pipes, the vertical chamber, and the upper heating pipes, and is heated to cracking temp. The lighter products rise from the vertical tube into the drum and the vapours pass into the reflux tower. D. K. MOORE.

410

Apparatus for hydrocarbon oil conversion. O. H. FAIRCHILD, ASST. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,811,621, 23.6.31. Appl., 10.10.27).—A cracking heater consists of vertical tubes of refractory material, *e.g.*, carborundum, mounted in a furnace. Deposits of C are removed by periodically passing air through the heated tubes. D. K. MOORE.

Apparatus for conversion [cracking] and transformation of hydrocarbons, liquids, and fluids. J. H. ADAMS, Assr. to TEXAS Co. (U.S.P. 1,811,834, 30.6.31. Appl., 26.1.16. Renewed 12.1.29).-Preheated oil is pumped through a converter consisting of a nest of horizontal tubes, the upper ones being staggered in respect to the lower ones. The oil passes to a trap to remove C and thence on to a horizontal plate in a separator, and, rising, overflows upstand pipes into the lower part of the separator, from which it is withdrawn, cooled to below 149-225°, and returned to the oil-feed tank. Alternatively, the separator may be fitted with a vessel so that the oil may be fed into the latter either at the top or through an uptake pipe at the bottom, the oil overflowing both the sides of the vessel and upstand pipes into the lower part of the separator. The vapours from the separator are condensed. The apparatus is operated under pressure. D. K. MOORE.

[Hydrocarbon oil-]cracking still. W. T. HAN-COCK (U.S.P. 1,814,249, 14.7.31. Appl., 21.8.29).—Oil is passed under pressure through one or more externally heated tubes within which metal balls are confined between a series of discs mounted at angles on a longitudinal and rotatable shaft. Liquid and vapours are discharged into a reaction chamber so inclined that solid C ppts. at one end and can be withdrawn.

H. S. GARLICK.

Cracking of hydrocarbons. E. C. HERTHEL, Assr. to SINCLAIR REFINING CO. (U.S.P. 1,810,048, 16.6.31. Appl., 7.3.29).—Heavy oil is pumped into the lower part of a tower fitted with baffles, and through it bubbles vapours from a coking vessel A. The oil is then passed into A, in which it comes in contact with hot vapours from a cracking coil, and these coke the oil. D. K. MOORE.

Cracking of hydrocarbons and treatment of residues. E. W. ISOM, ASST. to SINCLAIR REFINING Co. (U.S.P. 1,811,272, 23.6.31. Appl., 30.6.24).—The rates of tar withdrawal and supply of fresh oil to a pressure still are regulated to maintain the charge therein below saturation with tarry constituents, so that when the tar is withdrawn and treated with finely-divided absorbent material it can be separated from the oil (which is returned to the still) by centrifugal or other means. H. S. GARLICK.

Cracking of hydrocarbon oils. E. W. Isom, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,814,700, 14.7.31. Appl., 9.4.27).—Oil heated to 430—650° is passed into an expansion chamber which may be heated externally. The vapours from this are refluxed with fresh heavy oil which has been heated by vapours leaving the reflux tower. The light condensate produced by this heating, and fresh light oil with or without a part of the heavier reflux, are passed to the heater. The reflux is returned wholly or in part to the expansion chamber.

D. K. MOORE.

Treatment [cracking] of petroleum oil. C. P. DUBBS, ASST. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,811,617, 23.6.31. Appl., 31.7.22).—Cracked oil from a furnace flows through four vessels arranged in series with vapour and liquid connexions. The first and second vessels may be heated by burners. Liquid may be withdrawn directly from any of the vessels or returned through a common pipe to the raw-oil storage tank.

D. K. MOORE.

Conversion [cracking] of hydrocarbon oils. J. C. MORRELL and H. P. BENNER, ASSTS. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,811,642, 23.6.31. Appl., 21.4.23).—The vapours from a cracked oil pass into the bottom of an air-cooled dephlegmator the reflux from which passes through a cooler and down a second dephlegmator up which the vapours from the top of the first dephlegmator pass. D. K. MOORE.

Treatment [cracking] of hydrocarbon oil. D. PYZEL, ASST. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,811,646, 23.6.31. Appl., 29.6.27).—In the cracking treatment, the oil is fed from a heater into a vertical cylinder fitted with an agitator, the time of reaction being controlled by the depth of oil in the vessel.

D. K. MOORE.

Decomposition of organic [hydrocarbon] materials. R. J. DEARBORN and G. W. GRAY, Assrs. to TEXAS Co. (U.S.P. 1,814,621, 14.7.31. Appl., 20.4.26). -The material in liquid form is passed into the first of a series of interconnected, heat-insulated converting vessels to form a substantial depth of liquid in each vessel, and is maintained under conditions of pressure and temp.  $(> 149^{\circ})$  suitable for active decomp. The generated vapours are removed in heat-exchange relationship with the fresh charge and the less volatile constituents are condensed therefrom, raised to a cracking temp. without substantial decomp., and continuously introduced into the first vessel for further treatment, while withdrawing residual material from the last vessel. In order to compensate for heat losses and maintain a substantially const. predetermined decomp. temp., a gas containing free O2 is introduced into the lower part of each vessel. H. S. GARLICK.

Production of low-b.p. hydrocarbon oils. C. P. DUBES, ASST. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,811,308—9, 23.6.31. Appl., [A] 5.5.23, [B] 21.3.30).— Oil is cracked in heating and reaction zones and the vapours are dephlegmated by intimately mixing them with the incoming new oil in a series of pools, the temp. of which can be regulated to control the amount of condensation therein by locally circulating the contents through extraneous (A) heating, or (B) cooling, means. H. S. GARLICK.

Production or recovery of lighter hydrocarbons. A. CLARKE and H. BUTLER, Assrs. to UNITED HYDRO CARBONS CO. (U.S.P. 1,813,024, 7.7.31. Appl., 7.8.26).— Gas containing light hydrocarbons, *e.g.*, natural gas containing gasoline, is scrubbed with oil and the latter subjected to dry distillation and then to distillation in steam, the stripped oil being returned to the scrubber. The vapours from the fire still which is operated under vac. are compressed, cooled, and passed into a tank A. The vapours from the steam-distillation are cooled and the product is passed into a tank B in which  $H_2O$  is separated. The liquid in B may be withdrawn from the system or pumped directly, or the vapours obtained from it by distillation pumped into the vapour stream from the fire still, into which stream the vapours from B are also passed. The vapours from A are further compressed and cooled and passed into tank C, the vapours from which are recirculated to the fire-still vapour stream. The liquids from A and C are passed into another vessel where, on reduction of pressure, some vaporisation takes place and the vapours are recirculated. The liquid, being the desired hydrocarbons, is withdrawn from the system. Vent valves permit the escape of any excessive quantity of recirculation gases. D. K. MOORE.

Production of motor fuel or gasoline from petroleum oil. M. L. CHAPPELL, ASST. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,811,257, 23.6.31. Appl., 22.1.27).—The vapours from the topping of crude petroleum are dephlegmated to produce a gasoline-b.p. fraction as vapour, and a kerosene fraction as condensate which is vaporised, those fractions having relatively high b.p. within the gasoline range being separated therefrom. The remainder is subjected to reaction with a metallic halide under conditions of temp. and pressure which effect decomp. with the formation of sufficient fractions of relatively low b.p. within the gasoline range to give specification gasoline when blended with the two fractions previously mentioned. H. S. GARLICK.

Manufacture of hydrocarbon oil. G. H. B. DAVIS, Assr. to Standard Oil Development Co. (U.S.P. 1,815,022, 14.7.31. Appl., 3.5.30).-An aliphatic hydrocarbon, e.g., paraffin wax, is chlorinated at 60-150°. Then 1 pt. of the product (containing 10-12% CI) is added to 0.1-0.5 pt. of aromatic material, e.g., C6H6, C10H8, anthracene, aromatic gas oil, and the whole mixed with an equal wt. of AlCl<sub>3</sub> at 60-71° in the presence of a predominately saturated hydrocarbon solvent, e.g., kerosene. After 24 hr. the temp. is raised to 93°. After settling, the oil is cooled and unchanged paraffin removed by cold-pressing or vac. distillation. 1-10% of the product added to a wax-bearing oil reduces its pour point; 10-50% also reduces the change of viscosity of the oil with temp. D. K. MOORE.

Refining of hydrocarbon oils with metallic halide. J. H. OSMER, ASST. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,811,243, 23.6.31. Appl., 25.2.27). —Lubricating or other high-b.p. hydrocarbon oils are diluted with a previously purified low-b.p. oil and brought into intimate contact with a metallic halide at 93—121°. The reaction products and unchanged metallic halide are removed, and the light and heavy oils separated by distillation. H. S. GARLICK.

Refining of hydrocarbon oils. A. G. PETERKIN, JUN., Assr. to ATLANTIC REFINING CO. (U.S.P. 1,810,369, 16.6.31. Appl., 4.12.28).—Mercaptans are removed by passing crude oil, a refining agent, e.g., aq. CuCl<sub>2</sub>, aq. MgCl<sub>2</sub>, Pb compounds in aq. NaOH, and a stream of gas containing  $O_2$  to revivify the refining agent *in situ*, into a reaction chamber and thence into a settler from which the oil is withdrawn. The spent gas is withdrawn from the top of the settler and  $O_2$  added to it, and this

and the refining agent from the bottom are recirculated. Alternatively, the reagent may be revivified in a separate chamber. D. K. Moore.

**Refining of [hydrocarbon] oils.** W. T. BRYANT (U.S.P. 1,812,923, 7.7.31. Appl., 30.8.27).—Oil, after acid treatment, sludging, and neutralisation, is heated to  $> 100^{\circ}$  and below its flash point and agitated with clay. After filtration, moisture is removed by submitting the hot oil to the action of a vac. D. K. MOORE.

Refining of [hydrocarbon] oils. M. STUART and L. M. STUART (B.P. 367,848, 19.11.30 and 12.8.31).— Hydrocarbon oil fractions are treated with ozonised air to oxidise S compounds and those unsaturated compounds responsible for the formation of gums and resins, without oxidising free S and free C; such treatment may be either alone or combined with, or followed by, further treatment with a catalyst, condensing agent, or contact substance, the oil being subsequently washed with an aq. alkaline solution and redistilled. [Stat. ref.] H. S. GARLICK.

[Petroleum] oil refining. D. PYZEL, ASST. to SHELL DEVELOPMENT Co. (U.S.P. 1,811,645, 23.6.31. Appl., 27.12.20. Renewed 22.10.29).—Oil vapours are condensed by passing into a stream of  $H_2O$  and then separating the oil from the  $H_2O$  in settling tanks. By keeping the vapours free from heavy hydrocarbons the formation of emulsions is avoided. D. K. MOORE.

Improving pressure-distilled [hydrocarbon] oil products. V. VOORHEES and E. J. SHAEFFER, ASSTS. to STANDARD OIL CO. (U.S.P. 1,810,632, 16.6.31. Appl., 7.12.25).—The colour of the oil is stabilised by treatment with 10—70%  $H_2SO_4$  (concn. preferably 25%) before sweetening. If the acid is subsequently neutralised and steam-distilled a dark-coloured product is obtained which, on exposure to air, resinifies. D. K. MOORE.

Treatment [desulphurisation etc.] of hydrocarbon oils. J. C. BLACK and M. L. CHAPPELL (U.S.P. 1,810,068, 16.6.31. Appl., 31.1.28).—Unstable hydrocarbons and S compounds are removed from hydrocarbon oils, e.g., gasoline or naphtha, by mixing with liquified gas produced by cracking processes, consisting of  $C_3H_8$ ,  $C_4H_{10}$ , olefines ( $C_2H_4$ ,  $C_3H_6$ ), etc., and treating with  $H_2SO_4$  of at least 76% concn. at about —7°.

D. K. MOORE.

Sweetening of petroleum products. H. H. CANNON (B.P. 367,969, 22.12.30).—A controlled proportion of S is introduced into a stream of sour oil, which is then brought into intimate contact with a reagent containing an alkali hydroxide, Pb in a form capable of forming salts with alkali or alkaline-earth hydroxides, an alkalineearth hydroxide, and sufficient inert earthy material to reduce the reagent to an incoherent powder. The reagent is mechanically separated from the purified oil and continuously returned to the stream of unsweetened oil. H. S. GARLICK.

Treatment of petroleum emulsions. J. HYMAN and A. F. SCHLANDT, ASSTS. to PURE OIL CO. (U.S.P. 1,811,177, 23.6.31. Appl., 26.3.29).—Motor fuel produced by vapour-phase cracking and containing a large quantity of unsaturated compounds is oxidised and the product used to cut emulsions of petroleum with H<sub>2</sub>O or brine. D. K. MOORE.

Demulsification of petroleum. C. P. DENNHARDT (U.S.P. 1,814,298, 14.7.31. Appl., 6.9.27).—Oil emulsion is supplied to a treating chamber through a passage in which is positioned means for squeezing the emulsion as it passes therethrough, and opposite the mouth of which is a nozzle through which a liquid reagent can be sprayed. H. S. GARLICK.

Decolorisation of hydrocarbon oils. H. LOWERY and R. S. LANE, Assrs. to STANDARD OIL CO. (U.S.P. 1,813,628, 7.7.31. Appl., 27.1.27).—After  $H_2SO_4$  treatment, hydrocarbon oil, especially lubricating oil, is decolorised by mixing with 'a diluent, *e.g.*, furnace oil, and clay and heating to 290—480° in a pipestill from which the suspension is discharged into a separator from which the diluent and light products pass as vapour. The clay is removed from the residue after cooling to 120—200° by filter-pressing.

# D. K. MOORE.

**Decolorisation of [hydrocarbon] oils.** R. C. OSTERSTROM, ASST. to PURE OIL CO. (U.S.P. 1,811,185, 23.6.31. Appl., 18.6.28).—A mixture of oil and a suitable clay, *e.g.*, fuller's earth, is heated to 66—260° under 40—750 lb. per sq. in. pressure and then passed into a no. of chambers in series, with reduction in pressure, and thence into a column at atm. pressure. The lighter products distil, while the liquid flows through a screen at the bottom (which separates the greater part of the clay) into a settler in which the rest of the clay and tar are separated from the oil. D. K. MOORE.

Purification of petroleum products. H. G. SMITH, Assr. to GULF REFINING Co. (U.S.P. 1,813,642, 7.7.31. Appl., 16.6.26).—Petrolatum is agitated several times, usually six, with 10—20 wt.-% of AlCl<sub>3</sub> at 60— 120°, being allowed to settle (and the sediment removed) between each addition. The oil is washed with 5%  $H_2SO_4$  at 93°, then with hot  $H_2O$ , dried by air-blowing, and filtered through fuller's earth. Light petroleum products are removed by heating and the oil is filtered through clay, yielding a white product the m.p. of which may be adjusted by the addition of white oil. White oil is produced by this process, except that after the drying the oil is treated with oleum at 38°, washed, dried, and then the above treatment continued.

#### D. K. MOORE.

Treatment of hydrocarbon compounds. W. E. ELLIOTT, Assr. to BYPRO MANUFG. Co. (U.S.P. 1,814,745, 14.7.31. Appl., 28.7.27).— $C_{10}H_8$  (70 lb.) is brought into intimate contact with the atm., screened to 18—22mesh, and then mixed with 1 gal. of PhNO<sub>2</sub>. Medicinal paraffin (3 pints) and "oil-soluble red" (NH<sub>2</sub>Ph 2½ oz.) are then mixed in. After keeping for at least 24 hr., the compound, usually 1 lb. per 800—1000 gals., is added to fuel oil, petroleum emulsions, sludges, etc., and the mixture heated to about 50°. The emulsion is broken down, heavy hydrocarbons are deflocculated, and homogeneous oil separates. D. K. MOORE.

(A-D) Treatment of mineral oil material. (E) Production of oil-soluble material [from petroleum]. H. BEHM (U.S.P. 1,813,882-6, 7.7.31. Appl., [A] 14.7.22, [B] 8.5.23, [C] 27.5.25, [D] 17.1.28, [E] 16.1.28. Renewed [A] 3.6.31, [C] 20.10.28).-(A) Petroleum oils, e.g., kerosene, are exposed to aëration (with consequent fermentation) in the presence of a small % of  $H_2O$  and EtOH and traces of CaO, CH2O, and aq. NH3, the temp. being maintained at  $15 \cdot 5 - 40 \cdot 5^{\circ}$ . Turpentine is added as an O2 carrier. (B) An aq. solution containing a nitrate and the reaction products of I and NH<sub>3</sub> are added to the mineral oil mixture in (A). (c) A " starter " for the above processes is produced in two separate parts : the first (i) is made by mixing a large bulk of mineral oil material with smaller quantities of slaked CaO, rosin, aq. NH<sub>3</sub>, and CH<sub>2</sub>O, and keeping for several hr.; the second (ii) by mixing EtOH, COMe2, tincture of I, and orange peel, and adding them to a mixture of (i) and (ii). Chlorinated CaO is added, insol. material is removed, and turpentine added. (D) The reaction products of  $H_3BO_3$ , EtOH, NaOH, chlorinated CaO, H<sub>3</sub>PO<sub>4</sub>, and ground orange material are suspended in mineral oil, the mixture is acidified, and, after a while, filtered and over-neutralised, whereby a gelatinous material is pptd. (E) A product containing the oil-sol. reaction products of a petroleum product not substantially heavier than kerosene, CaO, rosin, aq. NH<sub>3</sub>, CH<sub>2</sub>O, chlorinated CaO, COMe<sub>2</sub>, and AcOH is claimed. Various applications of the products and by-products are indicated. (Cf. U.S.P. 1,541,242-3; B., 1925, 749.) H. S. GARLICK.

Manufacture of lubricating oil. W. T. BRYANT, Assr. to W. A. WILLIAMS (U.S.P. 1,812,924, 7.7.31. Appl., 30.8.27).—A small quantity of dil. caustic alkali solution is added to a charge of oil in a still, the lighter fractions are distilled off, and distillation is continued until substantially 5% of bottoms remain. The lubricating oil fraction is treated with acid of < 98% concn., the sludge removed, and the remaining oil heated to reaction temp. with a decolorising agent, continuously filtered, and the filtered distillate while still hot subjected to a vac. treatment. H. S. GARLICK.

Emulsifiable lubricant. E. W. ADAMS and F. W. KITTRELL, ASSTS. to STANDARD OIL CO. (U.S.P. 1,817,599, 4.8.31. Appl., 4.9.28).—Tallow (e.g., 5—18%) is emulsified with 20—40% of alkali soaps of oxidised mineral oil (oxidised, e.g., by blowing with air at 150—160°), with or without addition of mahogany soap stock (20—40%) and mineral oils (1—15%).

E. LEWKOWITSCH.

Treatment of petrolatum stocks. B. J. SOUTHER and W. A. GRUSE, Assrs. to GULF REFINING CO. (U.S.P. 1,813,327, 7.7.31. Appl., 5.6.28).—Petrolatum, freed from naphtha used in the cold-settling process by steaming, is dissolved in 4—6 pts. of BuOH at 80°. When the solution is cooled to 60° resin and asphaltic material separate and are removed. The solution is then cooled to room temp. and a large part of the wax separates and is removed. A further yield of wax is obtained by cooling the mother-liquor to  $-10^{\circ}$  to  $-18^{\circ}$ . The final mother-liquor on distillation yields a viscous oil of low cold and low C-residue tests. The addition of COMe<sub>2</sub> before the last cooling yields a purer wax and an oil of lower cold-pour test. D. K. MOORE.

Purification of crude paraffin wax. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 367,939, 4.12.30. Addn. to B.P. 320,921; B., 1930, 134).—Purification is conducted in the presence of a catalyst containing

Bi and/or V, in addition to Fe, Ni, or Co, or any combination of these three metals, with a further addition of substances containing metals of groups III, IV, and VI.

H. S. GARLICK.

Burning of pulverised fuel. W. Rogers (B.P. 369,340, 14.2.31).

Liquid-fuel burning plant. J. G. McKEAN and R. F. JONES (B.P. 369,224, 16.12.30).

Apparatus for mixing gas and air, in particular for gas firing. O. KLATTE (B.P. 368,810, 5.12.30).

Doors of gas-generating and coke ovens. H. J. LIMBERG (B.P. 369,121, 24.7.31. Ger., 29.10.30).

Separation of materials [dust from coal]. Effecting reactions between gases. Separation of substances from gases. Preventing rusting in gasometers.—See I. Petroleum-sec. alcohol mixtures.—See III. Bleaching of oils.—See VII. Asphaltic compositions. Waterproof products. Compositions for roads etc. Floor coverings.— See IX. Determining paraffin in fibrous materials. —See XI. Detergent. Wax compositions.—See XII. Coating containers for oils.—See XIII.

# **III.**—ORGANIC INTERMEDIATES.

Preparation of guanidine salts from ammonium thiocyanate. W. GLUUD, K. KELLER, and W. SCHULTZE (Ber. Ges. Kohlentech., 1932, 4, 21-33).-Guanidine forms well-cryst. salts which have up to the present found only limited applications, but aromatic derivatives (e.g., diphenylguanidine) are largely used as vulcanisation accelerators. If NH<sub>4</sub>CNS is heated at 200° the following reactions occur:  $2NH_4CNS = NH:C(NH_2)_2,HCNS + H_2S$ ;  $H_2S + NH_4CNS \equiv CS_2 + 2NH_3$ . The yield of guanidine thiocyanate is improved by introduction of NH<sub>3</sub>, which reverses the subsidiary reaction, and on a laboratory scale may be 80-85%, the product containing some thiourea. Larger-scale tests were made in porcelain vessels with Fe lids covered with bakelite. Difficulty was caused by the blocking of the exit gasline with (NH<sub>4</sub>)<sub>2</sub>CS<sub>3</sub>; this was avoided by omitting the circulation of NH<sub>3</sub> and absorbing the whole of the exit gases in H<sub>2</sub>O. Free CS<sub>2</sub> separates and the whole is reconverted into NH4CNS by autoclaving at 100°. The crude guanidine salt was purified by dissolving in hot H<sub>2</sub>O, warming to 100°, treating with animal charcoal, cooling, filtering off the recryst. salt, and drying at 50-60°. The thiocyanate can be converted into the sulphate by distilling with H<sub>2</sub>SO<sub>4</sub> and neutralising with CaO, and this in turn can be converted into other salts. The products are purified with animal charcoal, the yields being 75-90% of the theoretical.

C. IRWIN.

2:4-Dinitrophenol. Manufacture, properties, and uses. L. DESVERGNES (Chim. et Ind., 1931, 26, 507— 520, 1271—1281; 1932, 27, 278—292, 527—544).—The manufacture of 2:4-dinitrophenol, m.p. 112.5° [113° (block)], its application in the chemical and explosives industries, and its toxicology are described. The processes described include the nitration of PhCl in 2 stages to 1-chloro-2:4-dinitrobenzene, m.p.  $50.1^{\circ}$ , and alkaline hydrolysis of this, and the nitration of PhOH, including its nitration to a mixture of dinitrophenol and picric acid. Additive compounds (salts ?) of pyridine with 2 : 6-, m.p. 44—45°, and 2 : 4-dinitrophenol, m.p.  $71 \cdot 5°$ , and 2 : 4-dinitrophenyl Et, m.p. 84 · 8°, Pr and isoamyl (liquid), allyl, m.p. 86—87°,  $\beta$ -glyceryl, m.p. 83°, ethers, and 2 : 4-dinitrodiphenyl ether, m.p. 71°, are described. A method of separating 2 : 6- and 2 : 4-dinitrophenols, based on the different solubilities of their K salts in H<sub>2</sub>O, is given. H. A. PIGGOTT.

Summary of patents dealing with the syntheses of methyl alcohol. B. LÖPMANN and B. JAECKEL (Ber. Ges. Kohlentech., 1932, 4, 1–20).

Phenols from tar oils.—See II. Synthetic resins from guanidine.—See XIII.

See also A., April, 326, Catalyst for hydrogenation of PhOH. 364, Prep. of Et, pyrophosphite. 368, Prep. of pinacolone. 371, Preps. of diacetoneamine and substituted glyoxalines. 375, Reagent for purifying amines. 383, Synthesis of araldehydes. Preps of 385, cyclopentanone and glutaric acid : 386, substituted fluorenones ; 396, benzoin anilides, o-phenylenedi(phenylglyoxal), and anthraquinonesulphonic acids ; 400. dibromofluorescein. 401. Electrolysis of pyridine. Preps. of, 402, quinoline-2aldehyde and derivatives, 404, phthalazine and derivatives. 411, Determining CH2O, saligenin, salicylic acid, and salicylaldehyde. 428, Biological conversion of CO and CO2 into CH4. 429, Conversion of lactic into pyruvic acid. Production of trimethylene glycol.

#### PATENTS.

Stable mixture of petroleum hydrocarbons and secondary alcohols. M. D. MANN, JUN., Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,811,552, 23.6.31. Appl., 9.8.29).—A solvent, paint or varnish thinner, cleaning and degreasing agent, motor fuel, etc. consists of a mixture of liquid petroleum hydrocarbon, e.g., gasoline, with a sec. alcohol containing 3—6 C atoms. H. S. GARLICK.

Treatment and packaging of ether. W. G. CHRIS-TIANSEN and R. VAN WINKLE, Assrs. to E. R. SQUIBB & Sons (U.S.P. 1,813,664, 7.7.31. Appl., 15.2.30).—  $Et_2O$  is treated or stored with  $Cu_2O$  (<1% by wt.) or other Cu' compound. E. H. SHARPLES,

**Purification of methyl salicylate.** M. E. PUTNAM, Assr. to Dow CHEM. Co. (U.S.P. 1,814,853, 14.7.31. Appl., 13.1.30).—The impure salicylate is cooled below —1° and then seeded with the modification of the same compound having m.p. — $1\cdot0^\circ$ . Diluents may be added and cooling continued after the initial crystallisation.

E. H. SHARPLES.

Preparation of alginic acid and compounds thereof. F. C. THORNLEY and M. J. WALSH, ASSIS. to KELCO CO. (U.S.P. 1,814,981, 14.7.31. Appl., 6.8.27).— Fibrous Ca alginate, prepared by running Na alginate solution (prepared from kelp) into CaCl<sub>2</sub> solution,  $p_{\rm H}$ 11·5—10·0, with violent agitation and under conditions such that the  $p_{\rm H}$  is < 7.5 at any time, is washed free from NaCl, bleached, if desired, with NaOCl, and converted by treatment with dil. HCl into fibrous alginic acid, which is removed and washed with dil. HCl and finally with H<sub>2</sub>O. The acid may be agitated in the presence of H<sub>2</sub>O with metal oxides or carbonates (e.g., CuO, MgO, ZnCO<sub>3</sub>, Na<sub>2</sub>CO<sub>3</sub>) to yield alginates which may then be treated with aq. NH<sub>3</sub> to form, *e.g.*, Cu NH<sub>4</sub> or Zn NH<sub>4</sub> alginate. The viscosity of Na alginate solutions is decreased (a) by storing the kelp before treatment, (b) by prolonging the acid treatment of the Ca alginate, (c) by performing the acid treatment at a raised temp.  $(>50^{\circ})$ . L. A. COLES.

Alginates in finely-divided form derived by spray-drying dilute solutions thereof. M. J. WALSH, Assr. to KELCO CO. (U.S.P. 1,814,986, 14.7.31. Appl., 9.4.28).—Alginic acid in 3-5% solution is dried by spraying into air at  $150-320^{\circ}$ ; the product dissolves in H<sub>2</sub>O to yield a solution having all the properties of the original solution, Na alginate and NH<sub>4</sub> alginate solutions are dried by spraying into hot air and into hot gases containing NH<sub>3</sub>, respectively. L. A. COLES.

Manufacture of sulphonation products [cleaning, wetting, and emulsifying agents, etc.]. Soc. CHEM. IND. IN BASLE (B.P. 363,770, 29.10.30. Switz., 29.10.29). —An unsaturated hydroxy-fatty acid above  $C_8$ , or a derivative, is sulphonated in presence of a sulphonated aromatic acid or anhydride, *e.g.*, castor oil with  $\beta$ -sulphophthalic acid or anhydride. C. HOLLINS.

Cans or like containers for ether. MALLINCKRODT CHEM. WORKS (B.P. 368,816, 8.12.30. U.S., 20.12.29).— See U.S.P. 1,807,598 ; B., 1932, 172.

Effecting reactions between gases. Treatment of catalysts.—See I.  $C_2H_2$  from hydrocarbons. Distillation of tar.—See II. Glycerin. Synthetic drying oil.—See XII. EtOH.—See XVIII. Explosives.—See XXII.

# IV.—DYESTUFFS.

See A., April, 332, Adsorption of dyes by acid clay. 400, Anthraquinone-2:1:6:5-dixanthone.

# PATENT.

Colour lakes .--- See XIII.

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

Effect on wool of temperature and hydrogen-ion concentration of the scouring bath. G. M. BARMORE (Rensselaer Polytech. Inst. Bull., 1932, No. 35, 10-20). —Immersion of wool in distilled H<sub>2</sub>O at 20-80° has no effect on the tensile strength of the material after redrying. For scouring baths (aq. NaHCO<sub>3</sub>, Na<sub>2</sub>CO<sub>3</sub>, or NaOH) at the same temp. the tensile strength of the material decreases slowly with increasing  $p_{\rm H}$  from  $p_{\rm H}$  7 onwards until finally a very rapid decrease occurs. For baths of the same  $p_{\rm H}$ , the average rate of decrease of strength is doubled for every 13-14° rise in temp. Decoloration of the wool is also a function of  $p_{\rm H}$ ; it is very slight between  $p_{\rm H}$  7 and 9.5, but for more alkaline baths it increases more rapidly. The max.  $p_{\rm H}$  range for clean wool scoured for 4-5 min. is given at 50°.

B. P. RIDGE.

Behaviour of wool fibres towards chromic acid and dichromates. R. HALLER and H. VOM HOVE (Helv. Chim. Acta, 1932, 15, 357-375).—Wool placed in CrO<sub>3</sub> or K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solutions and dried develops a dark brown colour in sunlight because of reduction of

Cr<sup>VI</sup> to chromi-chromates, and finally a green colour due to formation of CrIII salts; reduction (and consequent discoloration) takes place slowly in the dark in H<sub>2</sub>O, somewhat more rapidly in dil. H<sub>2</sub>SO<sub>4</sub>. In sunlight reduction takes place at the following relative rates in the solutions (0.1N) named: (a) using  $CrO_2$ :  $H_2O 6.7$ , AcOH 5.2, HNO<sub>3</sub> 12.5,  $H_2SO_4 15.3$ ,  $H_2C_2O_4$ 85.0; (b) using  $K_2Cr_2O_7$ :  $H_2O$  2.5, AcOH 4.4,  $HNO_3$ 62.6,  $H_2SO_4$  63.5,  $H_2C_2O_4$  at least 100. Complex formation is considered to take place in the acid solutions. Addition of Na<sub>2</sub>SO<sub>4</sub> to the H<sub>2</sub>SO<sub>4</sub> solutions decreases the rate of reduction owing to the more rapid diffusion of CrO<sub>3</sub> from the fibre and consequently its lesser concn. thereon. Damage to the fibre during the reduction is shown by the presence of SO4" and proteins (ninhydrin and biuret tests positive, xanthoprotein and Millon's tests negative) in the solutions and by a decrease of tenacity of the fibre, the latter being approx. parallel to the degree of reduction. R. S. CAHN.

Effect of alkali on degumming of silk. I—III. I. TOYODA (J. Soc. Chem. Ind., Japan, 1932, 35, 43—44 B).—In degumming with Na oleate, stearate, or palmitate, the alkalinity of the bath diminishes as degumming proceeds, whereas the relative viscosity increases for about the first 30 min., then falls continuously, possibly in consequence of the peptisation and decomp. of the sericin. With increase in the amount of soap above about 15% of the wt. of the silk the loss in wt. on degumming tends to fall. R. CUTHILL.

Action of alkaline copper solution on silk fibroin. I. I. FUKUSHIMA and Y. TAKAMATSU (J. Soc. Chem. Ind., Japan, 1932, 35, 14-16 B).-The solubility of fibroin in a saturated solution of Cu(OH)<sub>2</sub> in aq. NH<sub>3</sub> or aq. ethylenediamine is increased if excess of Cu(OH), is added as a second solid phase, and the dissolution of the fibroin leads to the dissolution of more Cu(OH)<sub>2</sub>. The mutually increased solubility is attributed to the formation of a compound containing Cu and fibroin, and addition of EtOH to the ethylenediamine solution causes the pptn. of a substance believed to be a compound containing fibroin, Cu, and ethylenediamine in the mol. ratio 1:2:2. The max. increase in solubility of Cu(OH)2 in aq. ethylenediamine caused by fibroin corresponds with 1 mol. of Cu per 227 g. of fibroin dissolved. R. CUTHILL.

Liberation of ligninsulphonic acid from sulphited cellulose. C. KULLGREN (Svensk Kem. Tidskr., 1932, 44, 15-23; cf. B., 1930, 984; 1931, 670).-The permutit-like properties of ligninsulphonic acid were applied to its determination in sulphited celluloses. Cellulose was washed with  $H_2O$  followed by 0.2N-HCl to remove metallic ions and EtOH to remove resins. The final material was extracted with various concns. of HCl or H<sub>2</sub>SO<sub>4</sub> alone or in presence of NaCl or Na<sub>2</sub>SO<sub>4</sub> at 78°, the amount of acid extracted and the acid val. and OMe content of the residue being determined. The liberation of the acid was independent of the [H] of the extracting medium, but dependent on the extent of salt formation by the ligninsulphonic acid. Thus with  $H_2O$  or abs. EtOH at 78° for prolonged periods the liberation was fairly extensive, but when preceded

by treatment of the cellulose with NaCl it was negligible. Extraction with EtOH effected a partial esterification of  $CO_2H$ , but not  $SO_3H$ , groups. The extracted sulphonic acid was obtained as a partly cryst. residue on evaporating the extraction liquors. It did not reduce Fehling's solution either before or after hydrolysis and was optically inactive. The acid extracted by  $H_2O$ , approx.  $C_{26}H_{32}O_{13}S$ , contained two OMe groups, whilst that extracted by MeOH had a higher % of OMe. Moderate heating gave a black modification sol. in  $H_2O$ , a phenomenon not so prominent with the Na salt. The acid gave positive fuchsin- $SO_2$  and phloroglucinol tests and a violet fluorescence in ultra-violet light. F. O. HowITT.

Viscosity of cellulose esters. Acetylcellulose. I. SAKURADA (J. Soc. Chem. Ind., Japan, 1932, 35, 78—79 B).—It is incorrect to substitute infinity for c in the formula derived from Baker's equation (cf. B., 1931, 479), since c lies between 0 and 100d, where d is the sp. gr. of the substance. Further, it is not legitimate to calculate the viscosity of colloidal solutions, such as those of nitrocellulose, in terms of the viscosity of a normal substance such as glycerol. Atsuki and Ishii's paper on acetylcellulose (B., 1931, 1087) is adversely criticised. H. F. GILLBE.

[Viscosity of cellulose esters.] K. ATSUKI, N. ISHII, and S. ISHIWARA (J. Soc. Chem. Ind., Japan, 1932, 35, 79-80 B).—A reply to Sakurada (cf. preceding abstract). H. F. GILLBE.

Production of paper from deciduous wood. I. KOVALEVSKI and V. KHATZOV (Bumazhnaya Prom., 1931, 10, No. 9, 35-40).—Aspen waste from match manufacture is pulped by combined chemical and mechanical treatment in the cold. Experiments with abietic wood were less successful; birch and alder produced paper similar to, but darker than, that from aspen. CHEMICAL ABSTRACTS.

Cellulose in flour.-See XIX.

See also A., April, 370, Ethylcellulose. Cellulose xanthate. 415, Structure of wool and hair. 429, Aërobic pectin fermentation.

# PATENTS.

Extraction of fibre from vegetable matter. G. F. T. HOLLOWAY (B.P. 367,116, 12.1.31).—The fibrous material (hemp, sisal, hibiscus, etc.) is crushed between rollers, treated for  $\frac{1}{4}$ —2 hr. with boiling dil. Na<sub>2</sub>CO<sub>3</sub>, washed, again crushed and/or steeped for a few min. in 0.25% H<sub>2</sub>SO<sub>4</sub>, washed, and dried.

B. P. RIDGE.

Waterproof fibrous products. H. D. ELKINGTON. From FLINTKOTE Co. (B.P. 366,521, 30.10.30).—An aq. dispersion containing  $\gg 35$  dry wt.-% of feltable fibrous stock, an approx. equal wt. of non-feltable fillers, and a suitable amount of a waterproof thermoplastic substance (asphalt, pitch, tar, resin) is made into sheets or plates, which are dried and moulded under heat and pressure. F. R. ENNOS.

Cleansing of cellulose pulp. ZELLSTOFF-FABR. WALDHOF, H. MÜLLER-CLEMM, and W. LEUPOLD (B.P. 368,012, 23.1.31. Ger., 27.1.30).—The digested pulp is thoroughly mixed by means of air or steam with washing  $H_2O$  added intermittently or continuously, preferably on the countercurrent principle, after or during which  $H_2O$  is introduced into the container at the bottom and the waste  $H_2O$  containing sol. and insol. impurities is led off above the cellulose material through an overflow. F. R. ENNOS.

Manufacture of cellulose esters. Soc. CHEM. IND. IN BASLE (B.P. 367,312, 6.10.31. Switz., 17.10.30. Addn. to B.P. 349,322; B., 1931, 714).—Ripened viscose is spun into a bath containing 40% of KOAc and 20% of AcOH and the dried threads are acylated by treatment with Ac<sub>2</sub>O or (PrCO)<sub>2</sub>O, thus giving material of improved stability to boiling H<sub>2</sub>O and to ironing, and, in the case of the butyrate, enhanced resistance to alkalis. B. P. RIDGE.

Manufacture of new cellulose derivatives. Soc. CHEM. IND. IN BASLE (B.P. 363,897, 20.2.31. Switz., 21.2.30. Addn. to B.P. 342,167; B., 1931, 343).-Condensation products from cellulose and cyanuric halides etc., still containing reactive halogen, are further condensed with compounds having reactive H and at least one chromophore or group capable of dye-formation. Cellulose is thus condensed with cyanuric chloride and then with: 4:4'-diaminodiphenylmethane, diazotised, and developed with β-naphthol (yellow-red); H-acid, developed with diazotised p-nitroaniline (dark brick-red), or m-4-xylidine (red), etc.; S-acid, developed with diazotised anthranilic acid (brown; coppered, dark bordeaux); xyleneazo-maminobenzoyl-J-acid (red); p-nitroaniline, reduced,  $\rightarrow$   $\beta$ -naphthol (red); pararosaniline (red).

C. HOLLINS.

[Manufacture of] solubilised nitrocellulose. C. ELLIS, ASST. to ELLIS-FOSTER Co. (U.S.P. 1,816,146, 28.7.31. Appl., 24.12.23. Renewed 1.10.29).—Stripped nitrocellulose film is passed continuously through a drum or oven containing air or, preferably, an inert gas or superheated steam and maintained (for 2—5 min.) above 150° but below the puffing temp. of the nitrocellulose. F. R. ENNOS.

Boiling of sulphite-cellulose. PATENTAKTIEB. GRÖNDAL-RAMÉN (B.P. 367,235, 14.4.31. Ger., 15.4.30). —The raw material (wood chips etc.) is saturated with aq. alkali bisulphite containing no, or very little, free  $SO_2$ , and the excess liquor is withdrawn and replaced by another solution prepared from waste or washing liquor and containing the whole of the free  $SO_2$  required for the boil. B. P. RIDGE.

Manufacture and treatment of materials made of or containing organic derivatives of cellulose. BRIT. CELANESE, LTD. (B.P. 367,830, 20.11.30. U.S., 20.11.29. Addn. to B.P. 333,504; B., 1930, 986).— A solution of an org. derivative of cellulose containing a wetting agent (Na or K salts of ricinoleic, stearic, or oleic acids, or salts of an alcoholic NH<sub>2</sub>-compound, such as triethanolamine, with these fatty acids) is dryspun so that the wetting agent is retained in the product, which is treated continuously with its production with aq. liquids for delustring, F. R. ENNOS.

Preparation of planographic printing bases. KALLE & Co. A.-G. (B.P. 368,083, 16.3.31. Ger., 14.3.30). —The desired printing design is produced on a sheet of saponifiable cellulose derivative, e.g., cellulose acetate, and covered with a protective layer; the uncovered material is hydrolysed by 10% NaOH, and, after washing, the sheet is inked with a fatty ink which is retained by the unchanged ester, but not by the saponified areas.

F. R. ENNOS.

Centrifuges for production of artificial silk and the like. INTERNAT. GEN. ELECTRIC Co., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 365,963, 12.6.31. Ger., 14.6.30).—The spinning pot or centrifuge bucket is constructed throughout of annularly disposed cords, provided with a steel-wire core, combined together with artificial resin. H. ROYAL-DAWSON.

Precipitation of carbohydrates and carbohydrate compounds. LONZA ELEKTRIZITÄTSWERKE U. CHEM. FABR. A.-G. (B.P. 368,078, 16.3.31. Ger., 17.3.30).— The solution of the material after being finely divided, *e.g.*, by centrifugal force, is allowed to traverse a free path in space and impinge at any desired angle on the precipitant liquid, which has also traversed a free path, so that the liquids are thoroughly mixed, the amount of solution flowing through the apparatus being considerably less than that of the precipitant ; by regulating the velocity of flow and composition of the liquids, also their form and size, the nature of the ppt. may be regulated. F. R. ENNOS.

Manufacture of artificial threads from cellulose derivatives. I. G. FARBENIND, A.-G. (B.P. 367,274, 17.6.31. Ger., 17.6.30).—A condensation product of a polyvinyl alcohol with an aldehyde is mixed intimately with a solution of a cellulose derivative in the proportion 5—30% (calc. on the cellulose derivative) and the mixture is dry-spun. The resulting threads have high tensile strength in both the wet and dry states and improved resistance to creasing. B. P. RIDGE.

Manufacture of artificial threads, filaments, and the like. COURTAULDS, LTD., and C. DIAMOND (B.P. 368,022, 2.2.31).—In the downward dry-spinning process the evaporative atm. is draw off from a point at or towards the upper end of the spinning cell, a stationary deflector being placed at one end of the cell at a short distance below the spinning nozzle so that the atm. travelling up that side of the cell is made to pass obliquely or horizontally across the face of the nozzle.

F. R. ENNOS.

Treatment of artificial silk. BRIT. BEMBERG, LTD., Assees. of J. P. BEMBERG A.-G. (B.P. 365,016, 24.7.31. Ger., 24.7.30. Addn. to B.P. 312,034 and 363,958; B., 1930, 505; 1932, 258).—In order to avoid stressing and damaging during the twisting process, the moist aftertreated silk is supported in skein form by a reel rotating rapidly about a vertical axis and is withdrawn, while being twisted, in the axial direction of the reel by a withdrawal bobbin. F. R. ENNOS.

Spinning of artificial filaments, threads, ribbons, etc. BRIT. CELANESE, LTD. (B.P. 367,896, 27.11.30. U.S., 27.11.29).—A solution containing an org. derivative of cellulose with 2—20 wt.-% each of a di- or polyhydric alcohol (ethylene glycol, glycerol) and an oleaginous substance (fatty acid, glyceride, or mineral oil) is extruded into a coagulating medium or evaporative atm.

F. R. ENNOS.

Production of dull threads or other articles from viscose. FELDMÜHLE A.-G., VORM. LOEB, SCHOENFELD & Co. (B.P. 365,994, 29.10.31. Ger., 14.2.31).—Viscose is used with an admixture of dissolved factice, made by the action of  $S_2Cl_2$  on a benzine solution of groundnut oil. H. ROYAL-DAWSON.

Doubling and sizing of artificial silk. HEBERLEIN & Co. A.-G. (B.P. 367,965, 18.12.30. Ger., 28.10.30).— The thread is passed successively without interruption through revolving winding-off, sizing, drying, and receiving apparatus. F. R. ENNOS.

[Bobbin and sleeve] apparatus for production of artificial silk. H. A. GILL. From GLANZSTOFF-COURTAULDS GES.M.B.H. (B.P. 368,748, 5.11.30).

Waxed papers and manufacture thereof. WAXED PAPERS, LTD., and W. RALSTON (B.P. 369,208, 11.12.30).

Centrifugal separator [for pulp].—See I. Polishing felts for glass.—See VIII. Determining  $H_2O$ etc. in fibrous materials.—See XI. Leather substitutes.—See XIV. Cementing compositions.—See XV. Saccharification of cellulose.—See XVII. Cellulose ester films.—See XXI.

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

Viscose yarn dyeing. C. M. WHITTAKER (J. Soc. Dyers and Col., 1932, 48, 27-30) .- The fastness to light of vat dyes on viscose silk is largely influenced by the after-soaping treatment ; excessive soaping, especially with Indanthrene Pink B, reduces fastness. Triazogene Orange R after-coppered changes to red when exposed to light. The effect of covering dyed fabrics with ordinary window glass (1-1 in.) during exposure to light is negligible. Primuline, which fluoresces in ultraviolet light, continues to do so when exposed under 1 in. of window glass. Benzopurpurine 10B fades at the same rates under blue, frosted, and ordinary glass, but does not fade under red glass. The fast-to-light Chlorantine LL colours (C.A.C.), except Chlorantine Fast Blue GLN, are not suitable for dyeing viscose because they are uneven-dyeing. A test for evenness of dyeing with direct dyes which is more sensitive than those previously devised (Hall, B., 1930, 1023) consists in dyeing together over a range of rising temp. 150 (denier)/36 (filaments) and 150/21 yarns; even-dyeing dyes are fuller on the 150/36 yarn at low temp., but with rise of temp. the 150/21 yarn becomes the fuller. Satisfactory fast-tolight dyes are Rigan Green G and BL, Neolan Green BL, and Benzo Viscose Green B; their fastness exceeds that of Chlorantine Fast Blue BL. The light-transmission of Vitaglass when covered with a fine layer of dust is similar to that of ordinary glass. The dyes in dyed cellulose acetate yarn migrate along the filaments when maintained wet. Dyes may be classified according to their rate of migration : Silatose Fast Yellow is very rapid. A. J. HALL.

Dyeing with alizarin lake. J. W. ACKERMAN (J. Physical Chem., 1932, 36, 490—504).—Al<sub>2</sub>O<sub>3</sub> and a solution of Na alizarinate form a red lake, the alizarinate ion is adsorbed by the Al<sub>2</sub>O<sub>3</sub>. Alizarin lake can be peptised by Na alizarinate, but not by aq. NH<sub>3</sub> or AcOH. A method of dyeing with alizarin is outlined. S. LENHER (b).

Fading of lakes and dyed fibres. W. D. BANCROFT and J. W. ACKERMAN (Proc. Nat. Acad. Sci., 1932, 18, 147—150).—A colourless mordant, acting only as an adsorbent, lowering the chemical potential of the dye, increases the fastness of the dye to light. Using equal wts. of the same dye on different colourless mordants, the dye on the less saturated mordant is faster to light. Reversal of the relative absorbing powers of two mordants results in the reversal of fastness to light of the dyed mordants. The physical state of the mordant plays a part in deciding the fastness to light of a given amount of dye on the mordant. If a colourless mordant acts as an oxidising catalyst it may cause a dye to be less fast to light. A. J. MEE.

Measuring the detergent action of laundry supplies. O. M. MORGAN (Canad. J. Res., 1932, 6, 292-305).—An improved soiling material for use in the measurement of the detergent efficiencies of soaps has been devised, containing  $CCl_4$  2 litres, Russian tallow 3 g., Nujol 10 g., lampblack 2 g. The technique of its application to white, desized cotton sheeting has been studied in order to obtain uniformly soiled sheeting as sensitive as possible to small differences in washing efficiency. The effects on the latter of time, temp., soap concn., and mode of agitation have been investigated. An improved form of wash wheel (cf. Rhodes and Brainard, B., 1929, 364) is described.

D. R. DUNCAN.

Oil damage to cotton tenting materials. O. M. MORGAN (Canad. J. Res., 1932, 6, 306–308).—The effect of oil on the weathering of cotton duck tenting material has been investigated. The deteriorating action increases in the order : mineral lubricating oil, castor oil, linseed oil, cottonseed oil. This action is due partly to lubrication of the fibre, but mainly to oxidation, the unsaturated oils absorbing  $O_2$  from the air and oxidising the cotton to oxycellulose. Treatment with mineral Khaki considerably increases the resistance of the duck to oil and weather. D. R. DUNCAN.

Dry-cleaning soaps.-See XII.

#### PATENTS.

Dry vats [for loose goods]. OBERMAIER & Co., O. GÜMBEL, and G. FLUCK (B.P. 367,838, 25.11.30).— Each vat comprises two parts separated by a horizontal perforated plate, the lower part being subdivided by a vertical partition into two equal compartments each in communication with a rotary pump. Dye liquor is circulated upwards through one compartment and then through the textile materials in the upper part, thence returning downwards through the other compartment; the direction of flow is periodically reversed.

A. J. HALL.

Dyeing wool fast tints. Soc. CHEM. IND. IN BASLE (B.P. 364,148, 26.9.30. Switz., 26.9.29).—A prechromed dye obtained from 1-diazo- or nitro-1-diazo- $\beta$ -naphthol-4-sulphonic acid and  $\alpha$ - and  $\beta$ -naphthol is applied to wool in a bath having an acidity equiv. to < 3% H<sub>2</sub>SO<sub>4</sub> (on wt. of wool), and the colour is then developed with more conc. acid to give dyeings fast to rubbing.

C. HOLLINS.

Dyeing, printing, and like processes [using vat dyes]. IMPERIAL CHEM. INDUSTRIES, LTD., B. WYLAM, and J. S. WILSON (B.P. 366,543, 2.9.30).—Vat and Soledon dyes are printed on cotton fabric, using a paste thickened with cellulose esters, such as are produced by treating cellulose with pyridine–SO<sub>3</sub> in AcOH or pyridine and pptg. the product with EtOH. A. J. HALL.

Loading of natural silk. W. ZÄNKER (B.P. 366,513, 30.9.30. Ger., 21.3.30).—The silk is passed through a solution of a metal (Sn) salt and one or more neutral salts of alkalis,  $NH_4$ , Mg, or org. amines, and  $H_3PO_4$  or its salts, and then (without intermediate washing) through a fixing liquor containing  $Na_2HPO_4$ , whereby loading is secured without impairing the lustre of the silk. [Stat. ref.] A. J. HALL.

Impregnating and stiffening fibrous materials. I. G. FARBENIND. A.-G. (B.P. 367,126, 19.1.31. Ger., 18.1.30).—Textile fabrics, felt, straw plait, and asbestos fabrics are stiffened without loss of lustre and elasticity, and so that they are not liable to "spot" with  $H_2O$ , by impregnation with a solution of polystyrene (cf. Staudinger and Breusch; A., 1929, 434) or homologues thereof in an org. solvent, followed by drying and pressing into shape. A. J. HALL.

Making of rubberised sheet materials. H. D. RICE, ASST. to WOONSOCKET RUBBER CO. (U.S.P. 1,817,323, 4.8.31. Appl., 22.3.29).—A " bat," formed by blending fibres, e.g., rayon (50 pts.), wool (10 pts.), cotton (20 pts.), and hemp (20 pts.), after being " hardened " is impregnated with rubber, preferably by means of an aqdispersion, while still wet, and is then dried and coated with rubber. The coating is then treated with a halogenating agent, e.g.,  $S_2Cl_2$ , pressed between embossing plates, vulcanised, and finally surface-treated with a halogenating agent, e.g., Br and  $S_2Cl_2$  together or successively. The product has the characteristics of finished leather as used for upholstery etc. D. F. TWISS.

Drying of bodies, particularly breadths or strips of fibrous material impregnated or coated with a solution of synthetic resin in spirit. R., M., and E. SCHRÖDER, and S. R. and S. LEVIS (JAROSLAW'S ERSTE GLIMMERWARENFABR. IN BERLIN) (B.P. 365,922, 5.5.31. Ger., 5.5.30. Addn. to B.P. 354,099; B., 1931, 920).— The process described in the prior patent is operated in different parts of a single drying chamber.

B. M. VENABLES.

Treatment [lubrication] of [cellulose ester and ether] textile materials. BRIT. CELANESE, LTD., S. A. WELCH, and R. P. ROBERTS (B.P. 365,566, 15.10.30. Cf. B.P. 365,621; B., 1932, 338).—The hygroscopic solid is employed in an alcoholic medium which is also hygroscopic, and may be applied to the textile material by passing it across pads or wicks saturated with such solution. Suitable liquids are mono- or di-ethylene glycol, and glycerin. A plasticiser such as the Me ether of ethylene glycol and the Et<sub>2</sub> ether of diethylene glycol may be added. A. J. HALL.

Dyeing of yarn in package or wound form [on hollow perforated spindles]. ECLIPSE TEXTILE DEVICES, INC. (B.P. 368,973, 3.3.31. U.S., 3.3.30). [Sectional] vats [with acid-proof linings] for the dyeing of materials. J. DYSON (B.P. 369,475, 1.6.31. Austral., 11.6.30).

**Dye jiggers.** (SIR) J. F. NORTON & Co., LTD., and W. J. NORTON (B.P. 369,405, 27.3.31).

Machines for steaming fabrics. J. BAILEY (B.P. 369,416, 1.4. and 27.6.31).

Wetting etc. agents.—See III. [Delustring] treatment of materials.—See V. Imitation suède leather. —See XV.

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

Detection of nitrous oxides and iron in sulphuric acid. Note on Molisch's test. L. W. MARRISON (J.S.C.I., 1932, 51, 110 r).—Nitrous oxides are detected in amounts greater than 5 p.p.m. in conc.  $H_2SO_4$  by the green colour imparted to the acid layer in carrying out the Molisch test. Procedure for carrying out the test is identical with the usual Molisch test. If more than 0.06% N<sub>2</sub>O<sub>3</sub> is present, the upper (aq.) layer will be coloured yellow. Fe<sup>--</sup> interferes, about 300 p.p.m. giving the same depth of green in the acid as 5 p.p.m. N<sub>2</sub>O<sub>3</sub>. Chromates and molybdates also interfere, but none of the other common anions and cations. If only traces of carbohydrates are present, the Molisch test will be vitiated by traces of N<sub>2</sub>O<sub>3</sub> often present in "A.R." H<sub>2</sub>SO<sub>4</sub>.

Use of chromium-plated bolts [in nitric acid manufacture]. C. HÜTTER (Chem. Fabr., 1932, 59—60). —The use of ordinary nuts and bolts in making joints in stoneware acid lines leads to loss through fracture of stoneware parts in attempting to slacken corroded bolts. A great saving is effected by the use of Cr-plated nuts and bolts. A non-porous lining can be readily produced by the use of low current strength without a previous Ni-plating. Test pieces should suffer no loss of wt. after heating in  $HNO_3$  for 2—3 weeks, and the coat should be resistant without polishing. No evidence exists of corrosion due to galvanic currents between Cr-plated and non-plated parts. C. IRWIN.

Alkali-chlorine electrolysis. H. PAWECK (Z. Elektrochem., 1931, 37, 724–725).—Recent improvements in the process are described. E. S. HEDGES.

Acceleration of sedimentation in causticising sodium carbonate solutions with lime. M. O. CHARMANDARIAN and A. V. PETROV (J. Appl. Chem., Russia, 1931, 4, 464–468).—CaCl<sub>2</sub>, MgCl<sub>2</sub>, FeCl<sub>3</sub>, or colloidal solutions of  $Al(OH)_3$ ,  $SiO_2$ , or  $Fe(OH)_3$  have no appreciable effect; albumin produces a slight acceleration, but gelatin, starch, and raw potatoes at 95° are most effective. CHEMICAL ABSTRACTS.

Salt works design [and practice]. W. MÜLLER (Chem. Fabr., 1932, 57-59, 67-69, 73-75).—The physical structure and chemical purity of commercial NaCl are important. Brine conc. in open pans at 70-105° yields much coarser crystals than brine treated in vac. pans. The product of the latter is chiefly used in the chemical trade and as table salt, and the former for preservative purposes. The same works can usually make both kinds with advantage. A back-pressure turbine is suitable for power production, the exhaust steam going to the vac. pans. In many cases surplus current is used for electrolysis. Hot air for the dryer is obtained by a heat exchanger in the boiler flues. Costs are discussed. Another method of working is to use the surplus power for compressing the vapour from the evaporating pan and passing it into the heating chamber of the latter; a plant working on these lines is described. The heating elements of the evaporator are in the form of concentric rings, and scrapers are provided to remove salt crusts. The NaCl is removed at the bottom and falls into centrifuges. The connexions are so designed that the presence of hot, saturated brine in pipe-lines is excluded and no trouble with blockages has occurred. Fe is suitable for all plant construction except where NaCl, moisture, and air are simultaneously present. Corrosion is caused by badly de-aërated brine. Sharp bends in connexions tend to corrode. The NaCl produced in this plant is coarse enough for centrifuging. The output is about 11 tons per hr., and the plant requires 2 or 3 attendants. C. IRWIN.

The Gesellschaft für Kohlentechnik soda process in closed apparatus. B. LÖPMANN (Ber. Ges. Kohlen-tech., 1932, 4, 73-81).-When a new solution is started in this process (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> is formed instead of NH<sub>4</sub>HCO<sub>3</sub> and NH<sub>3</sub> losses will result unless the plant is closed. This loss is under 5%, but the higher  $HCO_3'$  concn. possible in closed apparatus will also give a higher yield of NaHCO<sub>3</sub>. To prevent the deposition of NaHCO<sub>3</sub> together with NH<sub>4</sub>Cl in this case it is sufficient to pass in 0.3% of gaseous  $NH_3$  before  $NH_4Cl$  crystallisation. A subsequent addition of the equiv. CO2 is then necessary. The NH<sub>4</sub>Cl produced in this way contains 2% of NaCl and 0.4-1.0% of NaHCO<sub>3</sub>, and the NaHCO<sub>3</sub> only traces of NaCl. Al was used as material for the vessels in this research, but was corroded, 0.02-0.08% of  $Al_2O_3$  being found in the NaHCO<sub>3</sub>. The yields obtained were 90-95% of NaHCO<sub>3</sub> and about 90% of NH<sub>4</sub>Cl. The latter salt was obtained much freer from NaHCO<sub>2</sub> if commercial NaCl was used than with pure NaCl, a result ascribed to the beneficial action of 0.3%C. IRWIN. Mg salts present in the former.

Preparation of pure sodium bicarbonate. B. LÖPMANN (Ber. Ges. Kohlentech., 1932, 4, 96–98).— Commercial NaHCO<sub>3</sub> contains some NH<sub>4</sub>HCO<sub>3</sub>. It can be purified in a pair of closed agitating vessels united by a gas connexion. In the first the crude salt is dissolved in H<sub>2</sub>O with heating, and the CO<sub>2</sub> evolved is passed into the second. The first vessel is then allowed to settle, any sediment drawn off, and the liquor transferred to the second, where it is saturated with CO<sub>2</sub> and cooled. The deposited NaHCO<sub>3</sub> is centrifuged and dried at 45° in a rotary dryer under a CO<sub>2</sub> atm.

C. IRWIN.

Preparation of alkali and alkaline-earth thiocyanates. W. GLUUD, K. KELLER, and W. SCHULTZE (Ber. Ges. Kohlentech., 1932, 4, 45–50).—KCNS and NaCNS can be prepared by warming NH<sub>4</sub>CNS and K<sub>2</sub>CO<sub>3</sub> or Na<sub>2</sub>CO<sub>3</sub> in the solid state with stirring, and finally heating to 120°. NH<sub>3</sub> is evolved, which is partly condensed in combination with CO<sub>2</sub> and the remainder absorbed in H<sub>2</sub>SO<sub>4</sub>. Steam or H<sub>2</sub>O is then added to

dissolve the product, the solution is filtered through animal charcoal and conc. in vacuo, and cryst. NaCNS is very sol. and hygroscopic and requires good cooling.  $Ca(CNS)_2, 4H_2O$  is prepared in an analogous manner, using milk-of-lime which is heated till no more  $NH_3$  is evolved. The crude solution produced is black and its purification offers difficulty as it is decomposed by animal charcoal or any cellulosic material. It might, perhaps, be centrifuged. Ba(CNS)\_2,3H\_2O is most economically prepared from BaS, and the same difficulty in filtration occurs. In both cases concn. in vacuo is necessary. C. IRWIN.

Obstacles to the concentration of potassium ferrocyanide in a cyanide washer [on coke-oven W. GLUUD, W. KLEMPT, and F. BRODKORB gas . (Ber. Ges. Kohlentech., 1932, 4, 51-71) .- An alkaline washing solution containing FeS recovers the C2N2 content of gas as K4Fe(CN)6. This process has been applied experimentally in gasworks, but not in coke ovens. The first difficulty is that with increasing K4Fe(CN)6 concn. the removal of C2N2 from the gas becomes increasingly slower. Secondly, FeS appears to "age" and become inert. Thirdly, there is a loss of insol. Fe2Fe(CN)6 which it is in practice impossible to decompose with alkali in the washer. It is shown from theory that for the production of  $K_4$ Fe(CN)<sub>6</sub> it is necessary for the gas to have a high C, N, content, a not too low H<sub>2</sub>S content, and a sufficiently low CO<sub>2</sub> content. These conditions are better met by coal gas than by coke-oven gas. High O2 content is also unfavourable. The presence of tar, benzol, and NH<sub>a</sub> in the gas has little influence. Experiments supporting these conclusions are described. No method could be discovered to prevent the "ageing" of FeS. The effect of CO2 is to liberate H<sub>2</sub>S from K<sub>4</sub>Fe(CN)<sub>6</sub> solutions containing FeS in suspension and to ppt. insol. Fe compounds. O2 acts similarly by forming Fe(OH)2, and in each case the evolved  $H_2S$  leads to a loss of  $C_2N_2$  as thiocyanate. C. IRWIN.

Determination of carbide in calcium cyanamide by means of cuprous acetylide. F. TROST (Annali Chim. Appl., 1932, 22, 63—80).—The defects of the determination of  $CaC_2$  in  $CaCN_2$  by the ordinary volumetric gas method are discussed. Treatment of  $C_2H_2$  with either excess or deficit of Ilosvay's reagent (B., 1899, 1158) yields a compound of const. composition.  $CaC_2$  in  $CaCN_2$  may be determined by boiling the substance with  $H_2O$  in a gas apparatus, pptg. the  $C_2H_2$  in the resultant gases by Ilosvay's reagent, and determining iodimetrically the Cu in the ppt. formed. The apparatus and procedure are described.

T. H. POPE.

Change of water-soluble phosphoric acid content of superphosphate during storage in bulk. V. Reaction between monocalcium phosphate and calcium sulphate. T. SHOJI and E. SUZUKI (J. Soc. Chem. Ind., Japan, 1932, 35, 27–29 B; cf. B., 1931, 1009).—Ca(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub> and CaSO<sub>4</sub> when intimately mixed with about 10% of H<sub>2</sub>O and kept at room temp. and also at 60° for 90 days do not react, and therefore the decline of H<sub>2</sub>O-sol. H<sub>3</sub>PO<sub>4</sub> in superphosphate during storage is not attributable to this cause.

N. H. HARTSHORNE.

Preparation of luminous zinc sulphide. J. EINIG (Chem.-Ztg., 1932, 56, 185—186).—The best product is an otherwise pure ZnS containing 0.01% Cu, which has been ignited at 1150° for 1 hr. The prep. of this is fully described. C. W. DAVIES.

S compounds from boiler-furnace gases.—See I. Building of HNO<sub>3</sub> works.—See IX. Glow-discharge electrolysis.—See XI. Dispersed red lead.—See XIII. Baking-powders.—See XIX.

See also A., April, 326, X-Ray examination of Pb oxides. 329, Prep. of COSe. 335, Prep. of Be oxide sols and of Cu sols. 349, Reactions of  $Cl_2$  with solid carbonates. 352, Luminescent compounds. Prep. of cryst. HIO<sub>3</sub>. 356, Determining Zr in rocks.

# PATENTS.

Contact process for preparing sulphuric acid. IMPERIAL CHEM. INDUSTRIES, LTD., F. A. F. CRAWFORD, and W. A. P. CHALLENOR (B.P. 367,381, 19,8.30).—In using the V catalyst described in B.P. 337,761 (B., 1931, 112), the burner gases, after passing through a dry dustremoving plant, are cooled only to the temp. required for the treatment in the catalyst chamber, there being no necessity to remove acid fog and As compounds, involving cooling to room temp., washing, and subsequent reheating. W. J. WRIGHT.

Manufacture of nitric acid by oxidation of ammonia. F. A. F. PALLEMAERTS, and UNION CHIM. BELGE Soc. ANON. (B.P. 367,709, 23.7.31. Addn. to B.P. 360,778; B., 1932, 145).-The waste gases are in part admitted to the fuel burner and then enriched with  $O_2$  to give a higher combustion temp. The burner gases are mixed with the remainder of the waste gases at the burner outlet, the mixture having the requisite temp. for effecting expansion in the expansion machine. Alternatively, the whole of the waste gases may be introduced into the burner with the fuel, the mixture being passed over an oxidation catalyst. Or a heat exchanger, placed in series with the burner, is traversed in one direction by the burned gases, and in the other by the inert gases of the absorption system, these being heated in the heat exchanger before admission to the burner in order to assist combustion. W. J. WRIGHT.

Manufacture of phosphorus pentoxide and phosphoric acid. W. J. TENNANT. From VICTOR CHEM. WORKS (B.P. 367,690, 23.6.31).—The P-containing gases obtained by reduction of P minerals, after being burned, pass into a combustion chamber, parts of the walls of this chamber being cooled by  $H_2O$  flowing through tubes enclosed in them. Fume and solid impurities are thereby pptd., and the  $P_2O_5$  is carried off by the gases into a flue, the walls of which are provided with a similar cooling device. W. J. WRIGHT.

**Production of ammonia.** F. LAWACZECK (U.S.P. 1,815,410, 21.7.31. Appl., 26.3.25. Ger., 13.12.23).—  $H_2$  under high pressure, produced by electrolysis of  $H_2O$  in a closed vessel, is passed to a collecting vessel through a heated pipe to convert traces of  $O_2$  into  $H_2O$ , and thence to a nozzle, where it is mixed with  $N_2$ , admitted as a liquid, but gasified by the  $H_2$ . The synthesis apparatus comprises a series of concentric cylinders, having catalytic material in the upper portions

of the intermediate annular spaces and of the central cylinder, and a series of vertical tubes. The gas mixture under high pressure is introduced at the base, which becomes cooled by the  $N_2$ , and is conducted to the catalyst in the central cylinder, whence the gases containing  $NH_3$  pass down the tubes to the base, in which  $NH_3$  is condensed, and subsequently through successive cylinders and catalysts, the downward-flowing gases serving in their passage to heat those flowing upwards.

W. J. WRIGHT.

Manufacture of base-exchange bodies. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 367,582, 9.3.31).—A mixture of water-glass and aq. NaAlO<sub>2</sub> containing, preferably,  $Al_2O_3$ :  $SiO_2 = 1:6-12$ , with the addition, if desired, of plasticisers (kaolin, sulphite-cellulose waste liquor), is shaped into cylinders, granules, etc. and heated rapidly to  $100-150^\circ$ ; the product is then washed and dried. L. A. COLES.

Production of alkali-carbon briquettes in and for the manufacture of alkali cyanides or cyanamides. A. MENTZEL (B.P. 367,598, 19.3.31. Ger., 14.4.30).—A mixture of NaHCO<sub>3</sub> (e.g., from low-temp. carbonisation of lignite), coke, and a binder (NaCl) is briquetted cold and the product calcined at  $300-400^{\circ}$ in the absence of air before use; calcination may be effected as the briquettes are heated up in the cyanidising furnace. L. A. COLES.

Production of potassium formate. E. HENE (B.P. 366,556, 8.10.30).—An aq. solution containing  $HCO_2K$  and  $(HCO_2)_2Ca$  (40—70 pts.: 60—30 pts.) is treated with  $K_2SO_4$  to convert the  $(HCO_2)_2Ca$  into  $HCO_2K$  and to ppt.  $K_2SO_4$ , CaSO<sub>4</sub>, which is removed and used for the treatment of fresh solution containing  $(HCO_2)_2Ca$ , so that the  $K_2SO_4$  in the ppt. is converted into CaSO<sub>4</sub>. L. A. COLES.

**Production of ammonium nitrate.** "MONTE-CATINI" Soc. GEN. PER L'IND. MINERARIA ED AGRICOLA (B.P. 367,660, 22.5.31. It., 2.6.30).—Preheated HNO<sub>3</sub> and NH<sub>3</sub> are caused to react at a pressure above that at which the resulting  $NH_4NO_3$  solution would boil at the reaction temp., and the heat developed is used to concentrate the  $NH_4NO_3$  solution. Corrosion is avoided by using a slight excess of  $NH_3$ , which is separated from the steam in a rectifying column. F. YEATES.

Manufacture of [dehydrating] material containing calcium chloride. LAVENDER BROS. & Co. (DEHYDRATIONS), LTD., and A. E. SHERMAN (B.P. 367,654, 19.5.31).—Fused CaCl<sub>2</sub> is run on to porous material (*e.g.*, coke, pumice) maintained under vac. and heated at 100—780°; air may be pumped at several atm. pressure into the vessel after the addition of the CaCl<sub>2</sub>. L. A. COLES.

Leaching of phosphate rock. KUNSTDÜNGER-PATENT-VERWERTUNGS-A.-G. (B.P. 366,547, 23.9.30. Swed., 5.10.29. Addn. to B.P. 313,036; B., 1929, 681). —Recrystallisation of the hemihydrate into dihydrate in the prior process is carried out in the presence of excess  $H_2SO_4$  or of an alkali or  $NH_4$  sulphate.

L. A. Coles. Dissolution of phosphate rock in nitric acid. Odda Smelteverk A./S., and E. Johnson (B.P. 366,970, 6.11.30. Norw., 7.11.29. Addn. to B.P. 339,340; B., 1931, 201).—The rock is treated initially with liquor, *e.g.*, dil. HNO<sub>3</sub> or the mother-liquor from the crystallisation of the Ca(NO<sub>3</sub>)<sub>2</sub>, containing sufficient HNO<sub>3</sub> to dissolve only part of the rock, particularly CaCO<sub>3</sub> present in it, yielding a viscous or plastic mass which is kneaded or stirred to release included gases before the remaining acid is added. L. A. COLES.

Decomposition of calcium phosphates. I. G. FARBENIND. A.-G. (B.P. 366,866, 15.7.31. Ger., 19.7.30). —The raw phosphates are heated under raised pressure with solutions containing, e.g.,  $NH_4H_2PO_4$  and  $(NH_4)_2SO_4$ , and  $NH_3$  liberated during the reaction is withdrawn. The  $NH_4H_2PO_4$  produced by the reaction is recovered as such, or is converted into  $(NH_4)_2HPO_4$ by treatment with  $NH_3$ ; the  $CaSO_4$  is converted by treatment with  $NH_3$  and  $CO_2$  into  $(NH_4)_2SO_4$  for re-use. L. A. COLES.

Production of pure white monocalcium orthophosphate. S. MARTIN (B.P. 368,114, 13.4.31).— Impurities in  $H_3PO_4$  are oxidised by the addition of  $HNO_3$ ,  $HClO_3$ ,  $HBrO_3$ , etc., in excess, and at the same time, or afterwards, a small quantity of  $H_2O_2$  or other substance capable of removing colour is added. The  $H_3PO_4$  so purified yields, when mixed with Ca compounds,  $Ca(H_2PO_4)_2$  suitable for baking-powders etc. F. YEATES.

Stabilisation of monocalcic phosphate in its solutions and its transformation into other soluble phosphates, particularly alkaline phosphates. J. MARGOLÈS (B.P. 368,155, 27.5.31. Fr., 27.5.30).— An org. acid, weaker than the (mineral) acid used for the solubilisation of the raw phosphate, is used to stabilise aq.  $Ca(H_2PO_4)_2$  by adding it either to the  $Ca(H_2PO_4)_2$  or its mixtures with reaction products, and/or to the  $H_2O$  used for its dissolution. Ca superphosphate may be used as a basis for obtaining such stable solutions, and these serve for the production of mono-, di-, and tri-alkali phosphates. The acid stabiliser is recuperated by pptn. F. YEATES.

Production of beryllium salts from berylliumbearing materials. H. FISCHER, ASST. to METAL & THERMIT CORP. (U.S.P. 1,815,056, 21.7.31. Appl., 13.12.28. Ger., 17.12.27).—The raw material is heated with an alkali fluosilicate to  $600-900^{\circ}$ , the sintered mass being comminuted and leached with cold H<sub>2</sub>O and the solution evaporated. Alternatively, an alkalineearth hydroxide may be added to the solution before evaporation, so as to yield less complex Be compounds. W. J. WRIGHT.

Treatment [dehydration] of mineral [magnesium] salts. K. HAASE and W. MICHELS (B.P. 366,971, 6.11.30).—MgCl<sub>2</sub>, $6H_2O$ , and double salts containing MgCl<sub>2</sub> and NaCl, KCl, or NH<sub>4</sub>Cl, are partly dehydrated by passage down a tower, lined with enamel or ceramic material, in which they are treated with an ascending air current introduced at a suitable temp. (120—140° for MgCl<sub>2</sub>, $4H_2O$ ; 140—160° for MgCl<sub>2</sub>, $2H_2O$ ) and withdrawn at a temp. (80°) such that the upper portion is not fused or sintered. Dehydration is completed in a second tower by a stream of HCl introduced at 450—550° and withdrawn at 150—250°. L. A. COLES. Preparation of [oil- or sugar-]bleaching materials. PROCESS DEVELOPMENT CO. (B.P. 367,407, 6.10.30. U.S., 5.11.29).—A mixture of hydrated basic MgCO<sub>3</sub>, prepared by treating Mg(OH)<sub>2</sub> slurry with CO<sub>2</sub>, with hydrated SiO<sub>2</sub>, *e.g.*, kieselguhr (SiO<sub>2</sub>: MgO =  $1 \cdot 5 - 6 : 1$ ), is treated in an autoclave with steam at, *e.g.*, 200 lb./sq. in. until most of the CO<sub>2</sub> is removed; the Mg silicate formed is filtered off, washed successively with dil. H<sub>2</sub>CO<sub>3</sub> or AcOH and H<sub>2</sub>O, and then dried.

L. A. COLES.

Production of aluminium oxide from aluminium nitrate, in which the decomposition of nitric acid is reduced to a minimum. G. A. BLANC (B.P. 367,525, 20.1.31).—Al(NO<sub>3</sub>)<sub>3</sub>,9H<sub>2</sub>O is first fused in its own H<sub>2</sub>O of crystallisation, and heating is continued (a) at  $\geq$  150° until about 40% of the HNO<sub>3</sub> and H<sub>2</sub>O of crystallisation, (b) at  $\geq$  250° until about 75% of the acid and H<sub>2</sub>O, has been expelled, and (c) at 250—500° until decomp. is complete. F. YEATES.

Treatment of leucite and other alkali aluminium silicates with nitric acid. G. A. BLANC (B.P. 367,524, 20.1.31).—The siliceous residue resulting from the treatment is washed free from nitrates by  $HNO_3$  and then added to the  $KNO_3$  solution [obtained after separation of the  $Al(NO_3)_3$  by strongly acidifying with  $HNO_3$ ] to facilitate evaporation of such solution. The mass obtained after evaporating to dryness is dissolved in  $H_2O$ , any Fe being completely retained by the residue.

F. YEATES.

Manufacture of the carbonyls of molybdenum and tungsten. J. Y. JOHNSON. From I. G. FARB-ENIND. A.-G. (B.P. 367,481, 3.12.30).—Material obtained by reducing Mo and/or W compounds in the presence of Cu or its compounds is treated with CO at, *e.g.*, 200° and 200 atm. L. A. COLES.

Electrolytic production of metallic compounds [lead carbonate or hydroxide] of low solubility. R. NADAL (v RAMON) (B.P. 367,460, 12.5.31).—White lead is produced by the electrolysis at room temp. of NaClO<sub>3</sub> solution,  $d \ 1.022$ , supersaturated with CO<sub>2</sub>, using a Pb anode without a diaphragm; the process is stopped before the CO<sub>2</sub> is exhausted, the pptd. carbonate is removed, and the electrolyte is again saturated with CO<sub>2</sub> for re-use. Electrolysis is effected in the absence of CO<sub>2</sub> for the production of Pb(OH)<sub>2</sub> and, after removal of the ppt., the electrolyte is treated with, e.g., CO<sub>2</sub> to ppt. dissolved Pb(OH)<sub>2</sub> before re-use. L. A. COLES.

**Production of hydrogen.** W. GLUUD, K. KELLER, R. SCHÖNFELDER, and W. KLEMPT, ASSTS. to BERGWERKS-VERBAND ZUR VERWERTUNG VON SCHUTZRECHTEN DER KOHLENTECHNIK, G.M.B.H. (U.S.P. 1,816,523, 28.7.31. Appl., 20.7.27. Ger., 30.7.26).—Gases containing equal vols. of CO and steam are passed through heated chambers filled with calcined dolomitic CaO to absorb CO<sub>2</sub>.

W. J. WRIGHT.

Nebulating [volatilising] sulphur [for fungicidal purposes]. G. RUPPRECHT (B.P. 368,156, 27.5.31).— S is heated approx. to its b.p. by a burning heating medium, the products of combustion of which (or specially-produced combustion products, *e.g.*, smoke gases), superheated to approx. the temp. of evaporation of the S, are forced through the molten S to carry off

its vapours, such vapours being rendered heavier or weighted by the combustion gases. Apparatus is claimed. F. YEATES.

Enrichment of gases in krypton and xenon. VEREIN. GLÜHLAMPEN U. ELECTRICITÄTS A.-G. (B.P. 367,313, 12.10.31. Austr., 22.10.30).—The gases are subjected to thermal diffusion at raised pressure, *e.g.*, 120 atm., in a cascade of diffusers having their opposite sides maintained at, *e.g.*, 300° and 20°, respectively.

L. A. COLES.

Ozone generator [with intermittent supply]. BAMAG-MEGUIN A.-G. (B.P. 369,461, 11.5.31. Ger., 28.2.31).

Effecting reactions between gases.—See I. Alginic acid and its compounds.—See III. Decomp. of H<sub>2</sub>O.—See XI. Fertilisers.—See XVI.

# VIII.—GLASS; CERAMICS.

Armenian pumice and obsidian as raw material in the glass industry. I. I. KITAIGORODSKI and S. V. RODIN (Trans. State Inst. Test. Building Mat., 1929, No. 26, 3—21).—Three varieties of pumice and two of obsidian contained  $SiO_2$   $65 \cdot 87 - 74 \cdot 24$ ,  $TiO_2$   $0 - 0 \cdot 70$ ,  $Al_2O_3$   $13 \cdot 39 - 16 \cdot 12$ ,  $Fe_2O_3$   $0 \cdot 85 - 4 \cdot 18$ , MnO or FeO  $0 - 0 \cdot 75$ , CaO  $0 \cdot 92 - 3 \cdot 66$ , MgO trace  $-1 \cdot 32$ ,  $K_2O$  $2 \cdot 65 - 5 \cdot 76$ ,  $Na_2O$   $1 \cdot 84 - 4 \cdot 68$ ,  $SO_3$   $0 - 0 \cdot 49$ . The use of these materials in glass manufacture is described.

CHEMICAL ABSTRACTS.

Layering of glass on solidification. B. S. SVECOV (Bull. Acad. Sci. U.S.S.R., 1931, 1141-1149).-Glasses of different kinds (9), when repeatedly ground and melted, give upper and lower solid layers of different chemical composition and sp. gr. T. H. POPE.

Spectrographic examination of Roman puzzuolana. G. BANCHI and A. MAFFEI (Annali Chim. Appl., 1932, 22, 138-142).—Ag, Sn, Pb, Cu, Bi, (Mo), Al, Fe, Mn, Ti, Si, V, Zr, Ca, Ba, Sr, K, and Na were found.

O. F. LUBATTI.

Hygroscopic equilibrium [of clay].—See I.

# PATENTS.

Glass-melting furnaces. HARTFORD-EMPIRE Co., Assees. of V. MULHOLLAND (B.P. 366,530, 1.11.30. U.S., 9.11.29).—Uniform lateral heating of a furnace is attained by arranging the combustion of fuel from a no. of burners in a combustion chamber extending the full length of the furnace so that a complete blanket of flame spreads from the outer edges over the whole bath without " cold spots." The batch is fed into one end of the furnace and progresses through suitable heat gradients.

C. A. KING.

Apparatus [furnace] for electric melting of glass. L. MELLERSH-JACKSON. From HARTFORD-EMPIRE Co. (B.P. 364,999, 27.5.31).—Glass is melted in a vertical furnace having a constricted intermediate horizontal section between electrodes arranged in the upper and lower sections so that molten glass in the constricted section is caused to boil. J. S. G. THOMAS.

Manufacture of laminated glass. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of L. E. BARRINGER and H. C. ROHLFS (B.P. 367,523, 19.1.31. U.S., 18.1.30).— Sheets of glass are united by a layer of an alkyd resin under a pressure increasing from 50 lb. to 200 lb./sq. in. and a temp. decreasing from 110° to room temp.

L. A. Coles.

Manufacture of splinterless glass. I. G. FARB-ENIND. A.-G. (B.P. 367,633 and 367,658, [A] 28.4.31, [B] 21.5.31. Ger., [A] 28.4.31, [B] 21.5.30. Addns. to B.P. 349,283; B., 1931, 720).—Glass sheets are united by (A) a polyvinyl alcohol or a derivative thereof (e.g., a product obtained by esterifying  $\Rightarrow$  one half of the OH groups) capable of dissolving or swelling in H<sub>2</sub>O, or (B) a mixture of a polymerisation product of a compound containing the CH<sub>2</sub>.C. group (a polyvinyl ester, polystyrene, Et polyacrylate) and a condensation product of maleic acid or anhydride with an unsaturated alcohol or a compound yielding one by splitting off H<sub>2</sub>O (e.g., castor oil,  $\alpha\gamma$ -butylene glycol).

#### L. A. COLES.

Treatment of felts employed for polishing glass. Soc. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNY & CIREY (B.P. 366,872, 13.8.31. Fr., 19.9.30).—The surface of the felt that faces the glass is dipped in molten pitch containing a plasticiser (wax, turpentine), and, after withdrawal, the layer of pitch adhering to the felt is allowed to solidify. In order to cause the abrasive to adhere on starting the polishing operation, or to clean the surface after use, the pitch is softened by suitable mixtures of  $H_2O$  and a pitch solvent (EtOH, benzine, turpentine). F. R. ENNOS.

Slabs or moulded blocks of glass for building purposes. Soc. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNY & CIREY (B.P. 366,476, 29.8.30. Fr., 31.8.29).—The resistance of glass blocks to heavy loads, temp. changes, and splintering is increased by a tempering process. C. A. KING.

Apparatus for fusing quartz. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 364,688, 20.8.30).— A hemispherical arch of a furnace capable of being heated by means of burners arranged radially rests on supports to which a water-cooled grate is also attached. A bed of quartz of gradually diminishing size rests on the grate, which may be raised or lowered. Sintered quartz of about 1 mm. diam. forms the top layer, which is heated by the hot gases passing over the surface and escaping under the side walls. The bottom structure may then be raised to reduce the flue gap, and some of the hot gas passes downwardly through the quartz bed which reaches sintering temp. Porous plates of considerable diam. and thickness may thus be made. C. A. KING.

Tunnel kiln. F. M. HARTFORD and S. M. KIER (B.P. 365,614, 15.9.30).—At a point approx. midway of its length, a tunnel kiln is heated by a no. of burners on each side. Fuel is also injected through ports arranged transversely through the kiln crown so as to direct combustion between the settings. C. A. KING.

Manufacture of shaped ceramic products of pure white shades. I. G. FARBENIND. A.-G. (B.P. 366,890, 10.10.31. Ger., 1.11.30).—Ware made from kaolin containing colouring impurities (e.g., Fe<sub>2</sub>O<sub>3</sub>) is treated with Cl<sub>2</sub> at above 600° while being fired in a continuous muffle (tunnel) kiln. J. A. SUGDEN.

Ceramic vessel and its production. W. I. TYCER (U.S.P. 1,814,870, 14.7.31. Appl., 28.5.28).—A body of clay + (approx.) 1% of powdered Mn is dipped in a clay slip and fired at 1300°. J. A. SUGDEN.

Dry moulding of ceramic materials. PORZEL-LANFABR. KAHLA ZWEIGNIEDERLASSUNG FREIBERG (B.P. 367,828, 18.11.30. Ger., 23.12.29).—Ceramic materials unmouldable when dry are rendered mouldable by admixture with a H<sub>2</sub>O-insol. metal salt of a high-mol. org. acid, which is preferably pptd. in the presence of the material; *e.g.*, the org. acid is added to a suspension of unburned porcelain material or  $Al_2O_3$  in  $AlCl_3$  solution, and the solid material is filtered off, dried, and moulded. L. A. COLES.

Plant for drying ceramic wares and the like. K. LÜHMANN (B.P. 367,846, 23.10.30).—Transverse flues are provided under a battery of parallel drying chambers by which air may be delivered to any chamber and pass zig-zag through them all, the point of entry being moved up one chamber at a time. A two-storied arrangement is also described having hot-air flues in the roof as well. B. M. VENABLES.

Plant for cleaning or treating glaze used in the manufacture of pottery and the like. W. PODMORE and O. G. COLLIER (B.P. 367,849, 20.11.30).—The plant comprises an electric motor, a dynamo, a magnetic separator, a rotary pump, and a sifter, all arranged in a casing near the glaze tank. [Stat. ref.] L. A. COLES.

Manufacture of abrasives, grinding wheels, blocks, and like articles. C. H. THOMPSON and R. A. DEMMÉ (B.P. 367,114, 12.1.31).—Mixtures of steatite of high vitrification temp. (e.g., about 1450°) with  $CaB_4O_7$ , Pb silicate, or FeCl<sub>3</sub>, or of steatite of low vitrification temp. (e.g., about 1400°) with monazite sand, zirkite, Mn silicate, pyrolusite, V oxide or silicate, W carbide, are shaped and fired; other abrasives, e.g., SiC, alundum, spinel, may also be added. L. A. COLES.

Vitrified argillaceous products. W. B. PINE, J. B. SHAW, and M. C. SHAW (B.P. 366,498, 21.10.30. U.S., 4.11.29).—Argillaceous material (containing <30%Al<sub>2</sub>O<sub>3</sub>) to which is added any necessary fluxes (*e.g.*, dolomite, chalk, slag) is heated to a temp. between the point of incipient fusion and the pyrometric cone-softening temp. (*e.g.*, 1270°), pressed into shape while in the hot plastic state, and annealed. The material may be pressed around metallic reinforcements. J. A. SUGDEN.

Improvement of certain physical properties of synthetic precious stones. M. JAEGER and H. ESPIG, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,814,219, 14.7.31. Appl., 20.2.29. Ger., 17.5.26).—The stones are heated at redness for several hr., if necessary in a reducing or non-reacting atm., and allowed to cool slowly. The treatment renders the stones less brittle; in some cases (e.g., the padparadjah) it improves the colour and imparts an appearance like that of moonstone to spinels supersaturated with  $Al_2O_3$ .

L. A. Coles.

Refractory materials. W. H. DANIELS, Assee. of H. KOPPERS A.-G. (B.P. 365,779, 19.1.31. Ger., 20.1.30). --Chrome-Fe ore (1-3%) is added to preps. comprising sintered magnesite and a binder (e.g., waste sulphite liquor) before the latter becomes set, to prevent the breaking-up action of dolomitic impurities and Ca silicates which may be present. H. ROYAL-DAWSON.

Manufacture of refractory material. G. S. Hous-MAN, Assr. to E. J. LAVINO & Co. (U.S.P. 1,814,088, 14.7.31. Appl., 31.12.29).—Finely-wet-ground chromite (5-30%) is ground in a wet pan with SiO<sub>2</sub> rock (70— 95%) with the addition of a small amount of binding agent (goulac or sulphite-pitch). J. A. SUGDEN.

Apparatus for making laminated glass. DUPLATE CORP., Assees. of J. H. SHERTS (B.P. 369,028, 14.4.31. U.S., 16.4.30).

Saggers for use in firing ceramic ware. J. Woolfson (B.P. 368,931, 2.2.31).

Sifter for potters' slip.—See I. [Envelope for] discharge tubes. Insulating materials.—See XI.

# IX.—BUILDING MATERIALS.

Apparatus for testing heat-insulating bricks. T. H. BLAKELEY and J. W. COBB (J.S.C.I., 1932, 51, 83-89 T).-A commercial insulating brick (9  $\times$  4 $\frac{1}{2}$   $\times$  2 in.) placed horizontally has its own lower face heated electrically, the temp. ranging up to 1040°. The heat transmitted upwards through the specimen is dissipated by radiation and convection from the dull black surface of a metal plate lying on the specimen. The temp. fall across the brick is measured by thermocouples, and abs. conductivities are derived by applying an expression for the loss of heat by radiation and convection, the temp. of the metal plate and of the air being taken. The results ranged from 0.00017 c.g.s. unit upwards, and agree with those obtained by more elaborate methods. It appears that insulating bricks of diatomaceous earths conduct about one third as much heat as insulating bricks of fireclay, one tenth as compared with ordinary firebricks, and one fiftieth as compared with MgO bricks.

Influence of autoclave conditions on the strength of sand-lime bricks. G. E. BESSEY and F. M. LEA (J.S.C.I., 1932, 51, 91-94 T).—The effect of changes in the time and pressure of autoclaving on the compressive strengths, wet and dry, and free CaO contents of sand-CaO bricks were determined for one selected mix containing 10% of hydrated CaO. On autoclaving at  $183 \cdot 5^{\circ}$  ( $P = 157 \cdot 4$  lb./sq. in.) little increase in strength is obtained after 16 hr.; for an autoclave period of 7 hr. the strength tends to a limit at about 160° ( $P = 89 \cdot 6$  lb./sq. in.).

The rotary kiln in cement manufacture. XI. W. GILBERT (Cement, 1932, 5, 36-45; cf. B., 1931, 955).—A marked advantage in the ratio 7:1 is gained by the direct contact of hot air with cascading coal rather than by admitting the air through the shell of the dryer. A graph constructed from results with unrestricted millboard tubes illustrates that the charge % which is retained in any tube varies as the rate of feed per revolution. The rate of advance of the charge is independent of the charge vol. and is 23.3% of the diam. per revolution. C. A. KING.

Setting of plaster of Paris. H. B. WEISER and F. B. MORELAND (J. Physical Chem., 1932, 36, 1-30).-No evidence that a jelly forms before the plaster sets was found. The period of inhibition in the time-temp. curve for the reaction is due to delay in pptn. from supersaturated solution because of a dearth of gypsum nuclei; if the nuclei are supplied there is no inhibition. The speed of setting of a plaster depends on the gypsum content. The supersaturated solution can be caused to crystallise and the rate of setting increased by stirring the mixture of plaster and H<sub>2</sub>O. The effect of varying concns. of NH4NO3, NH4Cl, NH4CNS, (NH4)2SO4, NaCl, and MgCl, on the rate of setting accords with deductions from von Weimarn's theory of the rate of formation of nuclei. NH4OAc, however, does not agree with the theory, which is attributed to an adsorbed film over the gypsum nuclei that retards their growth.

F. L. BROWNE (c).

The Kelly tube and sedimentation of Portland cement. C. G. DUNCOMBE and J. R. WITHROW (J. Physical Chem., 1932, 36, 31-51).—A modified Wiegner apparatus obviating the defects of the Kelly tube is described. F. L. BROWNE (c).

Building construction in a nitric acid works. E. BREINDL (Chem. Fabr., 1932, 89-92).-Corrosion risks exclude Fe, and the plant described, in which HNO<sub>3</sub> was manufactured by NH3 oxidation, was housed in a brick and concrete building with wood roof. The 8 absorption towers, weighing 70 tons each, stand on a ferroconcrete platform, and the roof is carried on concrete pillars. Wood frames to the windows are excluded owing to the risk of dil. acid vapour condensing on the glass. The concrete was painted with "palesite" on a priming of black inert oil, and the floor with asphalt and then covered with a pavement of acid-resistant material in two layers. Ample ventilation was provided. The roof beams were painted with a BaSO4-silicate C. IRWIN. prep. as a fire precaution.

Properties of porous building materials. III. Theory of the absorption and transmission of water by porous bodies. IV. Determination of the absorption constants of a homogeneous specimen. E. MADGWICK (Phil. Mag., 1932, [vii], 13, 632-641, 641-650; cf. B., 1932, 263).-III. Theoretical.

IV. The method of measuring the absorption and transmission of  $H_2O$  by slabs of building material is described. H. J. EMELÉUS.

Displacement of the alkalis in puzzuolana by the action of lime. A. MAFFEI and G. BANCHI (Annali Chim. Appl., 1932, 22, 93—98).—When puzzuolana is made into a paste with CaO and allowed to set into blocks which are immersed in CaO solution, displacement of the  $K_2O$  of the puzzuolana by CaO occurs rapidly and is scarcely influenced by the concn. of the CaO in the solution. Displacement of the Na<sub>2</sub>O is parallel to that of the total alkali, this being a function of the total CaO concn. and of the time of action. With high CaO concns. the velocity of displacement increases with the time. T. H. POPE.

Hygroscopic equilibrium [of wood].—See I. See also A., April, 350, Synthesis of Ca silicates. Treatment of porous light [cement-composition] stones or blocks. W. C. FAIRWEATHER. From INGENIEURBAUGES. CHRISTIANI & NIELSEN M.B.H. (B.P. 366,793, 31.3.31).—The blocks are treated for 1—2 days with moist gases or saturated steam below 100°, and are slowly dried, *e.g.*, with the aid of furnace gases. L. A. COLES.

Manufacture of cement-composition bricks, blocks, slabs, etc. G. R. SPEAKER & Co., and P. C. Coombes (B.P. 368,041, 17.2.31).—Pumice stone is washed, crushed, and graded, and subsequently mixed with cement and moulded. C. A. KING.

Asbestos-cement composition. G. MORBELLI (B.P. 367,815, 25.8.30. Ger., 24.8.29).—Sufficient finelyground  $SiO_2$  is added to the asbestos-cement mixture to combine with the free CaO liberated when heated under steam pressure. The product is then waterproof.

C. A. KING.

Gypsum concrete. G. D. KING and F. J. GOUGH, Assrs. to UNITED STATES GYPSUM CO. (U.S.P. 1,813,452, 7.7.31. Appl., 23.10.27).—A mixture of finely- and coarsely-ground gypsum is calcined so that the former is converted completely into plaster of Paris while the coarse particles are converted only on the outside. The material is then mixed with  $H_2O$  for use.

C. A. KING.

Coating of concrete with rubber. P. H. WATKINS, Assr. to NAUGATUCK CHEM. Co. (U.S.P. 1,816,922, 4.8.31. Appl., 19.9.28).—The concrete is coated with an Fe paste and aq.  $CuSO_4$  is applied to this. After drying, a compounded rubber solution and a compounded aq. dispersion of rubber are successively applied and dried, so as to give, after vulcanisation, a rubber coating the side of which adjacent to the concrete is preferably of the consistency of vulcanite. D. F. TWISS.

Concrete waterproofing compound. M. B. CHIT-TICK, Assr. to PURE OIL Co. (U.S.P. 1,814,292, 14.7.31. Appl., 19.4.30).—A mixture of 90% of a 2% solution of Al stearate in paraffin oil of 100 viscosity and 10% of a mixture of a sulphonated vegetable oil and an  $NH_4$  soap forms a waterproofing agent for cement work.

C. A. KING.

Production of waterproof mortar-forming materials. C. FLEISCH (B.P. 367,162, 6.2.31. Ger., 17.2.30).—A mixture of CaO, which has previously been immersed in  $H_2O$ , and beef tallow is rotated in a closed drum. The CaO is hydrated and the heat of the reaction assists in producing a waterproof powder which may then be mixed with cement, gypsum, etc. C. A. KING.

Porous building material [from slag].I. G.FARBENIND. A.-G. (B.P. 368,186, 20.7.31.Ger., 29.7.30).—Molten slag is poured on to a bed of sand which issaturated with  $H_2O$ , but with no depth of  $H_2O$  above thesand.C. A. KING.

Manufacture of artificial granite. H. B. TAFT, Assr. to N. M. WELLMAN (U.S.P. 1,814,012, 14.7.31. Appl., 31.10.29).—Powdered natural granite is melted at >1540°, poured into moulds, and annealed at 1200— 1325° for 4½ hr. to give similar physical and visual properties to those of the natural granite.

C. A. KING.

Compositions for roads, ways, tennis courts, floors, etc. L. LEWIS (B.P. 366,559, 31.10.30 and 6.7.31).—Use is made of compositions comprising an aq. emulsion of a bituminous binder and fragments of (e.g., vulcanised) rubber that has been plasticised with a mineral oil, d about 0.933. L. A. COLES.

Manufacture of compositions for surfacing roads, floors, pavements, platforms, playgrounds, tennis courts, roofs, etc. J. P. SPRATLING (B.P. 367,161, 6.2.31).—A mixture of 75—86% of sand, 11—15% of bitumen, and 3—10% of clay, preferably fireclay, with or without the incorporation of fillers, is spread (warm or cold) and rolled. C. A. KING.

**Production of floor coverings.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 367,782, 18.11.30).— A homogeneous mixture of powdered mineral matter, a colouring agent, and a more or less permanent bitumen emulsion in  $H_2O$  which must not contain soap or alkali is applied to a floor and pressed. A hydraulic cement may be incorporated. C. A. KING.

Manufacture of floor coverings and the like. H. PAULUS (B.P. 366,806, 14.4.31).—A dry mixture of caoutchouc, 15—35% of its wt. of S, and cork cuttings (50—85% of the total material), with the addition, if desired, of colouring material and fillers, is rolled or pressed into shape and vulcanised under heat and compression, the pressure being maintained during cooling. L. A. COLES.

Manufacture of plastic asphaltic compositions for pavements. F. C. ALSDORF (U.S.P. 1,815,089, 21.7.31. Appl., 15.10.28).—Material which has been coated with a cold bitumen composition is treated with a hydrocarbon- $H_2O$  emulsion which partly emulsifies the asphalt, mostly on the surface. C. A. KING.

Manufacture of bricks [by compression of clay in a mould]. R. and V. Dollé (B.P. 369,452, 30.4.31. Fr., 5.5.30).

Cooling of [cement] mills.—See I. Distillation of tar.—See II. Glass slabs etc.—See VIII. Cementing composition.—See XV. Saccharification of cellulose.—See XVII.

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

Volumetric determination of sulphur in cast iron and steel. D. N. MONASTUIRSKI and A. L. OSTA-SHEVSKAYA (J. Appl. Chem., Russia, 1931, 4, 696-700). —In the evolution method CdS need not be filtered off, but too much HCl should not be added with the I. It is unnecessary to dil. before titrating with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

CHEMICAL ABSTRACTS.

Desulphurisation of Thomas pig iron. E. SPETZ-LER and H. SPITZER (Stahl u. Eisen, 1932, 52, 233-235).—Addition of burnt CaO to the pig-Fe ladle and to the mixer effects a reduction of 5-15% of the S content of Thomas Fe and also protects the linings of the ladle and mixer from corrosion by the slag.

A. R. POWELL.

Effect of alloying elements on malleable cast iron. F. L. COONAN (Abs. Theses Mass. Inst. Tech., 1932, No. 9, 60-62).—The effect of alloying the elements Al, Ni, Cr. Mo, and V on the properties and microstructure of malleable cast Fe was determined. Al and Ni cause the C to exist in the free state. Cr, Mo, and V tend to retain the C in the combined state. Ni in small quantities gives higher tensile strength and malleability; Al lowers these. Cr, Mo, and V increase the tensile strength and hardness, and lower the ductility, due mainly to a retardation of the effectiveness of the annealing operation by these metals. The annealing process appears to take place through the medium of solid solution. To produce malleable Fe of commercial importance the material must be free from coarse primary graphite. The possibility of developing an alloyed malleable Fe that could be nitrided was investigated. Al, Ni, Cr, V, and Mo, when used alone, do not materially affect the hardness of the product when nitrided. An alloy containing 1.25%Al and 0.9% Cr when nitrided was as hard as nitrided steel. A. J. MEE.

Annealing of high-quality sheet [iron]. E. MARKE (Stahl u. Eisen, 1932, 52, 262-266).-The relative merits of annealing in boxes in a small furnace, in boxes in a continuous tunnel furnace, singly in a continuous normalising furnace, and over an open fire are discussed. Box-annealing in a 1- or 2-box furnace has the advantages over similar annealing in a continuous furnace that a more even temp. is obtained, the boxes can be taken out immediately annealing is finished and rapidly cooled, and the furnace can be recharged at once; on the other hand, fuel consumption is high and the life of the boxes relatively short. The normalising furnace does away with the use of costly boxes, is more satisfactory for large sheets, avoids the danger of sheets sticking together, and will anneal as much material in hrs. as can be annealed in days in the box furnaces. Normalised sheets have a fine-grained structure and are particularly suitable for deep drawing. A. R. POWELL.

Theory and practice of steel hardening. WEVER (Arch. Eisenhüttenw., 1931-2, 5, 367-375).-The transformation of austenite during the cooling of steel takes place in three stages which are separated from one another by regions of relatively high stability. In low-C steel with about 1% Cr and 4% Ni a slow transformation into practically pure  $\alpha$ -Fe and Fe<sub>3</sub>C (as pearlite or troostite) occurs at 650-500° and, if sufficient time is allowed, all the austenite is decomposed. Between 450° and the martensite point the transformation into lamellar pearlite and later into troostite proceeds much more rapidly, but is never complete above 350°. The third stage corresponds with the martensite transformation which proceeds spontaneously and is followed by a slow decomp. into ferrite and highly dispersed Fe<sub>2</sub>C. The course of these reactions and their temp. ranges are modified considerably by the presence of alloying elements. By quenching steel from above the Al point in a medium held at a temp. within the range of max. stability of austenite, e.g., in Pb-Sn alloy at 250°, it is possible, therefore, to subject steel directly to a hardening process by cooling it more or less rapidly in a suitable medium, or, alternatively, to submit it to an improving process by reheating it in a molten metal bath at 500-650° to convert the austenite into pearlite. A "stepped improvement" is also possible if the steel be heated within the second range above-mentioned to convert the austenite into lamellar pearlite; this treatment is equiv. to, but produces more satisfactory properties than, quenching from above the A1 point into oil or  $H_2O$  followed by tempering at 400—500°, as it avoids risk of cracking during the formation and decomp. of martensite. Examples of the effects produced by these heat-treatments in a 1% Cr steel are shown graphically. A. R. POWELL.

Martensite crystallisation in high-carbon steels. H. HANEMANN and J. H. WIESTER (Arch. Eisenhüttenw., 1931-2, 5, 377-382).-The process of separation of martensite from austenite has been followed by quenching steel with 0.8-1.7% C in Wood's metal at 200°, polishing the specimens while hot, cooling under various conditions, and observing the structural changes under the microscope. The temp. at which martensite begins to form is a function of the C content, falling from 240° with 0.83% C to 80° with 1.69% C. At any temp. within the transformation range there is a definite equilibrium between the austenite and martensite; the  $\gamma \rightarrow \eta$  change is practically independent of the rate of cooling. In high-C steels austenite is extraordinarily stable between 200° and the temp. at which  $\eta$  begins to separate, but at higher temp. it readily decomposes into cementite and ferrite without passing through the  $\eta$  stage. Hence it appears that the martensite transformation in steels is fundamentally different from the transformation of austenite into cementite and ferrite. A. R. POWELL.

Austenite-martensite transformation and the theory of steel hardening. S. STEINBERG (Arch. Eisenhüttenw., 1931-2, 5, 383-385).-Metallographic examination of and dilatometric tests on steels with 0.7, 0.98, and 1.32% C have shown that supercooled austenite is converted more or less slowly at 200-250° into martensite; the stability of the austenite increases greatly with fall in temp. within these limits and with increasing C content. Below 300° practically complete transformation into martensite occurs; this change begins at 130° for steel with 1.3% C, at 230° for the 0.98% C steel, and at 280° for the 0.7% C steel irrespective of the rate of cooling. Magnetometric measurements indicate that rapidly-cooled hardened steels contain more residual austenite than do similar steels which have been slowly cooled ; the converse is true, however, for steel with 1.6% C. These results are held to support the internal-stress theory of steel hardening and to be contradictory to Hanemann's phase theory (cf. preceding A. R. POWELL. abstract).

Mechanism of solidification and crystallisation in the steel ingot. B. MATUSCHKA (Arch. Eisenhüttenw., 1931—2, 5, 335—349).—The structure of cast-steel ingots has been examined by breaking the ingots in the temp. range of "blue brittleness" after removing the ingot from the mould at different periods after casting. The results show that, immediately after casting, the structure consists of non-equiaxed crystallites with their principal axes in the direction of heat flow; this transcrystallisation persists throughout the ingot. As the metal cools, the columnar crystals break up from the centre of the ingot outwards and the structure then CL. X.-METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

consists of a mass of fine equiaxed crystals. The lower is the casting temp. the finer are the columnar crystals and the more rapid and complete is their breakdown. Rapid cooling through the transformation range suppresses this breakdown, especially when the metal has been cast at a high temp. On reheating at high temp., the fine-grained structure becomes coarse again, whilst the transcryst. structure in the outer zones decomposes into the fine-grained structure at temp. above the transformation point. The importance of these structural changes in the mechanical working of various steels is discussed. A. R. POWELL.

Effect of silicon on the transformation points and structure of high-chromium steels. T. MURA-KAMI and K. YOKOVAMA (Tech. Rep. Tôhoku, 1931, 10, 57—78).—Steels containing 0.3% C, 9—16% Cr, and < 3% Si have been examined by magnetic, dilatometric, and micrographic methods. The A2 point is lowered, whereas the A3 and A1 points are raised and the thermal effects at these points reduced by addition of Cr or Si. Addition of Si to Cr steels within the above limits of composition produces no new constituent, but reduces the C content of the eutectoid. Characteristic microstructures of quenched and annealed Cr–Si steels are included. A. R. POWELL.

Corrosion tests on cold-rolled acid-resisting chromium-nickel steel. P. SCHAFMEISTER and A. GOTTA (Arch. Eisenhüttenw., 1931-2, 5, 427-430). —Corrosion tests on steel with 0.1% C, 0.5% Si, 0.4%Mn, 8.8% Ni, and 17.6% Cr in 13 and 61% H2SO4 show that there is a slight reduction in the resistance to corrosion with increasing degree of reduction by rolling; this effect is more marked in 61% H<sub>2</sub>SO<sub>4</sub> than in the 13% acid. Addition of 2-8% Mo to the steel renders the steel almost completely resistant to the more dil. acid irrespective of the degree of rolling, but in the 61% acid the rate of attack still increases rapidly with the degree of reduction. The steel without Mo becomes progressively more magnetic with rolling, due to the formation of  $\alpha$ -Fe which sets up local elements with the remaining austenite; the Mo steel, on the other hand, retains its austenitic structure and remains practically non-magnetic even after severe rolling and hence is more resistant to corrosion. The corrosionresisting properties of these steels are entirely dependent on the continuity of the passive layer; when this is broken by chemical or mechanical means it rapidly heals, but, while broken, local elements are set up which increase the rate of dissolution in the more conc. acid until the film is completely destroyed, leaving a clean homogeneous surface on which a new passive A. R. POWELL. film is rapidly formed.

Formation of acicular ferrite in tungsten steels. T. MURAKAMI and S. TAKEDA (Tech. Rep. Tôhoku, 1931, 10, 79-106).—Acicular ferrite in W steels is formed when the Ar points of the steel are displaced below 600° by suitable heat treatment. This displacement is the greater the higher is the annealing temp. and the greater the rate of cooling. With a fixed C content increase in W raises the temp. and increases the magnitude of the A3 point, but decreases the magnitude of the A1 point. With a fixed W content increase of C in low-C steels lowers the Ar2 point. The form (but not the composition) of acicular ferrite in W steels resembles closely that of acicular ferrite in overheated plain-C steels and is similar to that of martensite; rapid cooling to the Ar2 point followed by slower cooling thereafter produces acicular ferrite, whereas rapid cooling in both stages produces acicular martensite, and an intermediate rate of cooling a mixture of both constituents. An explanation of the formation of acicular ferrite based on Tammann's theory is given. A. R. POWELL.

Decarbonisation of carbon steels in salt baths. A. SEUTHE (Mitt. Forsch.-Inst. Verein. Stahlwerke A.-G., 1931, 2, 61-76; Chem. Zentr., 1931, ii, 2777).—Steel (0.84% C) was heated for 0.5—3 hr. at 800—1100° in baths of NaCl, KCl, CaCl<sub>2</sub>, BaCl<sub>2</sub>, and mixtures thereof. Increasing decarbonisation is exhibited in the series NaCl-BaCl<sub>2</sub>, NaCl-KCl, NaCl, CaCl<sub>2</sub>-BaCl<sub>2</sub>, NaCl-CaCl<sub>2</sub>; increasing erosion is exhibited in the series CaCl<sub>2</sub>-BaCl<sub>2</sub>, NaCl-BaCl<sub>2</sub>, NaCl-CaCl<sub>2</sub>, NaCl-KCl, NaCl. Decarbonisation is due chiefly to dissolved O<sub>2</sub>; addition of FeO and Fe<sub>2</sub>O<sub>3</sub> has little decarbonising effect. In presence of sulphates O<sub>2</sub> is liberated from these compounds. A. A. ELDRIDGE.

Determination of beryllium in high-alloyed steels and ferroberyllium. H. ECKSTEIN (Z. anal. Chem., 1932, 87, 268—273).—After removal of W, Si, Mn, Cr, Mo, and V from high-alloyed steel, the separation of Be from Fe is effected by treating the freshly pptd. hydroxides with 20% CO<sub>2</sub>-free aq. KOH, whereby Be(OH)<sub>2</sub> is dissolved and after acidifying can be pptd. with aq. NH<sub>3</sub>, collected, and calcined. Any trace of Fe left is determined by redissolving the BeO in dil. HCl and titrating the Fe with dil. KMnO<sub>4</sub>. When the proportion of Be is high, as in Fe-Be, the ppt. of Fe(OH)<sub>3</sub> and Be(OH)<sub>2</sub> is calcined directly, redissolved, and the Fe titrated with KMnO<sub>4</sub>. M. S. BURR.

Ferrosilicon. W. SCHUT and J. D. JANSEN (Rec. trav. chim., 1932, 51, 321-341).-Cases of poisoning among transporters of Fe-Si have probably been due to AsH<sub>3</sub> and PH<sub>3</sub>, which are evolved in contact with H<sub>2</sub>O or moist air. Unless the alloy contains other metals (such as Mn and Al), these gases are evolved only from samples containing > 25% Si. Alloys containing 40-60% Si disintegrate spontaneously; the reason is not clear, but as the evolution of gas is thereby increased alloys of these compositions are very dangerous during transport or storage. In determining PH3 by absorption in aq.  $AgNO_3$  the relation  $PH_3 = 6AgNO_3$  should be used. The Lorenz method is suitable for determining very small quantities of P. The amount of Si in Fe-Si can be determined within 3% from the sp. gr. and also from the stopping power for X-rays. Products containing about 80% Si are separable by sieves into powders of very different sp. gr. and Si content. It is essential, therefore, to analyse the whole of the ground E. S. HEDGES. material.

Mineral flotation. A. F. TAGGERT (J. Physical Chem., 1932, 36, 130–153).—When the powdered ore mixed with  $Ca(OH)_2$  is suspended in  $H_2O$  containing pine oil and K Et xanthate and air is blown in, the oil forms an envelope to the bubbles, enabling them to adhere to the sulphide particles in preference to the gangue, and stabilises the froth. The sulphide particles also greatly increase the stability. The most satisfactory froth producers are alcohols and ketones of the hydroaromatic series, such as terpineol. The xanthate increases the concn. of sulphide in the froth by forming a  $H_2O$ -repellant film on the solid surface, probably by chemical reaction. In pulp-body flotation processes bubbles are caused to separate from the liquid phase on the contaminated surface of the mineral, and the latter is in the gas- $H_2O$  rather than the oil- $H_2O$  interface. Experimental evidence in support of the above explanation of the flotation process is brought forward. F. L. BROWNE (c).

Properties of copper in relation to low stresses. Effect of cold-work, heat-treatment, and composition. II. Creep tests at 300° and 350° of arsenical copper and silver-arsenical copper. H. J. TAPSELL and A. E. JOHNSON (Inst. Metals, Mar., 1932. Advance copy. 6 pp.).—The resistance to creep at 300° and 350° of Cu containing (a) 0.34% As, and (b) 0.31% As and 0.072% Ag, has been determined after (i) fully annealing and (ii) after 5% cold-work followed by annealing for 2 hr. at 350°. The stresses corresponding with a min. creep rate of  $10^{-3}$  in. per in. per day at 300° after (i) were (a) 3.3, (b) 3.8, and after (ii) were (a) 4.4, (b) 5.7 tons per sq. in. The corresponding vals. at 350° were 1.9, 2.4, 2.5, and 3.3 tons per sq. in., respectively.

A. R. POWELL.

Effect of casting temperatures and of additions of iron on bearing bronze (Cu 80 : Sn 10 : Pb 10).— C. E. EGGENSCHWILER (Bur. Stand. J. Res., 1932, 8, 67—77).—Increasing the casting temp. from 1850° to 2120° F. increases both the resistance to wear and the grain size and slightly decreases the Brinell hardness. The resistance to pounding is increased within the casting range 1900—2000° F., and the notch-toughness of bronzes cast close to 2000° F. is decreased. Small additions of Fe (< 0.3%) decrease the resistance to wear and increase the Brinell and the Rockwell hardness. Larger amounts of Fe (0.3—1.0%) cause segregation of Pb, reduce the grain size, and are detrimental to the bearing properties. E. S. HEDGES.

Bronze specimens from the Royal Graves at Ur. C. F. ELAM (Inst. Metals, Mar., 1932. Advance copy. 8 pp.).—The composition and microstructure of 6 bronzes found at Ur have been determined; all the specimens contained Ni, in one case as much as 1.8%, and four contained > 1% Sn, in one case 11.1%. Corrosion products present were chiefly malachite and cuprite; sulphates were detected in most cases, but no chlorides were found. Some of the metals appeared to have been castings and showed signs of blowholes and coring; the remainder had been worked and annealed.

A. R. POWELL.

Synthetic metal bodies. VI. F. SAUERWALD and S. KUBIK (Z. Elektrochem., 1932, 38, 33-41; cf. B., 1929, 212).—Density, electrical conductivity, hardness, tensile strength, and grain size have been determined for specimens of Fe and Cu powders compressed at 1500—5000 atm. and subsequently heated for 30 min. at various temp. Density and conductivity both increase with the temp. of annealing up to about 900°, whilst the change of hardness with this temp. is not necessarily parallel with that of tensile strength. Comparison of compressed Fe containing 4% C and 5% Mn with specimens of the same metal solidified from a melt showed that the latter possessed superior hardness. Sintered material was more satisfactory in respect of hardness, more uniform, and less brittle than cast metal. A method of hot-pressing in a reducing atm. is described. F. L. USHER.

"Fogging" of nickel. W. H. J. VERNON (Inst. Metals, Mar., 1932. Advance copy. 14 pp.) .- When polished Ni is exposed to the atm. a creamy-white "fog" forms on the surface under certain conditions; in the early stages this film consists of NiSO4 with a small proportion of H2SO4 and in this state may readily be removed by wiping with a cloth. If not removed action proceeds further, the H2SO4 reacts with the Ni, and the film is converted into basic NiSO4 which can be removed only by abrasion. The tendency of Cu-Ni alloys to fog depends solely on the Ni content, Cu being inert. Cr-Ni alloys have a smaller tendency to fog than has pure Ni, the Cr appearing to inhibit the action. Thin films of grease, especially lanoline, prevent fogging. The phenomenon occurs only when the R.H. of the air exceeds 70%; in drier air the metal remains bright indefinitely. Fogging is due partly to the presence of suspended particles of sulphates in the air, but the presence of SO<sub>2</sub> produces fogging much more rapidly owing to the catalytic oxidation of this gas to H<sub>2</sub>SO<sub>4</sub> at the Ni surface when the crit. R.H. is exceeded. Light accelerates film formation; in the dark the rate of formation is about half that in sunlight. Fogging is completely inhibited by previous exposure of the Ni to air containing traces of H<sub>2</sub>S, and almost completely by prolonged exposure to stagnant air at room temp.; annealing at 225-350° has no effect on the rate of A. R. POWELL. fogging.

Change of the modulus of rigidity in different metals caused by cold-working. T. KAWAI (Sci. Rep. Tôhoku, 1931, 20, 681-709).—The modulus of rigidity of cold-drawn Cu wires decreases with the degree of reduction to a min. at 30% reduction in area, then increases slowly; that of Ni wires rises rapidly to a max. at 10% reduction, then decreases slowly with further reduction in area; and those of Al, 70:30 brass, and 60:40 brass decrease continuously during drawing. On annealing hard-drawn wires the rigidity of Cu and Al increases slowly with rise in temp., then rapidly between the temp. at which crystallisation commences and finishes, and finally, after reaching a max., decreases slowly. The rigidity of hard-drawn Ni wires increases slowly up to the temp. at which recrystallisation commences, and then decreases, rapidly up to 700° and finally more slowly up to 800°, when it reaches the val. it had before cold-working. The rigidity of pure Fe and mild steel decreases slightly with cold-work to a min., then increases slowly. On annealing, the rigidity increases slowly up to 450°, then falls rapidly to 600° and more slowly to 850°, and finally rises to its initial val. at 900°. Decrease in rigidity in all cases is attributed to internal stress and rotation of crystal grains, and increase to refinement of the grain size; the variations of these factors with mechanical and heat-treatment cause the changes noted above. A. R. POWELL.

Cause of high permeability in air-quenched permalloy. K. HONDA (Sci. Rep. Tôhoku, 1931, 20, 731-735).—On heating permalloy the permeability rises rapidly to a max. at 530° and it is suggested that this max. is retained by air-quenching the annealed alloy from 600°. The high permeability is attributed to distortion of the spherical shape of the atoms at high temp., an effect which is accompanied by an increase in the amount and amplitude of rotational vibration. Passage of an a.c. through the wire has a similar effect to thermal agitation, and hence also increases the permeability considerably. A. R. POWELL.

**Technology and use of electron metal.** W. SCHMIDT (Z. Elektrochem., 1931, **37**, 508—517).— Methods of production, properties, and applications of electron metal are reviewed. E. S. HEDGES.

Solubility of aluminium in magnesium in the solid state at different temperatures. P. SALDAU and M. ZAMOTORIN (Inst. Metals, Mar., 1932. Advance copy. 5 pp.).—The solubility of Al in Mg containing 0.21% Si, 0.09% Al, and 0.02% Fe is 6.08% at 20— $300^\circ$ , then increases rapidly to 10.9% at  $400^\circ$ , and is 12.6% at the eutectic temp. (436°). A. R. POWELL.

Interaction of aluminium and water vapour. R. SELIGMAN and P. WILLIAMS (Inst. Metals, Mar., 1932. Advance copy. 4 pp.).—Pure and commercial Al and alpax are not appreciably attacked by dry steam, either in the presence or absence of air, after prolonged exposure at 200—500°, even at pressures up to 6.7 atm. A. R. POWELL.

Intercrystalline corrosion of duralumin. A. J. SIDERY, K. G. LEWIS, and H. SUTTON (Inst. Metals, Mar., 1932. Advance copy. 17 pp.) .- A tendency of duralumin to develop intercryst. corrosion can be observed by subjecting the specimens to corrosion for 14 days at 20° in N-NaCl containing 1% of HCl (gas). Specimens which have been heat-treated and aged in air before immersion in the reagent are more susceptible to intercryst. corrosion than similar specimens which are allowed to age in the reagent. Cold-work by tensionstraining increases the tendency to this type of corrosion, but no definite relation between the elongation and the corrosion can be observed. Cold-work by compression increases greatly the susceptibility to intercryst. corrosion especially within the crit. limits of 14-16.9 tons per sq. in. Penetration of corrosive media along the grain boundaries is greater when the material is strained transversely to the direction of rolling than when the straining is in this direction. In non-aërated reagents intercryst. corrosion is greatest at the water-line, and in aërated reagents at the point of impingement of the air bubbles on the metal surface. High quenching temp. reduce the tendency to develop intercryst. corrosion, but increase the tendency to superficial pitting. Quenching in cold  $H_2O$  is preferable to quenching in boiling H<sub>2</sub>O. The ultimate stress, 0.1 and 0.5% proof stress, and Young's modulus vals. of material quenched in cold H<sub>2</sub>O are slightly higher than those of similar material quenched from the same temp. in boiling H<sub>2</sub>O and then aged. Variations in the quenching temp.

between 490° and 520° have little effect on the tensile or bending properties or on the hardness.

A. R. POWELL. **Determination of beryllium.** L. FRESENIUS and M. FROMMES (Z. anal. Chem., 1932, 87, 273-285).--In the examination of ores for traces of Be spectral analysis is a useful aid, the most sensitive Be line being  $3131 \cdot 1$  Å. The following five methods for the determination of Be in steels have been compared experimentally : hydroxyquinoline, KOH, acetate, tannin, and  $(NH_4)_2S$ . All the methods give good results if suitable precautions are taken. It is especially advantageous to increase the proportion of Be by a preliminary Et<sub>2</sub>O extraction of as much of the Fe as possible.

M. S. BURR.

Zirconium. VII. Corrosion-resistance of zirconium alloys. H. L. COLES and J. R. WITHROW (Amer. Inst. Chem. Eng., Preprint, 1931, 10 pp.).— Zr-Ni-Si-Fe alloys exhibit good resistance towards HCl and  $H_2SO_4$ ; the best alloy contained C 0.355, Zr 2.70, Si 9.15, Al 0.32, Fe 12.03, Ni 71.41%.

CHEMICAL ABSTRACTS.

Formation of sulphide layers on silver and silver alloys. K. FISCHBECK (Z. Elektrochem., 1931, 37, 593—598).—In Ag-Cd alloys, treated with S vapour or with solutions of S, the rate of growth of the sulphide film increases with the content of Cd up to 30 at.-%, alloys richer in Cd being scarcely attacked. The presence of Cu in Ag-rich Ag-Cu alloys increases the permeability of the sulphide film. E. S. HEDGES.

"Stone test" as preliminary test for commercial platinum-palladium and platinum-iridium alloys. F. GOLDBERGER and O. KIENBERGER (Mikrochem., 1932, 10, 397-406).-The streak produced by rubbing a specimen of metal on a piece of unglazed porcelain consists of uniform finely-divided particles which on account of their large surface are more reactive than is the metal in bulk. Valuable information may be obtained also as to the physical properties of the metal. 0.5-2 mg. forms a sufficiently large sample. By using for comparison streaks of alloys of known composition, the composition of Pt alloys may be determined. Details are given of colorimetric methods applicable to alloys containing 90-98% of Pt and Pd, Pd and Cu, or Ir. H. F. GILLBE.

Distribution of corrosion. S. C. BRITTON and U. R. EVANS (Trans. Amer. Electrochem. Soc., 1932, 61, 123-137).-Observations on specimens of steel and Zn which have been cut, scratched, or abraded before immersion in a vertical position in 0.1M-K<sub>2</sub>SO<sub>4</sub> indicate the determining influence of imperfections in the primary, air-formed oxide skin on the initial distribution of corrosion (cf. Bengough, Lee, and Wormwell, A., 1932, 27). This condition afterwards gives place to a secondary distribution determined by differential aëration, but if the solution be shaken so as to maintain uniform O<sub>2</sub> concn. over the metal surface, the primary distribution is more persistent. The importance of "oxygen screening" caused by secondary corrosion products is also emphasised. With vertical rods of steel in NaCl + NH<sub>4</sub>Cl solutions (total concn. 0.1M), the surface of the solution being exposed to the air, British Chemical Abstracts-B.

430

corrosion was confined to a small area at the bottom of the specimen when the proportion of  $NH_4Cl$  was <0.8%, but increasing this proportion from 0.87 to 0.95%caused a sudden extension of the corrosion over 87%of the surface : with > 10% of  $NH_4Cl$  the whole surface was corroded. This effect and certain other differences in the action of  $NH_4Cl$  and NaCl are attributed to the weak alkalinity of aq.  $NH_3$ , causing the film formed on the upper portions of the specimen to be less adherent than when NaOH is the alkali liberated at these cathodic regions. H. J. T. ELLINGHAM.

Type of fracture of cast metals and alloys. I. IITAKA (Proc. Imp. Acad. Tokyo, 1931, 7, 337–340).— In addition to previously recognised types, fracture along dendrite stems and branches is very common.

N. H. HARTSHORNE.

Determining the volume changes occurring in metals during casting. C. M. SAEGER, JUN., and E. J. ASH (Bur. Stand. J. Res., 1932, 8, 37-60).— Reliable data for shrinkage were not obtained by the sand-cast-cone method. It is necessary to construct the sp. vol.-temp. curves for the solid and liquid metal. Data are given for Al, red brass, cast Fe, Al-Cu alloy, Pb, Zn, and Sn. E. S. HEDGES.

Time law of recrystallisation of worked metals. F. SAUERWALD (Z. Elektrochem., 1931, 37, 531—534).— The time course of annealing of worked metals has been studied by measurements of hardness. The curves obtained are different for each annealing temp., but, in general, show an initial slight decrease of hardness, followed by a rapid decrease, gradually reaching a final const. val. The time curves cannot be expressed by a general law. E. S. HEDGES.

Elastic extension of metal wires under longitudinal stress. II. Experimental. L. C. TYTE (Phil. Mag., 1932, [vii], 13, 49–69).—All metals investigated show deviations from Hooke's law, the amount depending on the material and its pretreatment and increasing with crystal size for annealed metals.

H. J. EMELÉUS.

Principal factors governing flowability of pure metals. A. PORTEVIN and P. BASTIEN (Compt. rend., 1932, 194, 599—601).—Methods described previously (cf. B., 1930, 563) are applied to Al, Sb, Cd, Pb, Sn, and Zn, and a suitable formula is given for measuring the flowability. C. A. SILBERRAD.

Testing of electric arc welds [in iron] and the utility of coatings on welding rods by the Ehn cementation test. W. ZIELER (Stahl u. Eisen, 1932, 52, 236—239).—A homogeneous structure is obtained in arc welds in Fe made in atm. of  $H_2$ , CO and  $H_2$ , and CO and  $N_2$ , and welds made under these conditions show a normal structure in the Ehn cementation test. Characteristic microstructures after cementation of welds made with rods coated with fluxes are given to show how the Ehn test can be utilised to test the efficiency of a coating for gas or electric welding.

A. R. POWELL.

Electrometric titration of nickel in steel. W. BOHNHOLTZER (Z. anal. Chem., 1932, 87, 401-415).— Details are given for the direct electrometric titration of Ni in steel with KCN and for the indirect method of adding excess of KCN and titrating back with  $AgNO_3$ . The influence of  $NH_3$ ,  $NH_4$  salts, and neutral salts has been examined. Mn and Cr have no influence on the titration and a correction may be applied for Cu.

E. S. HEDGES.

Electrolytic production of heavy metals from fused electrolytes. III. Indifferent anodes for high temperatures. F. SAUERWALD (Z. Elektrochem., 1932, 38, 76).—In the electrolytic separation of Fe from a bath of fused FeO,SiO<sub>2</sub> at 1250—1300° a water-cooled Cu anode may be used, provided it is placed at a distance of 5—10 mm. from the surface of the melt and the current is then carried by an arc. Mn–Fe alloys, with a content of 68% Mn as against 5% previously obtained, can be prepared electrolytically, using a C cathode and a pure Mn ore preheated to 1300° to remove dissociating O<sub>2</sub> (cf. B., 1928, 526). M. S. BURR.

Sodium amalgam formation from sodium chloride solutions. K. S. TESH and H. E. WOODWARD (Trans. Amer. Electrochem. Soc., 1932, **61**, 143—151).— In the electrolysis of well-stirred NaCl solutions with a layer of Hg on the bottom of the vessel as cathode and a Pt point dipping into the solution from above as anode, the concn. of Na obtained in the Hg increases almost linearly with time (up to about 1%) at const. c.d. from 0.25 to 2 amp. and temp.  $0-50^{\circ}$ . The effects of varying the NaCl concn. and altering the position of the anode were examined. H. J. T. ELLINGHAM.

Electrolytically produced protective oxide films on aluminium. H. Röhrig (Z. Elektrochem., 1931, 37, 721—724).—A review of published work.

E. S. HEDGES.

Electrolytic [deposition of] chromium on light metals. A. KOENIG (Z. Elektrochem., 1931, 37, 718— 719).—A review. E. S. HEDGES.

Plant practice for nickel-plating aluminium. A. V. RE (Trans. Amer. Electrochem. Soc., 1932, 61, 139—141).—After buffing and cleaning in a hot alkaline solution, followed by dipping in a cold conc. solution of  $H_2SO_4$  and  $HNO_3$  containing FeCl<sub>3</sub>, the article is washed and "flashed" with brass in an alkaline cyanide solution. Ni deposited on this brass surface adheres firmly and will withstand bending or twisting of the article without rupture. H. J. T. ELLINGHAM.

Electrodeposition of ternary alloys of copper, cadmium, and zinc from cyanide baths. R. C. ERNST and C. A. MANN (Trans. Amer. Electrochem. Soc., 1932, 61, 153-184).-Electrolysis of a solution containing 40 g. of CuCN, 36.15 g. of  $Zn(CN)_2$ , 2.94 g. of  $Cd(CN)_2$ , and 138 g. of KCN per litre at 25° with 0.3 amp./sq. dm. gives a smooth, fine-grained deposit resembling 60:40 brass in appearance but containing 62.55% Cu, 20.63% Cd, and 16.72% Zn. Separate anodes of the three metals were used. Raising the c.d., increasing the concn. of KCN, or adding KOH increases the proportions of Zn and Cd and decreases that of Cu, whereas diluting the solution or adding NaHSO3 increases the proportions of Cu and Zn and greatly decreases that of Cd. Agitating the solution decreases the proportion of Cu, and raising the temp. increases the proportion of Cd at the expense of that of Cu. Additional excess of KCN improves the anode corrosion but gives

grey deposits, whilst addition of KOH leads to coarsergrained deposits. The theoretical principles of the co-deposition of these metals are discussed.

H. J .T. ELLINGHAM.

Strains in electrolytic copper deposited in presence of gelatin. P. JACQUET (Compt. rend., 1932, 194, 456-458).—The effect of adding gelatin (0.5-500 mg. per litre) to  $CuSO_4$  solution (150 g.  $CuSO_4, H_2O$ per litre with  $H_2SO_4$  to bring the  $p_{\rm H}$  to 0.85) has been examined by the previously described method (B., 1931, 846). As the amount of gelatin per litre increases from 0.5 mg., the contraction at first observed decreases; with 50 mg. it is preceded by dilatation, which gradually increases until with about 200 mg. the net result is dilatation which thereafter increases. With < 50 or > 250 mg. the deposit is matt, and with intermediate amounts brilliant (cf. A., 1928, 850).

C. A. SILBERRAD.

Concentrating iron ores in the Urals. M. F. ORTIN (Trans. All-Russian Sci. Tech. Min. Cong., 1928, VIII, 7-31).

Application of selective flotation for concentrating the polymetal ores of Sadonsk, near Vladikavkaz. N. N. VARLAMOV (Trans. All-Russian Sci. Tech. Min. Cong., 1928, VIII, 88—104).

Plasticisation [of metals].—See I. Electrochemistry of alkali metals.—See XI.

See also A., April, 324, Hydrogenised Fe. 328, Conductivity of Ru. 330, Systems Sn-Cd, Fe-Zr, Ag-Cu, Cu-Cd, Fe-Hg. Segregation of cementite from austenite. 340, System Fe-C-O. Systems in production of steel. 350, Interaction of Cu-Sn alloys and CaO or quartz in O<sub>2</sub>. 355, Analysis of alloys by X-ray spectroscopy. 357, Te-Bi thermocouple.

# PATENTS.

Manufacture of desulphurised [low-carbon] pig iron. H. STEINEBACH (B.P. 367,602, 23.3.31).—Fe ore, smelted in the blast furnace at 300—500° without desulphurising agents, is tapped off into separate containers and allowed to cool before being cast. [Stat. ref.] H. ROYAL-DAWSON.

Improvement [hardening] of nickel-tin alloys. VEREIN. STAHLWERKE A.-G. (B.P. 367,571, 28.2.31. Ger., 4.7.30).—The alloys in rolled or cast form, and containing 5—25% Sn, are rapidly cooled from above 900° and then annealed at 400—800°.

H. ROYAL-DAWSON.

Bright annealing of metals. R. G. GUTHRIE and O. J. WOZASEK (O. J. WILBOR), ASSTS. to PEOPLES GAS BY-PRODUCTS CORP. (U.S.P. 1,815,505, 21.7.31. Appl., 15.11.29).—Metal is heated to annealing temp. in an oxidising atm. to remove oily and carbonaceous films, and then in a reducing atm. free from unsaturated hydrocarbons. C. A. KING.

Heat-treatment [bright annealing] of metal parts. R. V. WILSON, ASST. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,815,691, 21.6.31. Appl., 31.10.28).— For the bright annealing of Fe a more easily oxidisable metal, e.g., Cu tinsel, with or without powdered Mg or Al, is introduced into the annealing box out of direct contact with the Fe. C. A. KING. Electrolytic deposition of silver. KODAK, LTD., Assees. of K. C. D. HICKMAN and W. J. WEVERTS (B.P. 364,711, 21.10.30. U.S., 22.10.29).—Solutions of Ag halides in  $Na_2S_2O_3$  and  $Na_2SO_3$  solutions containing gelatin, e.g., spent fixing baths from photographic work, are regenerated by removing the Ag by electrolysis after addition of a weak acid, e.g., AcOH, and an activator containing one of the groups of the thiourea, thiocarbinide, thioamide, and dithio-class. Examples of suitable compounds are given. A. R. POWELL.

[Sheet-Al or -Fe] fireproof material for archival documents. R. KUPFER (B.P. 367,816, 15.9.30).

Manufacture of metal foil [strengthened by gauze]. W. FENNELL (B.P. 368,832, 11.12.30).

Tin pots or baths employed in manufacture of tin-, terne-, and like metal-coated plates or sheets. H. S. THOMAS and W. R. DAVIES (B.P. 368,736, 5.12.30).

Building material from slag.—See IX. Filmforming electrode.—See XI. Anti-rust paints. Moulded articles.—See XIII.

# XI.—ELECTROTECHNICS.

Electrical determination of  $p_{\rm H}$ . M. PICARD and C. LAGNEAU (Ann. Falsif., 1932, 25, 17—25).—By the method described,  $p_{\rm H}$  may be determined electrically using relatively inexpensive apparatus and with an accuracy sufficient for ordinary industrial purposes.

W. J. BOYD.

Wholly automatic electrode for  $p_{\rm H}$  measurements. H. D. VAN OORT (Internat. Sugar J., 1931, 33, 578—581).—The H electrode devised by Aten and van Ginneken (B., 1925, 1014) is modified by providing it with an automatic valve to avoid irregularities in the dropping rate of the stopcock. Thus equipped, the apparatus worked very satisfactorily with little supervision, giving diagrams of the  $p_{\rm H}$  of second-carbonatation juice with a high degree of accuracy. J. P. OGILVIE.

Manganese dioxide for dry cells. III. S. MAKINO (J. Soc. Chem. Ind., Japan, 1932, 35, 69 B; cf. B., 1930, 1034).—The suitability of a specimen of natural  $MnO_2$ for use in dry cells can be ascertained by observing the loss of wt. on heating; the greater the loss, the larger is the depolarising capacity. H. F. GILLBE.

Alternating-current rectification properties of photo-sensitive crystalline aggregates. G. P. BARNARD (Trans. Amer. Electrochem. Soc., 1932, 61, 185—193).—Objections to Eccles' thermo-electric theory of the rectifying properties of crystal contact detectors are advanced, and the principles on which a satisfactory theory may be developed are discussed.

H. J. T. ELLINGHAM.

Glow-discharge electrolysis. A. KLEMENC (Z. Elektrochem., 1931, 37, 742—744).—The glow discharge enables electrolytic processes to be studied in the absence of a metallic electrode. Solutions of  $KNO_3$  are reduced to  $HNO_2$ , NO, and  $N_2O$ , whilst  $NH_3$  and  $NH_2OH$  appear at a metallic, immersed cathode ; in other cases, however, reduction proceeds further with the glow discharge than in ordinary electrolysis.

E. S. HEDGES.

Electrochemical behaviour of the alkali metals. R. THILENIUS (Z. Elektrochem., 1931, 37, 740—741).— A discussion of the production of Na in the Castner cell. E. S. HEDGES.

Position and prospects of technical electrochemistry. J. BILLITER (Z. Elektrochem., 1931, 37, 712-718).—A review. E. S. HEDGES.

Acid-resistivity of organic dielectrics. P. A. FIORENSKI and K. A. ANDRIANOV (Vestn. Elektrotekh., 1931, iii, 83—118).—85 natural substances and artificial products were examined as to rate of dissolution in  $H_2SO_4$  ( $d \ 1.52$ ); permanganate was added to org. substances. The solubility curves correspond approx. with  $Q = M\tau^{n/m}$ , where Q is the quantity dissolved,  $\tau$  is the time, and n and m are characteristic consts.

CHEMICAL ABSTRACTS.

Benzol purification.—See II. Alkali–Cl<sub>2</sub> electrolysis. NaCl works design.—See VII. Arc-welding of Fe. Titrating Ni in steel. Heavy metals by electrolysis. Na-Hg. Oxide films on Al. Crplating of light metals. Ni-plated Al. Electrodeposition of ternary alloys. Electro-Cu.—See X. Determination of soil H<sub>2</sub>O. Producing pigment in apples.—See XVI.

See also A., April, 324, Hydrogenised Fe. 328, Conductivity of Ru. 401, Electrolysis of pyridine.

#### PATENTS.

[Electric] furnaces having means for indicating the magnetic condition of furnace charges. HEVI-DUTY ELECTRIC CO., Assees. of W. B. COOLEY (B.P. 366,542, 2.9.30. U.S., 3.9.29).—The whole of the heating resistance is arranged within the furnace and interference with the indication of the magnetic condition of the charge is avoided. J. S. G. THOMAS.

Arc-welding electrode. J. M. WEED, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,814,878, 14.7.31. Appl., 13.1.25).—A flux composed of Na titanate (1 pt.) and Zn or Sn (2 pts.) is arranged between a ferrous-metal core and a one-piece metal sheath. J. S. G. THOMAS.

[Cores of] arc-lamp electrodes. E. and O. CONRADTY (C. CONRADTY) (B.P. 367,534, 28.1.31).—A binder composed of natural and/or artificial resins together with, if desired, balsamic resins, *e.g.*, myrrh, benzoin, or storax, is claimed. J. S. G. THOMAS.

[Alkaline] secondary or storage batteries. J. J. DRUMM, and CELIA, LTD. (B.P. 365,125, 17.7.30).— Active negative material composed of Zn electrodeposited on a plate having a smooth clean surface of Ni or monel metal is employed. J. S. G. THOMAS.

Storage-battery [plates]. W. L. REINHARDT, ASSI. to WILLARD STORAGE BATTERY CO. (U.S.P. 1,817,846, 4.8.31. Appl., 18.6.28).—About 0.1—1% of a substance containing humic acid is mixed with the active material of the plates to increase their capacity and life. J. S. G. THOMAS.

Manufacture of galvanic dry cells. J. J. PALA (B.P. 367,698, 3.7.31).—Thickening of electrolyte with starch paste is effected by heating the cell in a molten bath of insulating material, *e.g.*, paraffin, ceresin, bitumen. J. S. G. THOMAS. [Separators for] electric batteries. M. and P. WILDERMAN (B.P. 367,986, 10.1.31).—Cellulosic material, e.g., wood flour or peat, is incorporated in separators composed mainly of porous hard to soft rubber mixture. J. S. G. THOMAS.

Electrolytic decomposition of water. A. T. STUART (B.P. 368,115, 13.4.31. Can., 22.4.30).—The speed of circulation of electrolyte is controlled, and electrolyte collected from the top of electrode compartments of one polarity passes directly to the bottom of those of opposite polarity. J. S. G. THOMAS.

Electrolytic device. [Film-forming electrode.] H. O. SIEGMUND, ASST. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,816,875, 4.8.31. Appl., 27.4.27).—An electrode containing 98.0—99.4% Al and small proportions of other elements, e.g., Si, Fe, Mn, resistant to corrosion, is claimed. J. S. G. THOMAS.

Electrolytic processes. E. and O. CONRADTY (C. CONRADTY) (B.P. 366,717, 3.2.31).—Trapezium- or parallogram-shaped anodes of C, graphite, etc. are arranged with inclined separating surfaces so that the surface of the anode through which current passes remains approx. const. during the life of the anode.

J. S. G. THOMAS.

Electron-discharge tubes. E. A. FRITZ (B.P. 367,310, 15.9.31. U.S., 20.9.30).—A cathode composed of BaO and SrO and a grid coated with Ta oxide are used. J. S. G. THOMAS.

Electric-discharge tubes for emitting ultraviolet rays. N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 366,675, 31.12.30. Holl., 12.2.30).—Part of the inner side of the tube wall is covered with a mirror, *e.g.*, of Cr, Ni, or Al, which, preferably, does not amalgamate with the Hg filling. J. S. G. THOMAS.

Luminous electric-discharge tubes. GEN. ELEC-TRIO CO., LTD., Assees. of PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 367,309 and 368,209, [A] 8.9.31, [B] 22.10.31. Ger., [A] 12.9.30, [B] 17.1.31).---(A) A valve controlled by the expansion of a bimetallic strip is arranged at the mouth of a gas-choke placed between the tube and the reservoir, to serve as means for replenishing the gas filling of the tubes. (B) The inner wall of the envelope, which is transparent to ultraviolet light, is lined with an alkali-proof borosilicate glass containing  $\geq 50\%$  SiO<sub>2</sub>, a considerable proportion of H<sub>3</sub>BO<sub>3</sub>, and the usual glass constituents, *e.g.*, alkalis, alkaline-earths, and Al<sub>2</sub>O<sub>3</sub>. J. S. G. THOMAS.

Electric cathode glow-discharge tubes having cold electrodes and a gaseous filling. N. V. PHILIPS' GLOEHLAMPENFABR. (B.P. 366,861, 29.6.31. Holl., 26.7.30).—An auxiliary electrode, e.g., of wire gauze, having a no. of openings, is arranged between the electrodes and is electrically connected with the anode. The gas-filling, e.g., of Ne with about 0.5% A at 6 mm. pressure, is such that the starting voltage between the main electrodes in the absence of the auxiliary electrode is a min. J. S. G. THOMAS.

Introducing barium into electrical-discharge receptacles. INTERNAT. GEN. ELECTRIC Co., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 367,792, 19.8.30. Ger., 20.8.29).—Ba of purity  $\leq 98\%$  is distilled *in vacuo* or in a rare gas at above 1150°, rapidly cooled, coated with protective material, *e.g.*, paraffin oil, pressed into filaments etc., and introduced into discharge tubes, if desired, in small receptacles of Ni or Mo. J. S. G. THOMAS.

Increasing the reliable working of metallicvapour arc-discharge apparatus. L. Mellersh-JACKSON. From SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 367,475, 27.11.30).—A device (e.g., a bundle of wires of Cu, Fe, Pt, W, etc.) having a capillary or absorbent action for drawing up the liquid electrode metal is arranged above the electrode to provide a terminal point for the arc at such a distance from the electrode that excess of metal vapour is not produced.

J. S. G. THOMAS.

**Preparation [cleaning-up] of luminescence tubes.** F. W. ZONS, ASST. to AMER. NEON LIGHT CORP. (U.S.P. 1,817,333, 4.8.31. Appl., 13.2.29).—The electrodes are coated with alkaline-earth nitrates,  $P_2O_5$  is placed in the tube, and current is passed. J. S. G. THOMAS.

Photoelectric cells. GRAMOPHONE Co., LTD., and W. F. TEDHAM (B.P. 367,449, 21.11.30).—The Ag backing, upon which a layer of alkali metal is deposited, is superficially oxidised and the cell baked at  $\geq 160^{\circ}$ .

J. S. G. THOMAS.

Gaseous-conduction lamp electrode. F. SCHAEFER and O. S. DUFFENDACK, Assr. to AMER. SIGNS CORP. (U.S.P. 1,813,320, 7.7.31. Appl., 24.11.28).—Ni gauze coated with BaCO<sub>3</sub> is heated in an oxidising atm. until parts of the Ni and Ba are oxidised and become fused together to form a homogeneous mass.

J. S. G. THOMAS.

[Vacuum pump for evacuating] mercury-vapour rectifiers. A.-G. BROWN, BOVERI & Co. (B.P. 366,876, 28.8.31. Ger., 10.9.30).—The medium flowing in the cooling circuit of the pump is cooled in a refrigerator.

J. S. G. THOMAS.

[Insulating] coatings for filaments [of vacuum tubes] and their application. ARCTURUS RADIO TUBE Co., Assees. of J. KAUL (B.P. 367,820, 19.9.30. U.S., 24.9.29).—Filaments are coated with layers of a mixture of a saturated solution of  $Al_2(NO_3)_6$  and  $SiO_2$  (3—10%) and are heated, after the application of each layer, to reduce the Al compound to oxide. J. S. G. THOMAS.

Electron-emissive materials. F. MEYER and H. J. SPANNER, Assrs. to ELECTRONS, INC. (U.S.P. 1,817,636, 4.8.31. Appl., 6.6.27. Ger., 9.6.26).—The surface of a substance coating the core of a [Wehnelt] cathode is composed of a compound of a highly electropositive metal oxide, e.g., BaO, and a metal oxide, e.g.,  $Al_2O_3$ , which form, by chemical reaction, an amphoteric compound less acid than titanic acid. J. S. G. THOMAS.

Electrical precipitation apparatus. K. I. MAR-SHALL, ASST. to INTERNAT. PRECIPITATION Co. (U.S.P. 1,813,306, 7.7.31. Appl., 29.7.29).—A single rapping device is mounted so as to be readily movable into position to engage operatively any anvil- or impactmember of a no. of electrode units. J. S. G. THOMAS.

Electrical precipitation of suspended particles from gaseous fluids. LODGE-COTTRELL, LTD. From METALLGES. A.-G. (B.P. 367,719, 4.8.31).—The gases, after traversing one or more spiral passages between earthed walls enclosing the discharge electrodes, have their direction of flow reversed or otherwise deflected while still within the electrical field. J. S. G. THOMAS.

Electrostatic apparatus for the separation, by ionisation, of particles in suspension in a fluid. J. BREMOND (B.P. 368,091, 24.3.31. Belg., 24.3.30).— In plant comprising ionising electrodes and rotating electrodes on which deposition occurs, the latter pass through a zone which is screened from the electrostatic field or in which the field is of reduced intensity.

J. S. G. THOMAS.

Electrical gas-cleaning apparatus. LODGE-COTTRELL, LTD., Assees. of SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 366,877, 29.8.31. Ger., 30.8.30).—Collecting electrodes comprising bars of double T- or H-sections with wide abutting flanges are employed.

J. S. G. THOMAS.

Production of [corrugated] rubber diaphragms as separators for primary and secondary cells, electrolytic cells, filters, etc. H. BECKMANN (B.P. 365,971, 26.6.31. Ger., 2.8.30. Addn. to B.P. 238,870; B., 1927, 81).—To avoid the pressure of the rollers and expulsion of  $H_2O$  by evaporation, the flat sheet of rubber is partly vulcanised, and then placed between metal foil and waved, before being finally vulcanised.

H. ROYAL-DAWSON.

Electric insulating [or semi-insulating] materials. EDISON SWAN ELECTRIC CO., LTD., and F. SHERGOLD (B.P. 367,080, 10.12.30).—The surface of finished articles constructed, *e.g.*, of glass, porcelain, asbestos, uralite, is coated with an air-drying varnish, paint, or enamel, which is dried at room temp.

L. A. COLES.

Insulation of electrical conductors. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 366,502, 29.10.30).— Oily products derived from the polymerisation or condensation of olefines of low. mol. wt. in the presence of catalysts, *e.g.*, AlCl<sub>3</sub> or silicates, and having a viscosity  $\gg$  8° Engler at 20°, are arranged between the conductor and a container. [Stat. ref.] J. S. G. THOMAS.

Means for determining the percentage of moisture or paraffin or other dielectric substances in stratified or fibrous materials. STEMENS & HALSKE A.-G. (B.P. 367,839, 25.11.30. Ger., 26.4.30). —The fibrous material is introduced, as dielectric, between the plates of an electrical condenser in which the electric field is parallel to the plane of the fibres, and means responsive to changes of the capacity of the condenser are provided for regulating the drying or moistening of the material. J. S. G. THOMAS.

Electric arc furnace and crane structure. A. E. GREENE (U.S.P. 1,813,397, 7.7.31. Appl., 18.6.30).

[Device to prevent buckling of plates of] electric storage batteries. F. TEMPLE (B.P. 368,874, 23.12.30).

Electric dry cells. C. P. DEIBEL and W. G. WAITT (B.P. 368,081, 16.3.31. U.S., 15.3.30).

Electric lamps [containing spherical reflectors]. H. A. Guy (B.P. 367,930, 3.12.30).

Electric rare-gas illuminating lamps. A. LEDE-RER (B.P. 367,351, 15.11.30. Austr., 4.12.29. Addn to B.P. 364,088). Autoclave. Vibratory sifter.—See I. Graphite [for cells].  $C_2H_2$  from hydrocarbons.—See II. NH<sub>3</sub>. PbCO<sub>3</sub> and Pb(OH)<sub>2</sub>.—See VII. Glassmelting furnace. Plant for treating glaze.—See VIII. Electrodeposition of Ag.—See X. Oil-varnish [for coils etc.]. Moulded articles.—See XIII. Separation of caoutchouc. Insulating material.— See XIV.

# XII.—FATS; OILS; WAXES.

Colostrum fat. E. TCHETCHEROV (Mat. grasses Pétrole Dériv., 1931, 23, 9198—9202; Chem. Zentr., 1931, ii, 2801).—The only difference between colostrum and milk fat is a low Polenske val. Colostrum fat, 3 days after calving, had Reichert-Meissl val. 33·21, Polenske val. 1·81, xylene val.  $25 \cdot 24$ , sap. val.  $225 \cdot 2$ , I val. 39·0. The fat is preferably separated by pptn. of caseinogen with CuSO<sub>4</sub> (or boiling with HCl), filtration, drying, mixing with sand and Na<sub>2</sub>SO<sub>4</sub>, and extraction with Et<sub>2</sub>O. A. A. ELDRIDGE.

Marchand's modified method and its importance in the determination and identification of fat in foods and industrial products. S. CAMILLA (Annali Chim. Appl., 1932, 22, 83-87).-With fats containing little H<sub>2</sub>O, 1 g. is treated in a ground-stoppered 25-c.c. cylinder with 20 c.c. of anhyd. Et.O. The next day 10 c.c. of the clear supernatant Et.O are transferred to the butyrometer, together with 10 c.c. of 95% EtOH and 10 c.c. of H<sub>2</sub>O. After being shaken for some min., the butyrometer is left in a water-bath at 37-40° until no further fat drops separate. With materials containing small proportions of fat or sufficient H<sub>2</sub>O to render dehydration necessary, 2 g. are taken. Dehydration is effected by mixing the material in a crucible with 2 g. of burnt gypsum by means of a glass rod, transferring to the cylinder after 30 min., adding a fresh quantity of the gypsum (3 g. in all usually suffice) to the crucible, and transferring to the cylinder. The subsequent procedure is as described above. In all cases 0.3 c.c. of the ethereal fat layer corresponds with 0.1 g. of fat (cf. B., 1931, T. H. POPE. 133).

Soap boiling. IV. Cooling of neat soap. Y. KAWAKAMI (J. Soc. Chem. Ind., Japan, 1932, 35, 31—35 B; cf. B., 1932, 28).—For a no. of soaps (other than those containing coconut oil fatty acids, the soaps of which have disproportionally high f.p.) the relation between the setting points of the neat soap  $(T_s)$  and of its fatty acids  $(T_f)$  is linear  $(T_s = 1.34T_f - 8.5)$ . The relation  $T_s = 70 - 0.606J + 0.0062J^2$ , where J is the I val. of the fatty acids, is more general. A logarithmic (linear) temp./time relation is developed for the cooling rate of neat soap under varying conditions and the industrial application is illustrated.

E. LEWKOWITSCH.

Aqueous dry-cleaning soaps. C. L. BIRD (J. Soc. Dyers and Col., 1932, 48, 30–33).—A more detailed examination of solutions of K oleate (B., 1931, 1018) in white spirit is made. Unstable emulsions results from shaking 2 g. of K oleate (4% free oleic acid), 50 c.c. of white spirit, and 0.48 c.c. of H<sub>2</sub>O, which inverts with separation of the spirit on adding 0.52 c.c. of H<sub>2</sub>O; no inversion occurs when the K oleate contains 10% of free oleic acid. The amounts of H<sub>2</sub>O required for

dissolution and inversion (the inversion point is sharp with concns. > 0.5 g. K oleate per 50 c.c. of white spirit) of the K oleate are nearly directly proportional to the amounts of K oleate present, and the amount of H<sub>2</sub>O required for inversion is about twice that required for dissolution; these solutions resemble the vol.-reversible emulsions of the type described by Joshi (A., 1925, ii, 776), but when only H<sub>2</sub>O, K oleate, and white spirit are present the system is definitely unstable (cf. Bhatnagar, B., 1920, 551 A). Three types of behaviour are observed when H<sub>2</sub>O is absorbed by solutions in white spirit of various mixtures of K oleate, oleic acid, and methylated spirit (64 O.P.): A low-viscosity water-in-oil emulsion which does not invert is formed when large and small proportions of oleic acid and methylated spirit, respectively, are present. When the % of free oleic acid is small and that of methylated spirit is > 30-40%, a water-in-oil emulsion is formed which becomes increasingly viscous and finally inverts. If, however, the % of methylated spirit is > 50% no emulsion is formed and the absorption of a small amount of H<sub>2</sub>O causes diphasic separation. Dry-cleaning soaps required for use in conc. solution and for prolonged keeping with the aid of filtration should contain > 5% of methylated spirit and the oleic acid should be > 60% saponified; a satisfactory soap consists of K oleate 30, oleic acid 20,  $H_2O$  10, methylated spirit 4, and white spirit 36%, but this soap is not suitable for dry-dyeing since the EtOH produces increased viscosity and then inversion.

A. J. HALL.

Composition of Parkia oil. D. R. PARANJPE (J. Indian Chem. Soc., 1931, 8, 767—772).—The oil extracted from the seeds of Parkia biglandulosa by light petroleum (b.p. 60—80°) has  $d^{15} 0.9208$ ,  $n_2^{n+2} 1.4705$ , sap. val. 189.5, I val. 80.87, Polenske val. 0.25, Reichert-Meissl val. 1.12, acid val. 5.2, Ac val. 0, and Hehner val. 94.7. The unsaponifiable matter (1.11%) contains sitosterol. The total fatty acids consist of 30% of saturated and 70% of unsaturated, and are palmitic, stearic, behenic (7.9%), oleic (30.6%), and linoleic (39.4%). H. BURTON.

Lansium domesticum, Correa. I. Chemistry of the rind and pharmacodynamics of the resin obtained therefrom. P. VALENZUELA, R. GUEVARA, and S. GARCIÁ (Univ. Philippines Nat. Appl. Sci. Bull., 1931, 1, 71–91).—The rind (H<sub>2</sub>O 67, ash  $3\cdot 6$ — $4\cdot 1^{\circ}_{0}$ ) afforded a volatile oil,  $d^{15}$  0.8819,  $n_{D}^{25}$  1.5155 (0.2%), and resin ( $3\cdot 3^{\circ}_{0}$ ); no enzyme or alkaloid could be detected in the dried material.

CHEMICAL ABSTRACTS.

High-pressure hydrogenation of fatty oils. I. Action of Japanese acid clay in reacting medium. Y. TANAKA and R. KOBAYASHI (J. Soc. Chem. Ind., Japan, 1932, 35, 29–30 B).—Soya-bean or fish oil is hydrogenated very rapidly at 30 atm. pressure (temp. rising to 180°) when 0.2-0.5% of reduced Ni and 5-10% of Japanese acid clay are added separately; the unsaturated linking is apparently activated by adsorption on the clay. If the clay is used as an ordinary support for the Ni it remains inert, the special activity being destroyed by the alkaline treatment required to ppt. the NiSO<sub>4</sub> as carbonate etc.

E. LEWKOWITSCH.

Influence of hardening on the natural secondary ingredients of oil. J. A. VAN DIJK, R. T. A. MEES, and H. I. WATERMAN (Proc. K. Akad. Wetensch. Amsterdam, 1931, 34, 1206-1215).-The hardening of vegetable and animal fats under very high pressure and at a very low temp. gives rise to very different products from normal hardening, saturated fatty acids being formed in the presence of glycerides much more readily under these conditions. Cod-liver oil normally hardened loses its vitamin content, but material treated under these conditions still shows a Carr-Price reaction. It has a diminished vitamin-A content relative to the initial material, but the vitamin-D content is almost unchanged. It is suggested that this affords an industrial method of converting cod-liver oil into a less unpleasant form for consumption and into a form suitable for manufacture into margarine etc.

J. W. SMITH.

Hygroscopic equilibrium [of soap].—See I. Action of pumice etc. on oils.—See II. Deaminised gelatins as emulsifiers.—See XV. Lecithin as emulsifier. Coconut industry.—See XVI. Butter fat in cheese.—See XIX.

See also A., April, 347, Catalysed hydrogenation. 365, Synthetic glycerides. Prep. of glycerol esters. 365, Chemistry of fats. Action of KOH on halogenated palmitic acid. 366, *d*-Ricinoleates. 381, Cholesterol and its esters. Ergosterol derivatives. Lumisterol. Spinasterol and its esters.

#### PATENTS.

Continuous extraction of oils and fats. K. HILDEBRANDT (B.P. 367,563, 19.2.31).—The fatty material is dropped through one limb of a U-shaped container and conveyed by a worm through the horizontal member and up the other limb; the inlet pipes for the solvent (which flows in the counter direction) project into the interior of the container.

E. LEWKOWITSCH.

Purification of fats and oils. METALLGES. A.-G. (B.P. 366,792, 30.3.31. Ger., 2.5.30).—The oils are deacidified by treatment (preferably under vac.) with the theoretical amount (or  $\geq 5\%$  excess) of conc. alkaline lye, and after removal of the soap are decolorised etc. by treatment with dil. (8%) alkali.

E. LEWKOWITSCH. Eliminating the impurities contained in vegetable and animal oils and fats. METALLGES. A.-G. (B.P. 366,996, 14.10.30. Ger., 5.12.29).—Mucilage, lecithin, etc. are pptd. by treating the oil with, e.g., 7% of a mixture of dil. HCl (4 pts. by wt. ;  $d \cdot 1.04$ ) and (3 pts.) of an aq. solution of a metal chloride, e.g., 30%aq. CaCl<sub>2</sub> at  $\gg 50^{\circ}$ . E. LEWKOWITSCH.

Manufacture of butter, margarine, and other edible fat products. G. H. ABEL (B.P. 366,541, 5.8.30. Austral., 29.11.29).—In the final stage of manufacture the solidified plastic product (with or without salt as required) is worked at room temp. under high vac.; admixed air is removed, imparting a waxy brilliant finish to the homogeneous product. E. LEWKOWITSCH.

[Continuous] manufacture of soap and glycerin. J. B. E. JOHNSON (B.P. 367,513, 7.1.31).—A mixture of mol. proportions of fat and alkaline lye is passed in a rapidly moving stream through a narrow, externallyheated tube at  $250-300^{\circ}$  and 800-2000 lb./sq. in. pressure and the saponified mass is discharged into a low-pressure vessel (e.g., at 10-20 mm.) in which the soap collects as a dry powder, and the H<sub>2</sub>O escapes as vapour carrying with it all or some of the glycerin, according to the conditions of pressure and temp. selected for the discharge. E. LEWKOWITSCH.

Perfuming of toilet soaps. N. V. VEREENIGDE FABR. VAN STEARINE, KAARSEN EN CHEM. PROD. (B.P. 366,870, 29.7.31. Fr., 6.6.31).—The perfume composition is mixed with a liquid soap containing sulphonated fatty acids, or sulphonated naphthenic (or similar org.) acids, before working it in with the dried soap base. Antioxidants may be added to the sulphonated soap as required. E. LEWKOWITSCH.

Detergent composition. W. J. ZICK, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,812,507, 30.6.31. Appl., 1.11.26).—Free naphthenic acids ( $\gg 5\%$ ) with 0.25—0.5% of a light mineral oil ( $d \gg 0.87$ ) are incorporated in a soap substantially free from excess alkali. E. LEWKOWITSCH.

Extracting oil and other by-products from ground-nut sludge. A. F. YULL and S. R. BHATE (B.P. 366,969, 6.11.30).—The oily sludge (obtained in the filtration of expeller-expressed oil) is mixed with about 30% of 30% caustic lye at room temp., and after  $\frac{1}{2}$  hr. is boiled with about 7 vols. of 5—10% NaCl solution. The oil which separates is skimmed off, the solid residue being suitable for fertilisers; acid treatment of the aq. layer yields a ppt. of a useful caseinlike material and a filtrate from which protein matter can be recovered. E. LEWKOWITSCH.

Polymerisation of drying oils. W. L. BADGER, Assr. to SWENSON EVAPORATOR CO. (U.S.P. 1,811,290, 23.6.31. Appl., 16.8.28).—The oil is circulated rapidly through a tubular heater; the heating medium is the saturated vapour of a substance, e.g., Ph<sub>2</sub> or Ph<sub>2</sub>O (b.p. about 260°/760 mm., saturated vapour pressure < 200-250 lb.). The apparatus illustrated allows of continuous admission of fresh oil and withdrawal of the bodied oil. E. LEWKOWITSCH.

Treatment [polymerisation] of drying oils. OELWERKE NOURY & VAN DER LANDE G.M.B.H. (B.P. 366,520, 30.10.30. Holl., 30.10.29).—Bodying is accelerated by heating the oils in an inert atm. at, e.g., 280—300°, in the presence of finely-divided metallic catalysts comprising Ni, and/or Pt, Pd, Co, Cu, Fe, which may be deposited on a suitable carrier if desired. E. LEWKOWITSCH.

Manufacture of synthetic drying oil. A. M. ColLINS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,812,849, 30.6.31. Appl., 7.8.29).—Divinylacetylene (preferably containing 25-40% of a tetrameride of C<sub>2</sub>H<sub>2</sub>, formed as a by-product during the prep.) is polymerised by heating at 80—100° preferably in the presence of a solvent. The non-volatile, sol., liquid product dries to good films in air, with or without driers. Other polymerisable polymerides of C<sub>2</sub>H<sub>2</sub> or their reduction products or mixtures may be used.

E. LEWKOWITSCH.

Manufacture of wax compositions. J. Y. JOHN-SON. From I. G. FARBENIND. A.-G. (B.P. 367,339, 11.9.30).—The esters present in natural or artificial waxes are decomposed into their component alcohols and carboxylic acids (e.g., by saponification and acidification). The resulting mixture (or separate components) or the original crude wax may be bleached, preferably without chemical change (oxidation). The acids produced may be halogenated, hydroxylated, or converted into salts and/or amides, ketones, esters, anhydrides, etc. E. LEWKOWITSCH.

Lubricant.—See II. Petroleum-sec. alcohol mixtures. Wetting etc. agents.—See III. Bleaching of oils.—See VII. Waterproofing cement.—See IX. Rubber-like resins. Rubber substitute.—See XIV. Brominated fatty acids.—See XX.

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

Modern developments of inorganic pigments. N. HEATON (J. Oil Col. Chem. Assoc., 1932, 14, 373— 391).—A comprehensive survey. The effect of method of manufacture on the physical properties of pigments is examined. Diminution in particle size beyond a certain point results in reduction of opacity. Many older pigments have been superseded on account of their toxicity or lack of fastness to light and alkalis.

F. C. HARWOOD.

Dispersed red lead. P. DAUMER (Peint. Pig. Ver., 1931, 8, 1647).—Chemically pure red lead (PbO<sub>2</sub>,2PbO) is not generally found in commerce, and red lead for paint contains only about 25% of PbO<sub>2</sub> and 71.65% of Pb<sub>3</sub>O<sub>4</sub>, the remainder being PbO. This grade has min. hiding power, is toxic, and settles out of the paint medium. Dispersed red lead is a very pure colloidal substance of lower sp. gr. and contains only 2% PbO. Paints prepared from this quality do not coagulate nor settle out, but must always be mixed with pure linseed oil. F. C. HARWOOD.

Benzylcellulose dope. T. ARAKI and T. NAGAMOTE (J. Soc. Chem. Ind., Japan, 1932, 35, 68 B).—A specimen of benzylcellulose was insol. in COMe<sub>2</sub>, fairly sol. in COMeEt, sol. in CH<sub>2</sub>Ph·OH, and sol. in  $C_6H_6$  to a turbid solution which cleared on addition of 1% of EtOH. Tolyl phosphate, triacetin, castor oil, Stabilisal-A, and Palatinol-A were suitable plasticisers. A dope for application to aeroplane fabric contains  $C_6H_6$  40 c.c., EtOH 10 c.c., PhMe 20 c.c., xylene 10 c.c., COMe<sub>2</sub> 20 c.c., CH<sub>2</sub>Ph·OH 1 g., and benzylcellulose 10 g.; plasticisers and pigments may be added. The solution is stable for at least 1 year. Exposure tests indicate that a pigmented dope should be used. H. F. GILLBE.

Resins of Agathis labillardieri, Warburg, from Jappen Island. D. R. KOOLHAAS (Rec. trav. chim., 1932, 51, 388–395).—Two different types of resin were examined. The essential oil obtained by steamdistillation from A contained limonene, a little dipentene, d- $\alpha$ -pinene, and oxygenated sesquiterpene giving cadalene with Se; whilst that from B contained limonene, dipentene, l- $\alpha$ -pinene, and a little sesquiterpene alcohol. Resin A was a balsam, whilst B resembled copal. A. A. LEVI. Oleoresin production from long-leaf pine defoliated by fire. E. GERRY (J. Agric. Res., 1931, 43, 827-836).—Long-leaf pines show strong recuperative powers after fire damage, the yield of oleoresin after 2 years approximating to that of similar unscorched trees even though subjected to commercial turpentining during the interval. W. G. EGGLETON.

Synthetic resins from ammonium thiocyanate and guanidine. K. KELLER and W. NÜSSLER (Ber. Ges. Kohlentech., 1932, 4, 34-44).-Guanidine prepared from NH<sub>4</sub>CNS (cf. B., 1932, 414) forms condensation products with CH<sub>2</sub>O in the same way as does urea. Guanidine thiocyanate and carbonate, NH<sub>4</sub>CNS, and a mixture of the latter with CS(NH2)2, its decomp. product, all give resins of slightly varying properties, the first being most satisfactory in use. In each case 3 mols. of the starting material were shaken with 8 mols. of CH<sub>2</sub>O (40% solution) and heated under reflux, then distilled in vacuo at 60°, and the syrupy residue was held at 70-80° in air for 2-3 days. Polymerisation is hastened in acid solution, but too rapid treatment causes air inclusions. The resins produced are cryst. or amorphous and can be pressed. The presence of H<sub>2</sub>O renders the product opaque and liable to crack, but this is prevented if glycerin be added to the mixture. A transparent yellow resin is then obtained, at first soft but hardening with time, more satisfactory results being given by treatment in an autoclave at 90°/25 atm. in an atm. of No. The powder can be worked and moulded with or without fillers and has good insulating properties. Examples with working details are given. C. IRWIN.

# Resin from Lansium domesticum.-See XII.

See also A., April, 326, X-Ray examination of Pb oxides. 351, Rhombic  $PbCrO_4$ . 357, Micro-extraction [of paint films]. 397, Elemic acid from elemi resin.

#### PATENTS.

Production of anti-rust paints. K. WINKLER (B.P. 365,366, 16.2.31. Switz., 14.2.30).—The byproduct smoke dust obtained in roasting S ores, *e.g.*, pyrites containing Pb and As, is mixed with linseed-oil varnish or other vehicle. S. S. WOOLF.

Coating surfaces of containers for oils and other liquids. A./S. FREDRIKSSTAD SAAPEFABRIK (B.P. 365,628, 20.10.30. Nor., 19.10.29).—The surfaces are coated with a composition of CaCO<sub>3</sub> (and, if desired, kieselguhr) and water-glass containing  $2 \cdot 5 - 4 \cdot 5$  (preferably > 3) pts. by wt. of SiO<sub>2</sub> to 1 pt. of Na<sub>2</sub>O or K<sub>2</sub>O, and the coating is rendered insol. by heat, *e.g.*, 3 hr. at 120°, or blow-lamp treatment. S. S. WOOLF.

Manufacture of [colour] lakes. E. I. DU PONT DE NEMOURS & Co. (B.P. 365,943, 27.5.31. U.S., 27.5.30).—A basic dye, one or more aliphatic amines, e.g., NHBu<sup>a</sup><sub>2</sub>, NPr<sup>a</sup><sub>3</sub>, and a complex inorg. acid or its components, e.g., phosphotungstic acid, are brought together in an acid medium. If desired, the lake may be pptd. on a substratum, e.g., BaSO<sub>4</sub>. S. S. WOOLF.

(A) Cellulose nitrate lacquer. (B) Lacquer for application by brushing. H. FINKELSTEIN, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,801,340-1, 21.4.31.

Appl., [A, B] 13.7.28. Ger., [B] 22.12.24).—(A) Such a lacquer, which contains a mixed solvent comprising 80—20 pts. of ethylene glycol monomethyl ether and 20—80 pts. of a solvent of the type  $C_nH_{2n}(OH)OR$ [where R is an alkyl group ( $C_4$  or less), n is 2, 3, or 4, and the mol. contains  $\leq 5$  C atoms], e.g., ethylene glycol monobutyl ether, forms clear non-blushing coatings. (B) The lacquer consists of 8—25 pts. of a low-viscosity collodion cotton (of which  $\leq 15\%$  is sol. in 96% EtOH), a cyclic ketone (cyclohexanone) or vinyl ester resin, a plasticiser, 25—60 pts. of a solvent of the type described in (A), e.g., propylene glycol monopropyl ether, and 67—10 pts. of a solvent (alcohols) containing  $\leq 30\%$  EtOH. E. LEWKOWITSCH.

Compositions of matter containing cellulose derivatives. BRIT. CELANESE, LTD. (B.P. 366,586—7, 7.11.30. U.S., 7.11.29).—( $\Lambda$ ) Mixtures of cellulose derivatives, e.g., the acetate, and synthetic resins obtained by condensing a chloroketone, e.g., chloroacetone, with a phenol in the presence of an acid catalyst, e.g., HCl, together with suitable solvents, diluents, and plasticisers, are claimed as lacquers, adhesives, films, etc. (B) Dihydroxybenzophenones are used as plasticisers for compositions containing, e.g., cellulose acetate, synthetic resins, e.g., of the diphenylolpropane-CH<sub>2</sub>O type, and solvents. 25—150 wt.-% of plasticiser (on the cellulose derivative) is used.

S. S. WOOLF.

Manufacture of synthetic [phenolic] resin. E. E. NOVOTNY, ASST. to J. S. STOKES (U.S.P. 1,802,390, 28.4.31. Appl., 14.7.26. Cf. U.S.P. 1,767,696; B., 1931, 262).—Potentially reactive resins consist of phenolaldehyde condensation products containing relatively small amounts of resorcinol as hardening agent and an active :CH<sub>2</sub> compound. E. LEWKOWITSCH.

Artificial resinous substances. IMPERIAL CHEM. INDUSTRIES, LTD., W. J. R. EVANS, and R. HILL (B.P. 367,001, 1.11.30).—A natural resin, e.g., rosin, is heated with PhOH-CH<sub>2</sub>O type and "glyptal" type condensation products, either (or both) of which is (are) prepared *in situ*. The products, which have high m.p. and are sol. in drying oils, are suitable as varnish ingredients. S. S. WOOLF.

Preparation of resinous condensation product. E. S. DAWSON, JUN., ASST. to GEN. ELECTRIC Co. (U.S.P. 1,803,174, 28.4.31. Appl., 23.5.25. Renewed 28.11.28).— Drying-oil fatty acids, e.g., elecostearic acid (70 pts.), are heated at  $175-185^{\circ}$  with the product formed by condensing glycerol (92 pts.) with phthalic anhydride (200 pts.) in the presence of a small amount of  $H_2SO_4$  at 120°. The dark red resin hardens rapidly under heat. E. LEWKOWITSCH.

Manufacture of combinations of polyvinyl esters and fattyoils. CONSORT. F. ELEKTROCHEM. IND. G.M.B.H., Assees. of A. EIBNER (B.P. 367,102, 31.12.30. Ger., 31.12.29).—A vinyl ester, e.g., acetate, butyrate, is polymerised by known methods in the presence of a polymerised fatty oil or a low disperse phase of such oil [obtained by removing the non-polymerised portion therefrom by treatment with an aliphatic alcohol (C<sub>3</sub> or above), or with an ester from an aliphatic acid and a monohydric alcohol, e.g., Et malonate], in the presence,

if desired, of solvents. Pigments, fillers, etc. may be incorporated. S. S. WOOLF.

[Oil-proof resin] composition. W. H. WRIGHT, ASST. to SCHENECTADY VARNISH CO. (U.S.P. 1,802,071, 21.4.31. Appl., 13.10.27).—Covered electrical (transformer) coils are impregnated with a melted mixture of rosin and gum accroïdes (with or without 5—10% of castor oil, stearic acid, etc.). The product can also be used in solution in alcohols, solvent naphtha, etc.

E. LEWKOWITSCH.

Moulded articles containing [metal] inserts. BAKELITE CORP., Assees. of F. GROFF (B.P. 367,045, 18.11.30. U.S., 20.11.29).—Metal parts that are to be inserted in a moulding mixture, e.g., PhOH-CH<sub>2</sub>O, are coated with a reactive phenol-oil resinoid, e.g., a phenoltung oil condensation product, of better insulating qualities than the rest of the article, and, if desired, are baked before insertion. The composite articles, e.g., distributor heads for ignition systems, are finally consolidated by heat and pressure. S. S. WOOLF.

Decoration of materials [particularly floor coverings]. ARMSTRONG CORK CO., Assees. of G. ROTTMUND (B.P. 369,370, 5.3.31. U.S., 7.4.30).

Petroleum-sec. alcohol mixtures.—See III. Solubilised nitrocellulose.—See V. PbCO<sub>3</sub> and Pb(OH)<sub>2</sub>. —See VII. Laminated glass.—See VIII. Drying oils. —See XII. Rubber-like resins.—See XIV.

# XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Guanidine salts.-See III.

See also A., April, 361, Polymerisation of isoprene and butadiene derivative. 398, Oxidation of caoutchouc etc.

#### PATENTS.

Separation of caoutchouc from aqueous caoutchouc dispersions with aid of alternating current. L. MELLERSH-JACKSON. From SIEMENS-ELEKTRO-OSMOSE G.M.B.H. (B.P. 366,652, 10.12.30).—Electrodeposition of rubber, e.g., for the coating of wires, is effected with electrodes of different material, e.g., Zn and C, and substantially different in size (e.g., at least 1:10) connected with a source of a.c., preferably through a regulating transformer. The addition of a suitable electrolyte, e.g., NH<sub>4</sub>OAc, is beneficial. D. F. Twiss.

Manufacture of materials composed of rubber, balata, or the like, for use as leather substitutes etc. S. YAMAMOTO (B.P. 367,317, 19.11.31).—Rubber, balata, etc. are reinforced with the bared vascular network of bark or leaves of plants, especially the bark of the Kayutarap (*Artocarpus Kunstleri*), which is embedded in the material. D. F. TWISS.

Treatment of rubber. NAUGATUCK CHEM. Co., Assees. of W. A. GIBBONS (B.P. 367,183, 23.2.31. U.S., 8.3.30).—The physical properties of rubber are improved by adding, during mastication and before compounding, a material, e.g., H<sub>3</sub>PO<sub>4</sub>, salicylic acid, NH<sub>4</sub> chloroacetate, or Zn(NO<sub>3</sub>)<sub>2</sub>, capable of decomposing alkali proteinates or alkali soaps, sufficient being added to reduce the  $p_{\rm H}$ of the aq. extract to 7 or less. This treatment facilitates mastication and improves the flexing ability of the vulcanised product. D. F. TWISS.

STORIEGH THOMAS UNITAL

Treatment of unvulcanised rubber surfaces. W. A. GIBBONS, ASST. to MORGAN & WRIGHT (U.S.P. 1,817,363, 4.8.31. Appl., 14.10.27).—The tackiness of such surfaces is reduced by application of a material decomposable at vulcanisation temp. into portions which are volatile and rubber-sol. respectively, so that substantially no residue remains after vulcanisation. The preferred agents are volatile salts of org. acids above  $C_7$ , e.g., NH<sub>4</sub> stearate, applied in aq. solution.

D. F. Twiss.

Manufacture of insulating material [containing regenerated rubber]. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of E. W. SCHWARTZ (B.P. 367,649, 15.5.31. U.S., 16.5.30).—Mineral fillers and mineral rubber are incorporated in the waste rubber immediately after the "devulcanising" process so that in the subsequent refining operations of the "reclaiming" process their uniform dispersion is effected. New rubber, softening agents, vulcanisation accelerators, and S are added subsequently. D. F. Twiss.

Vulcanisation of rubber. RUBBER SERVICE LABS. Co., Assees. of R. L. SIBLEY (B.P. 366,554, 2.10.30. U.S., 28.5.30. Addn. to B.P. 287,001; B., 1928, 377).—A vulcanisation accelerator is prepared by combining an aromatic primary amine and a 2-mercaptoarylthiazole, e.g., by melting together 2:4-diaminodiphenylamine (1 mol.) and 2-mercaptobenzthiazole (2 mols.), and treating the product, preferably in powder form, with an aldehyde, e.g., with 1 mol. of an aliphatic aldehyde (below  $C_8$ ) such as MeCHO or crotonaldehyde. D. F. Twiss.

Treatment of rubber. [Vulcanisation accelerators.] NAUGATUCK CHEM. Co., Assees. of W. E. MESSER (B.P. 363,862, 20.1.31. U.S., 18.3.30).—As accelerators there are used thiocarbonic acids derived from pipecoline, piperidine, or other fully hydrogenated nitrogenous heterocyclic base, and in which the H of the SH group is substituted by Zn, dinitrophenyl, benzyl, etc.; or the corresponding thiuram mono- or di-sulphides are used. C. HOLLINS.

Vulcanisation of rubber. B. F. GOODRICH CO., Assees. of G. OENSLAGER (B.P. 367,305, 25.8.31. U.S., 19.1.31).—For the coating of surfaces with hard rubber (vulcanite) a rubber layer of suitable composition containing an ultra-accelerator is applied and, after being covered with metal foil, preferably Al, is vulcanised by contact with an aq. medium at approx. 100°.

D. F. Twiss.

Preservation of rubber and products obtained thereby. GOODYEAR TIRE & RUBBER Co. (B.P. 367,494, 13.12.30. U.S., 16.1.30).—Rubber is vulcanised in the presence of acenaphthene containing a OH, NH<sub>2</sub>, alkylamino-, or arylamino-radical attached to the  $C_{10}H_8$  nucleus. 3-Aminoacenaphthene and 3- $\beta$ -naphthylaminoacenaphthene, obtained from this by heating with  $\beta$ -naphthol at 260—290°, are especially indicated. D. F. Twiss.

Age-resisting vulcanised rubber compound. J. R. INGRAM, Assr. to RUBBER SERVICE LABS. Co. (U.S.P. 1,816,851, 4.8.31. Appl., 15.4.30).—Rubber is vulcanised in the presence of a reaction product of an aminodiphenyl and an aryl hydroxide, *e.g.*, the compound (m.p. 125—128°) obtained by heating mol. proportions of *p*-aminodiphenyl and  $\beta$ -naphthol at 180—200°.

D. F. TWISS. Manufacture of rubber substitutes and products derived therefrom. J. T. SHEVLIN. From Soc. ITAL. PIRELLI (B.P. 367,378, 13.8.30).—Oil, *e.g.*, colza oil, fully vulcanised with S at a high temp., is emulsified while still fluid, *e.g.*, at 90—100°, with an aq. solution of a dispersing agent. The emulsion may be blended with compounded or uncompounded rubber latex and the mixture applied to methods of manufacture already known for use with latex. D. F. TWISS.

**Preparation of rubber-like resins.** H. A. BRUSON (B.P. 363,859, 19.1.31. U.S., 8.7.30).—Castor oil, glycerol (or other tri- or poly-hydric alcohol), and an aliphatic dibasic acid  $(CH_2)_n(CO_2H)_2$ , where *n* is 4—8, with or without addition of drying oil, are heated at 175—200° or above. Examples are castor oil and glycerol with sebacic, azelaic, suberic, pimelic, or adipic acid. The products are vulcanisable rubber-like masses which become sol. in org. solvents when milled. C. HOLLINS.

Manufacture of artificial rubber-like masses. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 366,550 and 366,944, 30.9.30).—αγ-Butadiene and its homologues are polymerised in the presence of (A) substances of emulsifying and salt-like character, e.g., Na stearate, or (B) non-saline substances (e.g., proteins or saponin) and of a highly chlorinated aliphatic compound, e.g., CCl<sub>3</sub>·CO<sub>2</sub>H, or of a colloidal metal oxide, e.g., colloidal pyrolusite. The proportion of H<sub>2</sub>O present, however, is at the most 10% of the butadiene hydrocarbon and insufficient for complete dissolution of the emulsifying agent. In (A) substances known to accelerate the emulsification-polymerisation process, e.g., amyl ether or C2Cl6, may also be added. It frequently suffices to employ the constituents of the mixture in the atmospherically moist state. D. F. Twiss.

Brake linings.—See I. Rubberised sheet materials.—See VI. Rubber-coated concrete. Roads etc. Floor coverings.—See IX. Rubber diaphragms.—See XI.

# XV.-LEATHER; GLUE.

Chinese vegetable tanning materials. Y. C. TAO (J. Soc. Leather Trades' Chem., 1932, 16, 102—104).— The tannin and non-tan contents of the following materials are given, respectively: Quercus serrata (acorn cup) 16.43, 8.07; (bark) 2.94, 1.39; Salix babylonia 7.51, 4.92; Pinus massoniana 3.21, 1.08; Punica granatum (bark) 11.05, 5.15; (rind of fruit) 23.03, 21.31; Zizyphus sativa (fruit) 23.35, 22.62; Chinese galls 52.22, 19.62; Platycarya strobilacea, (fruit) 24.97, 9.15; Castanea mollissima (bark) 5.71, 3.89; Trapa natans (rind of fruit) 10.45, 13.03%. The tannin content of certain other materials, e.g., Rhus javanica, was very low. D. WOODROFFE.

Some uses of the microscope in leather manufacture. E. C. LINE (J. Soc. Leather Trades' Chem., 1932, 16, 93—102).—The microscope is recommended for controlling the soaking, liming, and bating of raw skins, the grinding of pigments for pigment finishes, the

investigation of faulty leathers, and in conjunction with various suitable solvents in determining the nature and no. of the coats constituting the films on patent and nitrocellulose-finished leathers. D. WOODROFFE.

Interpretation of photomicrographs [of skins and leathers]. D. J. LLOYD and R. H. MARRIOTT (J. Soc. Leather Trades' Chem., 1932, 16, 57-93).-The weave pattern of hides and skins is affected by the various leather manufacturing processes and the different weave patterns can be roughly classified into vertical, high-angle, medium-angle (about 45°), low-angle, and horizontal. Each different class of leather, examples of which are represented photomicrographically, is associated in its best quality with a particular type of weave. The angle of weave pattern is affected by the part of the skin from which the leather is derived. The quality of the leather is affected by the regularity of the weave pattern, which in turn may be disturbed by bad tanning processes. Irregular weave pattern is not usually observed in best quality leathers. The degree of compactness of the weave pattern also is an important factor in good leathers. Thickly outlined fibres are evidence of insufficient opening up of the structure, and thinly outlined fibres are observed when they have been well opened up. Good separation of the fibres into fibrils is found in full, flexible, light leathers and in thoroughly tanned heavy leathers of good wt. yield. Fibres and fibrils should still be held together in bundles. D. WOODROFFE.

Adhesion of glue. M. F. WITHERELL (J. Physical Chem., 1931, 35, 3583-3595).-The adhesion between animal glue and alundum grit was studied, this being assumed to be measured by the strength under tension of briquettes moulded from a mixture of grit and glue solution and then dried. No satisfactory relation was established between the capillarity attraction of the grit and the adhesion. Adhesion was improved by heating the grit to redness and quenching in aq. Na<sub>2</sub>CrO<sub>4</sub>, or, better, aq. Na silicate. When NaOH was added to the silicate there was less improvement in adhesion, but even better adhesion resulted when the grit was boiled in aq. Na silicate for 4 hr. Addition of a small quantity of glycerin to the glue increased the adhesion to untreated grit by 36-42%, but did not improve adhesion to treated grit. Adhesion of glue to grit is considered sp. rather than mechanical. The improvement in adhesion caused by treating the grit is attributed to removal of dirt and adsorbed air from the surface of the grit. F. L. BROWNE (c).

Deaminised gelatins of different origins as emulsifying agents. J. KNAGGS (J.S.C.I., 1932, 51, 61-64 T).—Highly purified gelatins extracted from the tissues of mammals and fish were treated with HNO<sub>2</sub>, and also with CH<sub>2</sub>O. Precautions were taken to keep both the inorg. and org. impurities at a min. By replacing the free NH<sub>2</sub>-groups in these gelatins by either OH or N:CH<sub>2</sub> their power for emulsifying an oil was considerably changed. Deaminisation of gelatins with HNO<sub>2</sub> decreases the relative viscosity, but increases their emulsifying properties for oils. With CH<sub>2</sub>O generally the relative viscosity is increased considerably, and the drop no. reduced. The degree of these changes in properties depends on the precursors from which the gelatin was extracted and its previous treatment. The results are further evidence in support of the view that gelatins derived from different animals and from different tissues have different mol. structures.

Testing of agar and gelatin jellies. F. L. DE BEUKELAER (J.S.C.I., 1932, 51, 94 T).—The omission by Lockwood and Hayes (B., 1931, 600) to mention the Bloom gelometer is noted.

#### PATENTS.

Unhairing and bating of [raw] skins. A. R. BOIDIN and I. A. EFFRONT (U.S.P. 1,812,921, 7.7.31. Appl., 17.10.29. Fr., 29.10.28).—The skins are soaked for 24 hr. at  $> 18^{\circ}$  in a solution of low alkalinity ( $p_{\rm H}$  8—9) containing a small amount of unhairing enzyme; then a further quantity of the unhairing enzyme is added, together with sufficient acid to render the liquor slightly acid. D. WOODROFFE.

Imitation suède leather. W. BETAMBEAU (B.P. 366,754, 4.3.31).—Powdered cotton or its equiv. is sprayed on to one side of a fabric base treated with adhesive, and a liquid rubber composition is applied to the other side of the base so that it penetrates to the cotton particles. D. WOODROFFE.

Destruction of moths and their eggs in furs. F. KNOTH (B.P. 368,179, 6.7.31).—An aq. solution containing As, green soap, and NaCl is mixed with methylated spirit and applied to the pelt side of furs. D. WOODROFFE.

Manufacture of a water-resistant adhesive. H. P. BANKS (U.S.P. 1,813,377, 7.7.31. Appl., 26.1.29).— Furfuraldehyde is added to adhesives comprising aq. suspensions of oleaginous seed (soya-bean) flour and material containing casein. L. A. COLES.

Manufacture of a water-resistant double-decomposition adhesive. G. DAVIDSON and I. F. LAUCKS (U.S.P. 1,813,387, 7.7.31. Appl., 25.1.29).—In an adhesive comprising an oleaginous seed flour and CS<sub>2</sub> [or, e.g., Na<sub>2</sub>CS<sub>3</sub>, K xanthate, thiocarbanilide,  $CS(NH_2)_2$ ], at least 50% of the desired alkalinity is formed on the addition of H<sub>2</sub>O by the double decomp. of NaF and Ca(OH)<sub>2</sub>. L. A. COLES.

Manufacture of a water-resistant, animal-protein adhesive. H. F. RIPPEY, C. N. CONE, G. DAVIDSON, I. F. LAUCKS, and H. P. BANKS (U.S.P. 1,814,768, 14.7.31. Appl., 24.4.26).—CS<sub>2</sub>, or a xanthate, thiocarbonate, or thiocarbanilide, is added to an adhesive mixture comprising an animal protein (*e.g.*, glue, albumin, milk-casein) in a neutral or alkaline medium. L. A. COLES.

Cementing composition for preparing laminated material. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of A. COHEN (B.P. 366,889, 26.10.31. U.S., 24.10.30).— Layers of wood, cloth, fabric, paper, etc. are cemented together with a prep. derived from blood-albumin or other albuminous substances, CH<sub>2</sub>O, NH<sub>3</sub>, oxide of Co (or of U or Cr), with or without MnO<sub>2</sub>.

D. WOODROFFE.

Apparatus for tanning skins in stationary vessels or pits. J. E. SCHUH (B.P. 369,526, 24.9.31, Ger., 29.9.30).

British Chemical Abstracts-B.

440

Rubberised sheet materials.—See VI. Byproduct from ground nut.—See XII. Leather substitutes.—See XIV.

# XVI.—AGRICULTURE.

Available water [in soil] and the wilting of plants. A. D. MOINAT (Plant Physiol., 1932, 7, 35-46). —The permanent wilting of plants is not a criterion of a sp. soil- $H_2O$  content. The  $H_2O$  content at which wilting occurs is influenced by the general level of available  $H_2O$  in the soil during the growth of the plant. Transference of  $H_2O$ -cultured plants to 0.08M-NaCl caused wilting, which was rare when 0.07Msolutions were used. A. G. POLLARD.

Wilting and withering of grasses in greenhouse cultures as related to the water-supplying power of the soil. J. D. WILSON and B. E. LIVINGSTON (Plant Physiol., 1932, 7, 1—34).—Measurements of the  $H_2O$ supplying power of the soil by the porous-point method, and of evaporation rates by an atmometer are used to compare the drought-resistance of various grass species. A. G. POLLARD.

Mineralisation of the humus-nitrogen in lowmoor soils under grass. R. REINCRE (Zentr. Bakt. Par., 1932, II, 85, 348–359).—There is a general parallellism between the activity of nitrifying organisms in these soils and the N intake of the pasture. The N requirement of the grass was met, but no appreciable accumulation of NO<sub>3</sub>' in the soil was observed. Nitrification was greatest in spring and autumn and is restricted by the low  $H_2O$  content of the soil in summer.

A. G. POLLARD.

Relative effectiveness [in soils] of limestone particles of different sizes. T. L. LVON (Cornell Univ. Agric. Exp. Sta. Bull., 1931, No. 531, 13 pp.).—Finelyground limestone (< 200-mesh) had a rapid action on acid soils, but the resulting crop increases were mainly confined to the years immediately following the application. Very coarse particles (5—10-mesh) showed no cumulative effect in the 10 years under examination. Most satisfactory results are indicated with samples of soft limestone ground completely to pass 10-mesh. A suitable proportion of finer particles is thereby obtained. Hard stone should be more finely ground.

A. G. POLLARD. Influence of sulphur on the nature of soils. I. Effect on reclaimed soils. A. ITANO and A. MATSUURA (Ber. Ohara Inst. landw. Forsch., 1931, 5, 163—170).— Applications of S decreased the  $p_{\rm H}$  of soils, increased the proportion of dialysable salts, and facilitated the removal of NaCl. Elementary S was more effective than CaSO<sub>4</sub> or (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, but differences between the action of two forms of S were not apparent. A. G. POLLARD.

Manganese in Texas soils and its relation to crops. E. C. CARLYLE (Texas Agric. Exp. Sta. Bull., 1931, No. 432, 37 pp.).—Among numerous soils examined, applications of  $MnSO_4$  in only one case increased and in several cases decreased crop yields. In sand cultures the intake of Mn by maize and cotton was proportional to the amounts supplied. Correlation between the amount of acid-sol. Mn in soils and the intake by plants was not close. Applications of complete fertilisers increased the Mn intake of crops. Clay soils have higher Mn contents than sandy ones. A. G. POLLARD.

Rôle of aluminium in decalcified soils. M. TRÉNEL (Ernähr. Pflanze, 1931, 27, 385—389; Chem. Zentr., 1931, ii, 2772).—Diseased roots of fir grown on acid soils of N.W. Germany showed a considerable increase in Al content ( $8\cdot3$  to  $41\cdot6\%$   $Al_2O_3$ ) and a large decrease in CaO content ( $22\cdot5$  to  $6\cdot9\%$  CaO). Experiments with permutit suggest that hydrated Al silicate is half debasified and then decomposed into a gel of hydrated  $Al_2O_3$ and SiO<sub>2</sub>; further debasification leads to a residue consisting chiefly of SiO<sub>2</sub>. A. A. FLDRIDGE.

Determination of soil moisture by the method of multiple electrodes. W. H. McCorkle (Texas Agric. Exp. Sta. Bull., 1931, No. 426, 20 pp.).—Conductivity measurements with multiple C electrodes permit the determination of variations in soil-H<sub>2</sub>O content in localised areas. A. G. POLLARD.

Effect of liming on the structure of podsol soils. N. P. REMEZOV and O. I. IZMAILOVICH (Trans. Sci. Inst. Fertilisers, Russia, 1930, No. 77, 64—79).—CaSO<sub>4</sub> decreased the dispersion soon after it was applied; a similar effect was observed the next year only with CaCO<sub>3</sub> and Ca(OH)<sub>2</sub>. CHEMICAL ABSTRACTS.

Effect of lime, superphosphate, and potash on reaction of soil and growth and composition of lucerne. M. C. SEWELL and W. L. LATSHAW (J. Amer. Soc. Agron., 1931, 23, 799-814).—Reduction of soil acidity effected by superphosphate is of brief duration. Good yields were obtained by addition of superphosphate and CaO; the rate of absorption of some of the primary nutritive elements was thereby changed. Application of CaO decreases the K content of lucerne.

CHEMICAL ABSTRACTS.

Movement and fixation of phosphates in relation to permanent pasture fertilisation. A. R. MIDGLEY (J. Amer. Soc. Agron., 1931, 23, 788—799).— Superphosphate applied as a surface dressing moves downward very slowly;  $P_2O_5$  is not readily removed by leaching. NaNO<sub>3</sub> increases the movement of superphosphate through soils, whilst  $K_2SO_4$  and  $(NH_4)_2SO_4$ slightly decrease it. CHEMICAL ABSTRACTS.

Phosphate penetration in field soils. R. E. STEPHENSON and H. D. CHAPMAN (J. Amer. Soc. Agron., 1931, 23, 759-770).—Heavy applications of  $P_2O_5$ appear to cause more rapid penetration than more numerous lighter ones; little penetration took place in very heavy soils. Deeper penetration was obtained with  $P_2O_5$  from superphosphate and manure than from bone meal. CHEMICAL ABSTRACTS.

Potash fertilisers on sandy loam soils. V. N. PROKOSHEV (Udobr. Urozhai, 1931, 3, 432-443).—A record of experiments at Solikamsk on podsolised soil. K alone was ineffective on hay crops. With root crops K, when added to N and P, gave large increases. CaO did not affect K effects. CHEMICAL ABSTRACTS.

Aspergillus method for determining potash [in soils]. H. NIKLAS and H. POSCHENRIEDER (Ernähr. Pflanze, 1932, 28, 86-88).—Technique is described and limiting vals. are recorded. In soils rich in CaCO<sub>3</sub> abnormally high yields of mycelium are obtained and limiting vals. must be reduced accordingly.

A. G. POLLARD.

Rapid practical method of determining the oxidising power of arable soil. A. C. GALLETTI (Annali Chim. Appl., 1932, 22, 81-83).-0.5 g. of the soil, ground to pass a 0.5-mm. sieve, is rotated gently on an 8-cm. clock glass with 2 c.c. of aq. (apparently saturated) benzidine solution and left for 30 min. The depth of the blue colour developing indicates the oxidising power of the soil. T. H. POPE.

Rice-field studies. IV. Relation of the ammonifying, nitrifying, and denitrifying powers of soil to the yield of rice. A. ITANO and S. ARAKAWA (Ber. Ohara Inst. landw. Forsch., 1931, 5, 151-161).—Seasonal changes in ammonification in soils are not related to fertiliser treatment or cropping, except that org. composts favour the activity of this process. Denitrification is more active in soil treated with rice straw, and nitrification in that receiving org. compost. Relationships between the activity of these bacterial changes and crop yields are apparent only where crops are heavy. A general parallellism between nitrification and the buffer capacity of the soil is indicated. A. G. POLLARD.

Influence of potash on root development in summer barley. J. GÖRBING and HESSBERG (Ernähr. Pflanze, 1932, 28, 81-86).—The liming of acid soils increased root production and tillering in barley. Similar effects produced by K fertilisers were apparent 20 days after sowing and reached a max. 110 days after sowing. A. G. POLLARD.

Effect of boron on the growth of lettuce. J. S. McHARGUE and R. K. CALFEE (Plant Physiol., 1932, 7, 161-164).—B is essential to the growth of lettuce, and a deficiency results in the burning of leaves and the death of growing points at an early stage of development. In plant metabolism B cannot be replaced by Mn, Cu, Zn, Ni, Co, Ba, Sr, I, or As. A. G. POLLARD.

Green manuring of paddy. L. LORD (Trop. Agric., 1932, 78, 3—10).—Late ploughing-in of green manure is preferable and leads to more complete ammonification. Simultaneous applications of superphosphate, with large dressings, or of  $NH_4$  phosphate, with smaller dressings, are recommended. A. G. POLLARD.

Analyses of by-products of the coconut industry. A. W. R. JOACHIM and S. KANDIAH (Trop. Agric., 1932, 78, 15—17).—Details are recorded of manurial contents of residual oil cakes and sweepings, of the feeding val. of various cakes, and of characteristic vals. of several grades of oil. A. G. POLLARD.

Factors affecting the efficiency of contact insecticides. II. Chemical and toxicological studies of pyrethrum. A. HARTZELL and F. WILCOXON (Contr. Boyce Thompson Inst., 1932, 4, 107—117).—A direct spraying method for determining the toxicity of pyrethrum extracts to aphis is described. The relationship between pyrethrin content and toxicity is not a linear one. Losses in pyrethrin content and in toxicity of samples due to storage, exposure to heat, sunlight, etc. were parallel. Conc. pyrethrum extracts, exposed to sunlight in thin layers, showed a lowered toxicity accompanied by the separation of insol, material. Pyrethrum has no lasting protective action when sprayed on plants. No significant loss of toxicity occurred when MeOH extracts of the flowers were heated under reflux for several hr. Pyrethrum concentrates were toxic to a number of insects when applied externally to the integument and in regions far removed from vital organs. Toxic symptoms were immediate when injections were made in the body cavity. A. G. POLLARD.

Evaluation of sulphur suspensions used in spraying. R. M. WOODMAN (J.S.C.I., 1932, 51, 103— 107 T).—An examination of the methods of analysis of S suspensions has been made, attention being given to determinations of total solids, total S, non-combined S, and protective colloids. The properties which a good S suspension should possess are discussed, and the importance of the free or non-combined S is pointed out. Sedimentation experiments are made on three S suspensions.

Lecithin as an emulsifier. The drop method of determining emulsion type. R. M. WOODMAN (J.S.C.I., 1932, 51, 95-100 T).-Aq. lecithin dispersions form dual or H<sub>2</sub>O-in-oil types of emulsions with various oils, and are therefore unsuitable for the prep. of insecticidal and ovicidal spraying emulsions. The formation of dual types with such fat solvents as CHCl<sub>3</sub>, CCl<sub>4</sub>, PhMe, C<sub>6</sub>H<sub>6</sub>, etc., where it is reasonable to assume non-formation of solute-solvent complexes with the lecithin, suggests that the partition of the emulsifier between the liquid phases probably plays a large part in the mechanism of dual emulsion formation in a system. Drop tests made on emulsions immediately after their formation may be misleading; the surest drop tests are those carried out both in H<sub>2</sub>O and in the oil phase concerned on the aged emulsion or portion of an emulsion.

Seed-treatment experiments with yellow dent maize. J. R. HOLBERT and B. KOEHLER (U.S. Dept. Agric. Tech. Bull., 1931, No. 260, 63 pp.).—Successful use of dusts (largely Hg preps.) is described for the control of *Diplodia* and *Gibberella* and other soil- and seed-borne diseases. Seed treatment is frequently followed by stimulation of the growth of seedlings, particularly in the early stages. A. G. POLLARD.

Adhesion of [seed] pickling dusts. A. WINKEL-MANN (Angew. Chem., 1932, 45, 238-241).—No relationship was observed between the amount of Cu, Hg, and As dusts adhering to seed and their  $H_2O$  content or wt. per 1000 grains. Wheat infected with *Tilletia tritici* retained more dust than clean grain and a smaller proportion was lost during drilling. This effect was due, not to the increased surface area of the infected wheat, but to the high retentive power of the fungus spores which appear to induce the formation of larger aggregates of particles. Cu carbonate dust differed from other materials in this respect, and is therefore unsuitable for testing mixing machines etc. A. G. POLLARD.

Onion spraying and dusting experiments. W. L. DORAN and A. I. BOURNE (Massachusetts Agric. Exp. Sta. Bull., 1931, No. 279, 176-185).—The onset of "blast" in onions is delayed, but not prevented, by Bordeaux mixture. No injury resulted from the use of 4:4:50 or 8:4:50 mixtures, but Cu-CaO dusts were

sometimes harmful to the onions without markedly affecting the disease. Where nicotine sprays are used against onion thrips, a combination with Bordeaux mixture is suggested. A. G. POLLARD.

Control of the cherry case-bearer (Coleophora pruniella) by dormant and other sprays. R. HUT-SON (J. Econ. Entom., 1932, 25, 116—120).—Rather high concns. of oil and tar washes are needed for dormant spraying. Against active larvæ, nicotine and derrisol showed promising results. A. G. POLLARD.

Use of insecticides on pineapple plants in Hawaii. W. CARTER (J. Econ. Entom., 1931, 24, 1233—1242).—Of numerous insecticides examined, oil emulsions alone were effective. A. G. POLLARD.

Storage of superphosphate.—See VII. Apples. Feeds and egg quality.—See XIX.

See also A., April, 353, Determining Cl' in soils. 360, Peat soils. 437, Microbiology of soils. Fixation of protein by soils. 438, Potash manuring and susceptibility of plants.

#### PATENTS.

Production of fertiliser mixtures. KALI-FORSCH-UNGS-ANSTALT G.M.B.H. (B.P. 366,737, 17.2.31. Ger., 21.2.30).— $K_2SO_4$  is dissolved in HNO<sub>3</sub> and the solution mixed rapidly with finely-pulverised crude phosphate, there being added at the same time so much dry finished product that, on completion of the reaction, a solid, friable decomp. product is obtained.

#### F. YEATES.

Manufacture of products for combating diseases of plants, grain, or soils. Soc. ANON. ETABL. & LABORATOIRES G. TRUFFAUT (B.P. 364,046, 16.8.30. Fr., 16.8.29).—A H<sub>2</sub>O-sol. org. dye is mixed with a substituted amide of a fatty acid (as penetrating agent), with or without another fungicide etc. Examples are : crystal-violet and stearic  $\beta$ -dimethylaminoethylamide (for seeds); diaminoazobenzene and oleic  $\beta$ -diethylaminoethylamide in EtOH or glycol (plant spray against mildew); auramine and stearic  $\beta$ -methylethylaminoethylamide with a Cu salt or an insol. arsenate; eosin and oleic  $\beta$ -diethylaminoethylamide methosulphate.

C. HOLLINS.

Leaching of phosphate rock. Stable  $Ca(H_2PO_4)_2$ solutions. Volatilising S.—See VII. By-product from ground-nut.—See XII.

# XVII.—SUGARS; STARCHES; GUMS.

Clarification of beet juice : lime economy. W. BEVTE (Deut. Zuckerind., 1931, 56, 660).—Raw juice from the measuring tanks was allowed to flow into a large collecting tank beneath, where it was mixed with a definite vol. of pre-defecated juice, which was pumped to the defecation station. There it was dry-limed, sent to the first carbonatation tanks, and subsequently treated as usual. Defecated juice to the amount of 12-17% was returned in this way, giving a mixed juice showing 0.08-0.12% CaO by titration. A good clarification resulted, it being unnecessary to "sulphur" the juices. No irregularities in carbonatation and filtration were experienced, and raw sugar of high type was constantly produced. J. P. OGILVIE. Colloids present in molasses. K. SMOLENSKI (Gaz. Cukr., 1931, 18, 535-545; Deut. Zuckerind., 1931, 56, 1106-1107).—The viscosity of beet molasses was never greater than that of a solution of pure sugar at the same Brix degree. At alkalinities above  $p_{\rm H}$  11 the viscosity increased distinctly, both with molasses and pure sugar solutions. Two groups of colloids are present: (1) irreversible, insol. in H<sub>2</sub>O or acid, of amphoteric character, and having a very intense dark-brown colour and a high N content; (2) reversible, H<sub>2</sub>O-sol., neutral, light-brown colour, low in N. Some colloids were separated having 150-200 times the colour of the original molasses. J. P. OGILVIE.

Viscosity of cane molasses. T. A. E. BARKER (Rep. Dept. Sci. and Agric., Barbadoes, 1930—31).— Applying the capillary-tube method, it was shown that the viscosity of cane molasses rapidly decreases up to 50—55°, above which the rate of decrease is slow, becoming practically const. at 65°. Assuming that a low viscosity favours a rapid crystal growth, the optimum curing temp. of a massecuite so far as viscosity is concerned is above 50°. J. P. OGILVIE.

Rapidity of [sugar] crystal growth. P. HONIG and W. F. ALEWIJN (Java Proefstat. Med.; Internat. Sugar J., 1931, 33, 595) .- Figures are given showing how considerably the rate of crystallisation depends on the temp., so that if at 65° about 10% requires 10 min. to crystallise out on already formed crystals, then under the same conditions of supersaturation and purity at 45° about 50 min. will be required for the same quantity of sugar to crystallise out. Calculations of the wt. and surface of the individual crystals making up an average strike indicate the importance of avoiding the presence of false grain, the surface of which may comprise an important part of the total. This fine material will take up a great proportion of the sugar crystallising out subsequently, most of which will be thrown through the screen of the centrifugals with the molasses. J. P. OGILVIE.

Torula inoculation process for preventing deterioration of raw sugars. ANON. (Internat. Sugar J., 1931, 33, 611).—In this process the fructose is fermented by the *Torula*, the polarisation of the product being thus increased and a certain loss in wt. incurred. It is estimated that 1 short ton of raw sugar of average composition which rises 1° V. in polarisation would lose 6·12 lb., which loss of wt., therefore, would be amply compensated by the increased value of the higher polarisation. J. P. OGILVIE.

Decomposition of sucrose and glucose in lowgrade massecuites. J. GOMERI (Philippine Agriculturist, 1931, 20, 199-216).—In a Philippine canesugar factory the loss was observed of as much as 2% of sucrose (on solids) within 30-50 hr. after dropping the massecuite. Some massecuites held in the crystallisers for 70-90 hr. showed considerably smaller losses, e.g., 0.12-0.45%. It is suggested that the temp. at which the massecuites had been boiled may account for the difference of behaviour observed. J. P. OGILVIE.

Treating [sugar-]refinery products with the calcium salts of phosphoric and sulphurous acids. K. K. LIUBITZKI (Nauk. Zapiski, 1931, 13, 149–175).— Green syrups and after-product syrups can be decolorised

to the extent of 35% by treatment with CaO and  $H_3PO_4$ , using a temp. of about 80°. Even better results are obtainable with  $H_2SO_3$  used in the following way. A current of  $SO_2$  is passed into a solution of the product warmed to 60—65° and milk-of-lime added up to 0.5-2.0% CaO on the wt. of sugar, the reaction being maintained at  $p_H 6.5$ . After filtering, the solution is brought to  $p_H 7.2$  with milk-of-lime, heated to 80°, and again filtered with the addition of about 0.25% of kieselguhr. Under these conditions the decolorisation may reach 50%. J. P. OGILVIE.

 $p_{\rm H}$  measurements.—See XI. Sugar-beet pulp for baby beef. Determination of molasses in feeds.—See XIX.

See also A., April, 338, Physical chemistry of starch. 341, System CaO-sucrose- $H_2O$ . 370, Starch. 416, New sugar in human milk.

#### PATENTS.

Saccharification of cellulose and the like under pressure with dilute acids. H. SCHOLLER and W. KARSCH (B.P. 367,916, 29.11.30).-Compact charges of sawdust in closed vessels are hydrolysed, at rising temp. between 120° and 220°, by dil. acid, limited amounts of which are forced through the material at intervals, so that sugar formed is removed from time to time, but conversion is continuous as the material remains moist with acid between successive passages of liquid. The process is preferably operated on the countercurrent principle in a battery of hydrolytic vessels, the acid liquors passing through all the vessels in turn from the most exhausted charges at the highest temp. to the least exhausted at the lowest temp. Suitable apparatus is claimed. The sugar solutions obtained may be treated with 0.2-2% of  $P_2O_5$ , 1-5% of  $SO_2$ , and 0.5-5%of N, calc. on reducing sugar, to facilitate fermentation. J. H. LANE.

Bleaching of sugars.—See VII. Yeast. EtOH from molasses.—See XVIII.

# XVIII.—FERMENTATION INDUSTRIES.

Influence of the nutrition of yeast with different types of nitrogenous food on the yield and quality of yeast obtained by the aëration process. H. CLAASSEN (Angew. Chem., 1932, 45, 80-84) .- The influence of NH4 salts, asparagine, aspartic acid, malt extracts, and oil-cake residues when added to a standard molasses-superphosphate or sugar-superphosphate medium has been examined. The yield, quality, and protein content of the yeast obtained vary considerably. NH<sub>3</sub>-N produces much smaller yields than does equally assimilable org. N, but leads to a much higher protein content; the total N assimilation is reduced if NH<sub>3</sub>-N predominates in the medium. NH<sub>3</sub>-N also produces a poor-quality yeast. Increase of aëration is least effective in increasing the yield when NH3-N is added. The results thus confirm Henneberg's recommendation, viz., that org. N should not be replaced by, but may be supplemented by, H. F. GILLBE. NH<sub>3</sub>-N.

Relation between sugar content and attenuation of worts. W. PIRATZKY (Woch. Brau., 1932, 49, 59-62).—The "apparent maltose" is determined by boiling 2 c.c. of undiluted brewery wort with 30 c.c. of mixed Fehling's solution; after cooling, the excess Cu is found by adding 2—3 g. of KI, acidifying, and titrating with 0.1N-Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. A graph is given for the conversion of the Cu nos. to maltose vals. Similar determinations are made on 10 c.c. of completely fermented wort and a const. representing the proportion of maltose fermented by the particular yeast employed is obtained. "Apparent maltose"  $\times$  yeast const. gives the amount of fermentable material. F. E. DAX.

Acid wines and their manufacture. C. LUCKOW (Pharm. Zentr., 1932, 73, 177—179).—No definite standards for the treatment and production of acid wines can be established. Each individual case must be judged separately on the basis of the analytical and tasting tests. Examples of the properties and treatment of faulty wines are given. E. H. SHARPLES.

Sloeberry wine. P. VASTERLING (Pharm. Zentr., 1932, 73, 165-168).-Sloeberries were exposed for 3 days to frost, to decrease their content of acidity, before being pulped and allowed to ferment without the addition of yeast. If the juice was seeded with yeast, the fermentation was quicker and the wine was characteristic of the particular yeast. The fermentation ended after 4 weeks and yielded a yellow-red wine with an agreeable bouquet and flavour of liquorice, which after a year in bottle gave the following vals.:  $p_{\rm H}$  4.0,  $d^{15}$  1.0575, EtOH 15.36 vol.-%, extract content 19.92, invert sugar 17.35, total acidity (as malic acid) 0.40, volatile acidity (as AcOH) 0.125, lactic acid 0.32,  $H_3PO_4 0.01$  g. per 100 c.c. Although the fresh must contained approx. 3 g. of tannic acid per 100 c.c., traces only were found in the wine. C. RANKEN.

Polarographic studies of fermentation products. VI. K. SHOJI and M. ONUKI (Bull. Inst. Phys. Chem. Res., Tokyo, 1932, 11, 277–283).—According to reduction-potential measurements "Shōyu" (Japanese sauce from soya beans) contains Ac<sub>2</sub>, furfuraldehyce, an aliphatic and an aromatic aldehyde, none of which causes the characteristic odour. R. S. CAHN.

Torula inoculation of sugar.—See XVII. Brewery yeast for bread.—See XIX.

See also A., April, 428, Allantoinase. Uricase. Zymase fermentation. Action of I on yeast. Biological gas reactions. 429, Conversion of lactic into pyruvic acid. Production of trimethylene glycol. Pectin fermentation.

#### PATENTS.

Manufacture of yeast. C. N. FREY, A. SCHULTZ, and A. P. HARRISON, ASSTS. to STANDARD BRANDS, INC. (U.S.P. 1,814,210, 14.7.31. Appl., 30.6.27).— Yeast is propagated in a solution of molasses with added  $CO(NH_2)_2$ , the splitting up of which into  $NH_3$  at a suitable rate for assimilation by the yeast is controlled by adjusting the initial  $p_H$  of the solution to  $4 \cdot 2 - 4 \cdot 5$ .

C. RANKEN.

Manufacture of yeast [and alcohol]. S. JANSEN (B.P. 366,753, 9.9.31).—Fermenting wort is agitated by being circulated at a rate of flow of  $\prec$  15 cu. m. per hr. from the bottom of the tank, through closed channels, into the same vessel at points below the

British Chemical Abstracts-B

444

surface of the liquid; or the gas at the top of the vessel is withdrawn and forced back into the liquid at a velocity of at least 25 cu. m. per hr. for each cu. m. of wort. C. RANKEN.

Manufacture of alcohol from molasses or the like. J. SCHINDELMEISER and J. E. HODCKIN (B.P. 366,992, 15.8.30).—Yeast of the character of Steinberg yeast is grown in a medium containing phosphate and nitrogenous substances to which is added later sulphite followed by a proportion of molasses. The whole is then added to and allowed to ferment a solution of molasses which has been pretreated with  $H_2SO_3$ .

C. RANKEN.

Manufacture of vitamin-containing beer. F. LUX, Assee. of H. KRÖNIG (B.P. 367,063, 28.11.30. Ger., 29.11.29).—Yeast is destroyed by some mechanical method such as by treatment in colloid mills, or mixed with quartz and pressed through closely disposed rollers. The cell sap or a centrifugate is added to the finished beer. The destruction of the yeast may take place without withdrawal of the yeast from the fermenting liquid. C. RANKEN.

Manufacture of vitamin-containing beverages, particularly beer. F. Lux (B.P. 367,909, 29.11.30. Ger., 3.12.29. Addn. to B.P. 367,063; preceding abstract) .-- Yeast which has been wholly or partly destroyed by mechanical, chemical, or physiological treatment without destruction of the fermentation enzyme is added to the wort before fermentation. The addition increases the fermentative capacity of the living yeast, which is again destroyed and the cell sap admixed with the finished beer. In a second method the yeast and bacteria in the yeasty residues from finished beer are destroyed by heating the sediment at 34-37° for 1-2 days, so that the vitamins contained in the cells are transferred to the beer residue, which is then mixed with the beer. C. RANKEN.

Measurement of proteolytic activity. H. C. GORE and C. N. FREY, Assrs. to STANDARD BRANDS, INC. (U.S.P. 1,815,709, 21.7.31. Appl., 19.6.29).—The  $[\alpha]_D$  of a standard gelatin solution is determined at 35° and 20°. Aliquot portions of the buffered solution are incubated at 45° with different amounts of the enzymic solution, cooled to below 20° for 1 hr., and the  $[\alpha]_D$  again determined at 35° and 20°. The proteolytic power follows from the formula P = W/wt, where W = g. of airdry gelatin digested, w = wt. of substance containing enzyme, and t = time in hr. C. RANKEN.

Saccharification of cellulose.—See XVII. Malted milk. Nutritious powders.—See XIX.

# XIX.-FOODS.

Reaction of flour of some varieties of hard red spring wheat to bleaching agents. R. K. LARMOUR, W. F. GEDDES, and J. G. MALLOCH (Canad. J. Res., 1932, 6, 255—264).—Seventeen varieties of flour were bleached by three methods, viz.,  $\frac{3}{4}$  oz. Betachlor per brl. (I); 1 lb. Novadel per 40 brl, (II); and  $\frac{1}{2}$  oz. Betachlor per brl. followed by 1 lb. Novadel per 40 brl. (III). The colours of gasoline extracts of the bleached and unbleached samples were compared with that of K<sub>2</sub>CrO<sub>4</sub> solution. All the flours could be reduced to approx. the

same colour, the more highly pigmented varieties being the more sensitive to bleaching agents. (III) was most efficient and (I) least. The effect of different baking formulæ on the colour and vol. of the bread is considered. D. R. DUNCAN.

Comparison of glutenin and gliadin prepared from one flour by various methods. R. K. LARMOUR and H. R. SALLANS (Canad. J. Res., 1932, 6, 38-53).-Determinations of the N distribution in glutenin and gliadin prepared from the same flour by five different methods show that differences in manipulation have little effect on the gliadin, but that the NH<sub>3</sub>-N and the basic N of the glutenin vary widely according to method of prep., the vals. tending to be inversely proportional to each other. Glutenin isolated by Blish and Sandstedt's method (B., 1930, 637), using 0.007N-AcOH, contained the most NH3-N, and lost 4.8% of its total N as  $NH_3$  when kept in solution in 0.025N-NaOH for a week. The basic fraction was unchanged. The use of dil. NaOH or of 0.07N-AcOH in preparing glutenin W. J. BOYD. should be avoided.

Determination of cellulose in flour and its by-products. L. BELLUCCI (Annali Chim. Appl., 1932, 22, 25—31).—König's method (A., 1903, ii, 764) adopted officially in the Italian Government Regulations gives results which agree closely with those obtained by Kürschner's method (B., 1930, 608) when applied to these products. O. F. LUBATTI.

Milling and baking quality of frosted wheat of the 1928 crop. W. F. GEDDES, J. G. MALLOCH, and R. K. LARMOUR (Canad. J. Res., 1932, 6, 119–155).— In grades I, II, and III the % of sound kernels is 75-85%, whilst the % of kernels showing damage by heavy frost is negligible. In grade VI the % of sound kernels is 20, of those showing "heavy damage" 50, and of those frosted 25%. Green kernels have less influence than damaged ones on flour yield. The protein content of a flour is a better indication of its baking quality than the % of sound kernels.

P. G. MARSHALL.

Effect of storage at various moisture contents on baking quality of Marquis wheat. R. K. LAR-MOUR (Canad. J. Res., 1932, 6, 156-161).—Some deterioration of baking quality occurs in wheat containing >15% of moisture when stored at 21° for 4 months. P. G. MARSHALL.

Utility of cooked potato in baking bread and its relation to crude protein and baking strength. R. H. HARRIS (Canad. J. Res., 1932, 6, 54-67) .--Addition of dried, mashed, cooked potato containing all the original material, or of the liquor drained from boiled sliced potato, to the basic baking formula gave progressive improvement in colour and loaf vol. with increasing amounts of potato or liquor added. Loaves made with potato remained fresh longer than the controls. Doughs treated with varying amounts of boiled mashed potato showed corresponding increase in gas production. The gas lost from the doughs increased also, but to a less degree. Loaves baked with >10 g. of potato per 100 g. of flour were inferior in grain and texture, and colour was also poor at higher concns. The higher-protein flours gave larger loaves throughout the test. Raw grated potato or potato ash added to the flour had no improving effect. W. J. BOYD.

Calcium lactate in baking powders. P. HERR-MANN and L. VOGELER (Deut. Nährungsmittel-Rundsch., 1931, 162—164; Chem. Zentr., 1931, ii, 2801).—From NaHCO<sub>3</sub> at 100° 17% of the theoretical quantity of CO<sub>2</sub> is evolved; with Ca lactate 100% is liberated in 20 min. at 100°, whilst with tartaric acid  $84 \cdot 2\%$  is rapidly liberated at 40° and 100% in 10 min. at 80°. An apparatus for the determination is described.

A. A. ELDRIDGE.

Vitamin content of bread and its improvement by brewery yeast. Y. HASHITANI and T. SAKO (Woch. Brau., 1932, 49, 65—68).—Nutrition experiments with pigeons confirm the results of others that white bread is deficient in vitamin-B. The deficiency is removed by the addition of 0.3—0.5% of carefully prepared dried brewery yeast. During baking the temp, within the loaf hardly reached 100°, and only exceeded 90° for about 10 min. F. E. DAY.

Storage of rice. V. Influence of a desiccating material on the preservation of the germinating power of hulled rice having different moisture contents and stored at different temperatures. M. KONDO and T. OKAMURA (Ber. Ohara Inst. landw. Forsch., 1931, 5, 221—242).—Storage of rice in the presence of CaCl<sub>2</sub> minimises the loss of germinative capacity. The effects of the initial moisture content and temp. of storage of the grain are recorded. Although storage conditions tending to reduce germinating power are also those causing the destruction of catalase, no sp. relationship between these two factors was observed. A. G. POLLARD.

**Poor-quality rice grains.** V. Decolorisation of green-coloured hulled rice, "Aomai." M. KONDO and T. OKAMURA (Ber. Ohara Inst. landw. Forsch., 1931, 5, 57—66).—The action of light of various colours on the decomp. of chlorophyll in the grain was parallelled by its effect in reducing the activity of the catalase and peroxidase present. In neither case was the light action affected by prior drying of the grain or exposure to  $O_{a}$ . A. G. POLLARD.

Butter fat in cheese. C. E. HYNDS (N.Y. State Dept. Agric. Ann. Rep., 1930, 91).—A modification of the Gerber method gives results in close accord with those given by the Schmidt-Bondzynski method.

CHEMICAL ABSTRACTS. Effect of cottonseed meal and other feeds on the storage quality of eggs. R. M. SHERWOOD (Texas Agric. Exp. Sta. Bull., 1931, No. 429, 19 pp.).—The use of cottonseed oil or meal in chicken foods decreases the storage capacity of eggs and induces abnormal coloration of both yolks and whites. Such eggs have less fat and more  $H_2O$  in the yolk. The substance causing these abnormalities is associated with the oil, but is not contained in highly refined grades. Simultaneous feeding of lettuce did not reduce injurious effects.

A. G. POLLARD. Origin of diacetyl in butter. G. TESTONI and W. CIUSA (Annali Chim. Appl., 1932, 22, 44-48).—Ac<sub>2</sub> is not the const. aromatic principle of butter; it is not found in fresh milk, nor in milk which has soured spontaneously. Its presence is due to the action of rennin preps. when these substances are used in the process of manufacture, the intermediate product being acetylmethylcarbinol. O. F. LUBATTI.

Red pigment production in apples by means of artificial light sources. J. M. ARTHUR (Contr. Boyce Thompson Inst., 1932, 4, 1—18).—The ultra-violet, violet, and blue regions of the solar spectrum are the most active in producing colour in picked apples. Infra-red rays from artificial sources produced severe burning. Best results in the artificial colouring of apples (picked green) were obtained with Hg-vapour lamps, the fruit being well coloured after 40 hrs.' exposure. Colour production is a function of the living cells of the epidermis. Failure to produce colouring artificially in fruit after long storage is attributed to the death of these cells.

A. G. POLLARD.

Colorimetric determination of saccharin in beverages. L. J. CROSS and J. L. PERLMAN (N.Y. State Dept. Agric. Ann. Rep., 1930, 89–90).—The method depends on the formation of phenol-red from the Et<sub>2</sub>O-extracted residue from acidified samples of the beverage. CHEMICAL ABSTRACTS.

Rapid freezing. I. Critical rate of cooling. II. Temperature of storage. T. MORAN (J.S.C.I., 1932, 51, 16—20 T, 20—23 T).—I. An attempt has been made to fix the limits of rapid cooling in terms of the cooling curve and the thickness of material. The distinction between rapid and slow freezing in a 5% gelatin gel is sharply defined; it is not so sharp in mammalian muscle, but the physical properties of the frozen muscle suggest a limiting state when the time to cool and freeze from 5° to  $-5^{\circ}$  is in the range 48—32 min. depending on the temp. of the freezing medium. Figures are given for the corresponding thicknesses of tissue for different conditions of cooling.

II. High temp. of storage induce changes in rapidly frozen muscle. This has been shown by measurements of the drip or free fluid on thawing, the composition of the drip, and the size of the ice crystal in the muscle. The effect of temp. is marked after 2 days' storage.

Sugar-beet pulp for baby beef. H. W. GARDNER and J. HUNTER-SMITH (J. Min. Agric., 1931,38,993—997). —Sugar-beet pulp, beet-molasses pulp, and crushed oats are of equal feeding val. The molasses pulp is more relished. W. G. Eggleton.

Relative values of oats, beet-molasses pulp, and dried sugar-beet pulp for the fattening of store cattle. H. E. WOODMAN, W. S. MANSFIELD, and F. H. GARNER (J. Min. Agric., 1931, 38, 985-993).—Beetmolasses pulp and dried sugar-beet pulp are equal in feeding val. to crushed oats when fed, wt. for wt., to mature fattening cattle. W. G. EGGLETON.

Determination of salt and molasses in mixed feeds. G. S. FRAPS (Texas Agric. Exp. Sta. Bull., 1931, No. 425, 12 pp.).—To the sample (5.85 g.) is added saturated picric acid solution to make a total vol. of 200 c.c. After occasional shaking during 1 hr., 50 c.c. of the clear liquid are treated with slight excess of CaCO<sub>3</sub>. A brownish colour, sometimes produced by molasses feeds, may be removed by means of C black. The Cl' is determined by titration with AgNO<sub>3</sub>, using K<sub>2</sub>CrO<sub>4</sub>. Sugar is extracted from the sample

 $(5\cdot333 \text{ g.})$  with 100 c.c. of  $\text{H}_2\text{O}$ . The filtered extract is cleared with basic Pb acetate and diluted to 200 c.c. To 75 c.c. are added 5 c.c. of 10%  $\text{Na}_2\text{SO}_4$  solution and 5 c.c. of 10%  $\text{CuSO}_4$  solution, and the mixture is diluted to 200 c.c. and filtered. Sugar is determined after inversion by the A.O.A.C. method of Munson and Walker. A. G. POLLARD.

Examination of animal feeding-stuffs. Calculation of nutritive units. P. BRUÈRE (Ann. Falsif., 1932, 25, 6—15).—The scheme outlined for the analysis and evaluation of animal feeding-stuffs is that adopted in preparing reports presented to the Commission d'Alimentation rationelle et économique du cheval de guerre. The method of determining acidity of feeding-stuffs, prescribed in the Bull. Off. Ministère de la Guerre, Partie Permanente, No. 51, 23.12.29, p. 4863, is also given.

W. J. BOYD.

Relation of  $p_{\rm H}$  value to toxicity of preservatives to micro-organisms. W. V. CRUESS and J. H. IRISH (J. Bact., 1932, 23, 162—166; cf. A., 1929, 1491).— The min. concn. of AcOH, CH<sub>2</sub>O, H<sub>2</sub>SO<sub>3</sub>, BzOH, and salicylic acid necessary to prevent the growth of typical micro-organisms on fruit juices was much greater at  $p_{\rm H} 2 \cdot 5$ —3.5 than at neutrality. The retarding effect of NaCl and CH<sub>2</sub>O on fermentation was not appreciably affected by [H]. A. G. POLLARD.

Determining fat in foods. Hardening of [codliver] oil.—See XII. Coconut industry.—See XVI. Packing-house effluents.—See XXIII.

See also A., April, 338, Starch and bread-making. 433-4, Vitamins (various).

# PATENTS.

Treatment of cereal products in preparation for baking. M. VUK and S. GOMORY (B.P. 367,799, 20.11.30. Hung., 21.11.29).—An improver for addition to flour for bread-making is prepared by heat-treating wheat flour in steam for 20 min., adding  $H_2O$ , and again heating until the outline of the starch grains is no longer visible; the mass is then dried and powdered.

# E. B. HUGHES.

Manufacture of food products and medicinal preparations from corn-milling by-products. L. BERNARDINI (B.P. 366,516, 18.10.30. It., 19.10.29).— The oily substances containing fat-sol. vitamins are extracted by a solvent (EtOH) from millings containing cereal germs. After removing the solvent, the oily substance is added to various cereal products (macaroni etc.). The residue is then milled and the cortical elements are sifted from the flour, which contains other vitamins and nitrogenous substances. This flour may also be added to cereal foods. E. B. HUGHES.

Treatment of soya beans and similar leguminous seeds. F. Gössel (B.P. 367,082, 11.12.30. Ger., 10.1.30).—Soya beans (etc.) may be immersed in a hot oil such as parafin for 5 min. at 100°. The beans will then husk easily and the products be free from objectionable flavour. Other oils or fats may be used, with or without the addition of some hygroscopic salt such as CaCl<sub>2</sub>. E. B. HUGHES.

Preventing the oxidation of soya beans and bran obtained therefrom. L. BERCZELLER (B.P.

367,865, 25.8.30. Austr., 26.8.29).—The development of rancidity in soya beans is prevented by a short steamdistillation treatment. E. B. HUGHES.

Manufacture of nutritious powders. R. NOMURA (B.P. 367,340, 12.9.30).—A food for infants is manufactured from (a) two or more cereal flours, (b) Japanese yam powder and meat juice or fish. (a) and (b) are allowed to ferment and then sterilised with ultra-violet rays; they are then mixed with fresh milk and fermented, sterilised by ultra-violet rays, and then powdered and packed into hermetically sealed containers.

E. B. HUGHES.

Manufacture of solid milk and cream preparations. N.V. NEDERLANDSCHE GRUYÈRE-BLOKMELK FABRIEK (B.P. 366,421, 25.7.31. Ger., 26.7.30).—Solid preps. are made by adding sugar to milk or cream, evaporating the bulk of the  $H_2O$  at 50—65° in vac. without agitation, further evaporating in vac. at below 50° while stirring (to leave 7—15%  $H_2O$ ), and moulding into shapes. E. B. HUGHES.

Manufacture of liquid malted milk. W. O. FROHRING, ASST. to TELLING BELLE VERNON CO. (U.S.P. 1,810,518, 16.6.31. Appl., 16.9.25),—Liquid malted milk in non-perishable form is prepared by boiling a mixture of starch and  $H_2O$  to swell the starch, adding an enzyme, and heating at 65° until malted; the liquid is conc., milk is added, and the mixture homogenised and run into containers, which are hermetically sealed. The product is sterilised at 115.5°. E. B. HUGHES.

Treating [detecting abrasions in] fruits. H. T. O'NEILL and A. J. HARRIMAN (U.S.P. 1,813,357—8, 7.7.31. Appl., [A] 13.5.30, [B] 27.5.30).—Fruit free from abrasion is not subject to attack by mould. Abrasions can be detected by dusting powders or by immersion in a solution of dye which colours damaged parts. It is preferred in (A) to immerse the fruit in a solution of a Fe<sup>...</sup> salt, which shows up abrasions by formation of ferric tannate; or in (B) to add a mineral salt which is absorbed in the abrasion and to detect it by further treatment with a lake-forming dye or  $H_2S$  etc.

E. B. HUGHES.

Treating fresh fruit for retarding decay from blue and green mould. H. B. SLATER, ASST. to FOOD MACHINERY CORP. (U.S.P. 1,813,127, 7.7.31. Appl., 28.7.25).—Fresh fruit for the market is sterilised by HOCl, a mixture of solutions of NaOCl and NaHCO<sub>3</sub> being used. E. B. HUGHES.

Removing the bitter taste from orange peels. H. TEUSCH (U.S.P. 1,813,366, 7.7.31. Appl., 3.10.27. Ger., 11.10.26).—The peel is soaked in  $H_2O$ , fruit juices, or  $H_2O$  previously used for treating peel. The process is hastened if fermentation of the peel is allowed to occur. E. B. HUGHES.

Production of citrus fruit concentrates. TROPICAL VITAMIN Co., INC. (B.P. 367,105, 1.1.31. U.S., 28.7.30). —Whole fresh citrus fruits are crushed and sieved to give juice and pulp and eliminate seeds and squeezed rind. The juice is centrifuged, giving peel oil and a resinous portion; the pulp is homogenised, separating a palatable fruit "concentrate" to which may be added syrup, citric acid, peel oil, and preservative as required. This "concentrate" has the natural properties of the fresh fruit and does not separate into layers on keeping. E. B. HUGHES.

Manufacture of concentrated fruit pulp, jams, and jellies. E. MONTI (B.P. 366,010, 20.10.30. Fr., 25.10.29).—Fruits to which sugar and org. acids are added in known manner are subjected to the ripening action of biochemical catalysing agents (present or added) on perforated trays at  $0-4^{\circ}$ . Syrups and fruit pulps so obtained can be further conc. to jams or jellies by evaporation at  $40^{\circ}$  in a current of dry air heated to  $50-60^{\circ}$ . Apparatus is claimed. E. B. HUGHES.

Preparation of concentrated fruit juices in the form of jelly. P. C. LEMALE (B.P. 367,612, 1.4.31).— Fruit juices are conc. in vac. till the % of sugar is such as to prevent fermentation, and to the concentrate of suitable acidity gelose or pectin is added to form a jelly without further heating. This method is claimed to preserve the proteins and vitamins usually lost during juice preservation. E. B. HUGHES.

Preservation of fish. P. C. LAVENDER and A. E. SHERMAN (B.P. 365,530, 15.9.30).—Filtered or aërated sea-water, cooled to below 0°, is caused to percolate through the mass of fish placed in the bins or hold of trawler. H. ROYAL-DAWSON.

Preparing and preserving the beverage, tea. R. T. METZGER and P. D. POTTER, ASSTS. to SPRAGUE, WARNER & Co. (U.S.P. 1,813,120, 7.7.31. Appl., 11.1.30).—Tea infusions may be preserved in sealed containers if pretreated under pressure at 115° for a period sufficient to bring about rapid coagulation and pptn. of the substances causing sediment, and then filtered or decanted. E. B. HUGHES.

Production of caffeine-free coffee and the ine-free tea. T. GRETHE (B.P. 366,305, 23.2.31. Ger., 26.9.30).—Coffee beans or tea leaves are subjected to cold or hot extraction with EtOAc, or  $Pr^{\beta}OAc$  in the presence of  $H_2O$ , without opening up the beans or leaves. H. ROYAL-DAWSON.

Making a mineral food composition. P. A. KOBER, Assr. to G. D. SEARLE & Co. (U.S.P. 1,813,936, 14.7.31. Appl., 19.5.27).—Mineral foods suitable for oral or intravenous administration are made from mixtures of sol. salts of alkaline earths (Ca and Mg), sol. phosphates, and a sol. alkali lactate; the  $p_{\rm H}$  of the mixture in aq. solution is adjusted to be  $\geq 8$ . No ppt. will form in solution even on keeping. E. B. HUGHES.

Production of frozen [liquid] comestibles [e.g., ice-cream]. W. T. COMER (B.P. 368,976, 4.3.31. U.S., 31.5.30).

Cooling and screening granular substances. Mixing of dough.—See I.  $Ca(H_2PO_4)_2$  [for bakingpowders etc.].—See VII. Edible fats.—See XII. Vitamin-containing beverages.—See XVIII.

# XX.-MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Borntraeger colour reaction and therapeutic activity of cascara sagrada. S. W. MORRISON (J. Amer. Pharm. Assoc., 1931, 20, 1276–1281).—The intensity of the red colour produced in the Borntraeger reaction (addition of aq. NH<sub>3</sub> to the Et<sub>2</sub>O-sol. portion of cascara preps.) does not always bear a direct relationship to the therapeutic activity and the test is not reliable. The bitter and colour-producing principles of cascara extracts can be salted out with  $Na_2SO_4$  without greatly affecting the activity. Preliminary extraction of cascara bark with org. solvents before preparing the aq. extract gives a more palatable, but less active, fluid extract. E. H. SHARPLES.

Spectrographic researches on ergot. I. Its active constituents. F. SCHLEMMER. II. Spectral analysis in the determination of ergot constituents and the evaluation of ergot drugs, fluid extract, and preparations. F. SCHLEMMER and H. SCHMITT (Arch. Pharm., 1932, 270, 15—29, 29—44).—I. The similarity between the ultra-violet absorption spectra of pure ergotamine and ergotoxine suggests a similar structure. The similar behaviour of the spectra of ergotamine and tyramine subjected to varying conditions, and the fact that the aq. residue after extraction of the alkaloids from an old prep. of ergotamine with  $Et_2O$  shows a spectrum like that of autoclaved tyramine, indicate that the two compounds produce the same substance on decomp.

II. The ultra-violet absorption spectrum of a solution containing < 1% of histamine and < 0.0066% of ergotamine is indistinguishable from that of pure ergotamine, showing the impracticability of the method for histamine determination. Mixtures of tyramine and ergotamine show distinct spectra and allow of accurate ergotamine determinations. The ergotamine concn. in ergot drugs, and in preps. having the effects of ergot, is determined by spectral analysis and compared with the advertised vals. The superiority of the method over that given in the D.A.B. is proved. H. DAVSON.

Relationship of potency and hydrogen-ion concentration in tincture of digitalis. J. C. KRANTZ, JUN., and J. C. MUNCH (J. Amer. Pharm. Assoc., 1932, 21, 16—20).—The [H'] of tinctures prepared with abs. EtOH is greater than those made with 80% EtOH; also the potency is greatly diminished and the ash content much smaller. The significance of these three facts is discussed. E. H. SHARPLES.

Extent of emulsification of alkaloid-containing preparations with immiscible solvents at different degrees of  $p_{\rm H}$ . W. W. F. ENZ and C. B. JORDAN (J. Amer. Pharm. Assoc., 1932, 21, 34—36).—Attempts to determine whether the extent of emulsification occurring during the prep. of tinctures and extracts could be controlled by the  $p_{\rm H}$  val. indicated no general uniformity. Fluid extract of belladonna showed least emulsification at the neutral point, tincture of stramonium showed least in acid solution, but fluid extracts of cinchona and hydrastis and tincture of nux vomica showed no uniformity. Of the solvents, CHCl<sub>3</sub>, Et<sub>2</sub>O, C<sub>5</sub>H<sub>11</sub>·OH, C<sub>6</sub>H<sub>6</sub>, and light petroleum, the last showed least emulsification in acid solution, with the exception of fluid extract of cinchona, but the remainder gave varying results.

E. H. SHARPLES.

Isolation of nicotine from waste tobacco dust. T. WADA and M. SUMI (Bull. Inst. Phys. Chem. Res., Tokyo, 1932, 11, 124—134).—Distillation at 400° with superheated steam of waste tobacco dust soaked in dil. alkali gives the best yield (85%) of nicotine. Use of a solvent is wasteful and gives a smaller yield.

F. R. SHAW.

Microchemistry of medicinal derivatives of cinchonic acid. G. DENIGÈS (Mikrochem., 1932, 10, 430-436).-COMe, is the most suitable solvent for atophan; on evaporation of a droplet of solution a characteristic grouping of roughly spherical particles appears at the periphery, with elongated crystals in the central zone. Solutions in aq. NH3 or NaOH yield long prisms which radiate from a common centre, and on addition of acid characteristic crystals of free atophan separate. NaOBr and acid yield an amorphous orange Br-derivative; the analogous I- and Cl-derivatives are maroon and yellowish-white, respectively. 1-2 mg. of atophan may be recognised by these reactions, and confirmed by the sublimate obtained by heating above the decomp. temp. Fantan is insol. in most of the ordinary solvents. Characteristic prismatic crystals are obtained by evaporation of solutions in COMe, or CHCl, and AcOH. The Br- and I-derivatives resemble those of atophan. H. F. GILLBE.

Detection of hemlock [coniine] in anise. P. N. SCHÜRHOFF (Arch. Pharm., 1932, 270, 145-148).-The periodide method is more sensitive than the chloranil method for microdetection of coniine in anise, whilst sublimation of the hydrochloride is inapplicable because anise contains NH<sub>4</sub> salts. R. S. CAHN.

Volumetric method of determining quinine. D. JATRIDÈS and G. THOMIS (J. Pharm. Chim., 1932, [viii], 15, 230-242) .- A variety of methods for the determination of quinine in pharmaceutical preps. is studied. The method finally recommended is to extract the alkaloid from an alkaline solution with CHCl<sub>3</sub> and titrate an aliquot part of the extract, after dilution with 95% EtOH, with 0.2N-HCl, using lacmoid as an indicator. The end-point is determined by colorimetric comparison with a standard adjusted to  $p_{\rm H}$  4.9-5.0. H. A. PIGGOTT.

Simple tests for some modern local anæsthetics. K. W. MERZ (Arch. Pharm., 1932, 270, 97-100) .--Tests are described for differentiation between percaine, pantocaine, larocaine, novocaine, tutocaine, and psicaine. R. S. CAHN.

Modern cocaine substitutes. K. W. MERZ (Arch. Pharm., 1932, 270, 125-139).-A lecture. R. S. CAHN.

Determination of caffeine, amidopyrin, and phenacetin in presence of each other. J. von Mikó (Pharm. Zentr., 1932, 73, 179-181).—The phenacetin is dissolved in hot H<sub>2</sub>O and the amidopyrin separated from the residue by extraction with Et<sub>2</sub>O. E. H. SHARPLES.

Volumetric and gravimetric determination of theobromine sodium salicylate. J. von Mikó (Arch. Pharm., 1932, 270, 163-167).-Standard methods are modified. R. S. CAHN.

Reactions of several salvarsan preparations. L. EKKERT (Pharm. Zentr., 1932, 73, 197-200).-Details are given of the colour reactions of myo-, neo-, and sulphoxyl-salvarsan, neoiacol (Na diaminodihydroxyarsenobenzenemethylenesulphoxylate), and revival (Na diaminodihydroxyarsenobenzenemethylsulphoxylate) with Gaebel's (A., 1911, ii, 448) and Abelin's (A., 1911, ii, 948) reactions, and of their behaviour with the following oxidising agents : FeCl<sub>3</sub>, H<sub>2</sub>O<sub>2</sub>, chloroamine-HCl, KClO<sub>3</sub>-HCl, K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>-H<sub>2</sub>SO<sub>4</sub>, and K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> alone. J. W. BAKER.

Physical and chemical properties of the volatile oils from plant products. J. F. CLEVENGER (J. Amer. Pharm. Assoc., 1932, 21, 30-34).-The limiting and usual characteristics obtained in the examination of essential oils distilled from samples representing large nos. of shipments of each of 12 oil-bearing plants are given. E. H. SHARPLES.

Composition of machine-extracted lemon oil. A. H. BENNETT and D. J. TARBERT (Annali Chim. Appl., 1932, 22, 32-44).-The "alcohol index" (ratio alcohol: citral) is 1.6-2.6 for ordinary terpeneless concentrates and 2.9-4.4 for machine-prepared concentrates. A rapid method for determining this index, based on the solubility of salicylic acid in the constituents of these oils, is described. Results obtained by the application of Grignard's reagent for the determination of alcohols according to Kohler, Stone, and Fuson (A., 1928, 160) is given. O. F. LUBATTI.

Use of the analytical quartz lamp for the examination of essential oils. E. EKMAN and A. SAMY-SCHLAYEVA (Riechstoffind., 1931, 6, 140-144; Chem. Zentr., 1931, ii, 2671).-The tabulated results can be used to indicate constituents, manner of extraction, age, and origin. A. A. ELDRIDGE.

2 : 4-Dinitrophenol.-See III. Lansium domesticum.-See XII.

See also A., April, 356, Determining Al [in pharmaceutical preps.]. 367, Avertin. 370, Aloin and 380, Quaternary NH4 halides of peristaltin. ephedrine. Resorcyl alcohols. 396, Condensation of p-tolyl Me ether with benzoquinone. 403. Quinoline derivatives. 404, isoPropylallylbarbituric acids. Alkaloids of, 405, Anabasis aphyllia, 406, Stemona tuberosa and Holarrhena antidysenterica. Determination of quinine. Separation of pilocarpine from quinine. neoStrychnine and derivatives. 407, Strychnos alkaloids. 408, Alkaloids similar to berberine. Arylthioarsenites. p-Xylylarsinic acid. 411, Determination of caffeine. 424, Local anæsthetics. Micro-detection of soporifics. Ethylharmol. 433, New cryst. sexual 433-4, Vitamins (various). hormone. 437, Isolation of carotene.

#### PATENTS.

Preparation of organic derivatives of bromine [brominated unsaturated fatty acids]. S. HERMANN, and PHARMACEUTISCHE SERKE "NORGINE" A.-G. (B.P. 364,394, 16.2.31. Austr., 10.5.30) .- A natural fat or the corresponding unsaturated fatty acid is converted into its Br additive product without substitution, and HBr is then removed (and ester groups are hydrolysed) by KOH-EtOH without hydrolysis of Br to OH. Suitable starting materials are olive, ground-nut, linseed, poppy, rape, and cod-liver oils. C. HOLLINS.

Manufacture of anhydrous alkali-metal salts of 5:5-dialkylbarbituric acids. E. LILLY & Co., Assees. of H. A. SHONLE (B.P. 364,041, 21.6.30. U.S., 22.6.29) .-A purified 5-alkyl- (C3-C3) -5-ethyl- or -allyl-barbituric acid in alcoholic suspension or solution is treated with a carbonate-free alkali (NaOH or NaOEt), and the solution evaporated in vac. to give a stable anhyd. salt. The prep. of Na 5-ethyl-5-isoamylbarbiturate is described. C. HOLLINS.

Preparing liquids containing active principles or hormones from parathyroid glands. Allen & HANBURYS, LTD., N. EVERS, C. J. EASTLAND, and J. H. THOMPSON (B.P. 366,940, 10.11.30).—The glands are extracted with an acid-containing liquid, such as aq.alcoholic  $H_2SO_4$ , of suitable  $p_{\rm H}$ , and the extract is neutralised or made slightly alkaline, set aside to complete pptn., filtered, and the filtrate conc., adjusted to  $p_{\rm H}$  4.6-5.0, allowed to clarify with or without addition of EtOH, and the liquid separated. The glands may first be extracted with a fat-solvent and the extract treated with the acid liquid, or a mixture of the fatsolvent and the acid liquid may be used for the first extraction. The residual glands after the main extraction are treated with acid again or with fat-solvent and/or finally treated with dil. alkali. The pptd. insol. materials can be redissolved in dil. alkali and the extracts treated as above. E. H. SHARPLES.

Manufacture of a therapeutically active product from tumorous tissue. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 366,503, 29.10.30).—Fresh tumorous tissue is pressed and/or extracted with  $H_2O$ or NaCl solution at or below 0° and the extract is purified or evaporated. E. H. SHARPLES.

Manufacture of a preparation or medium for treating tuberculous or other conditions in the thorax. R. A. and J. HUNTER (B.P. 366,623, 18.11.30). —Gelatin is dissolved in physiological saline, the solution made slightly alkaline with NaOH, and, after autoclaving and filtering, a dil. solution of acriflavine or other flavine antiseptic in normal saline is added. The sterile solution is suitable for injection.

E. H. SHARPLES. Recovery of essential oils from lemons and other fruits of trees of the genus *Citrus*. A. H. BENNETT (U.S.P. 1,814,888, 14.7.31. Appl., 9.8.30. It., 2.6.30).—The fruits are rasped or grated and during this process are sprayed with an alkaline solution (*e.g.*, NaHCO<sub>3</sub>), which may contain a sol. neutral salt. The liberated oils are collected from the effluent liquid by centrifuging. E. H. SHARPLES.

Packaging of  $Et_2O$ .—See III. Perfumed soap.— See XII. Caffeine-free coffee etc. Mineral food. Medicinal preps.—See XIX.

# XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Latent images on bare glass. A. REVCHLER (Bull. Soc. chim. Belg., 1931, 40, 642-646).—Good images are obtained on developing plates of glass which have been wetted with 0.1N-Na halide and excess AgNO<sub>3</sub> and exposed for 20 sec. or longer. The latent image consists of a layer, not exceeding 10 Å. in thickness, of metallic Ag directly attached to the glass. C. W. DAVIES.

See also A., April, 332, Adsorption of org. materials by Ag halides. 349, Optical sensitisation.

#### PATENTS.

Light-sensitive emulsions for photographic purposes for metal films. R. KUPFER (B.P. 368,082, 16.3.31. Ger., 15.3.30).—Good adherence of an emulsion to a metal-band film is obtained by adding 2.5%of Na<sub>2</sub>SiO<sub>3</sub> solution (d 1.23) to the warm emulsion before coating. Cr acid salts or alums may be added (preferably prior to the silicate addition) to prevent blooming. J. LEWROWITSCH.

Cellulose ester films for photographic purposes. G. S. WHITFIELD, and ILFORD, LTD. (B.P. 367,028, 14.11.30).—The film base is coated with a layer of a colloid impervious to org. solvents or dyes, preferably a hardened colloid, *e.g.*, "formalised" gelatin or dichromated albumin. A thin layer of collodion is then coated on top. This layer may be dyed (*e.g.*, for filters) or have dyes printed on it (*e.g.*, for colour screens). The intermediate layer prevents the film base from being stained by the dyes. (Cf. B.P. 366,517 and 366,380; B., 1932, 369.) J. LEWKOWITSCH.

Photographic film free from halation. I. G. FARBENIND. A.-G. (B.P. 366,517, 24.10.30. Ger., 5.11.29). —The film base is protected from undesirable coloration by the antihalation dye layer by the presence of an intermediate thin layer of a substance sol. or swelling in H<sub>2</sub>O, *e.g.*, gum arabic, glucose, cherry gum, or a cellulose ether. A dye which does not stain the film may be incorporated in this layer (cf. B.P. 366,380; B., 1932, 369). J. LEWKOWITSCH.

Production of photographic images. H. D. MURRAY and D. A. SPENCER (B.P. 363,616, 20.6.30).-A metallic (Ag) image is treated with a diazo solution, after bleaching if necessary, e.g., with 5% CuCl2, washed, and developed with a coupling component, preferably in presence of fixing salt  $(Na_2S_2O_3, NaCN, etc.)$ . The diazo solution may be stabilised with org. acid, e.g., citric acid, and the acidity lowered by addition of K citrate etc. The washing is preferably performed with 2% aq. KI. Coupling is accelerated by addition of 10% of Na (or K) citrate to the coupling solution, which may also contain Na<sub>2</sub>SO<sub>4</sub> etc. to insolubilise the dye. Two or more coloured images so obtained may be superimposed on a single support to produce pictures in several colours. Prints taken through three suitable filters are, e.g., bleached with 5% CuCl2, washed, and immersed for 7-10 min, in a bath containing K citrate, citric acid, KI, and diazotised p-aminodiphenylamine sulphate. After washing in H2O and in 2% aq. KI, the prints are developed with Na2B4O7, Na2S2O3, KOH, and K citrate containing J-acid for red, H-acid for blue, or m-cresol for yellow, and are washed in 5% K Al alum until free of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. Two images, at the front and back, respectively, of a film, may be treated with the same diazo solution and developed successively with different coupling components. C. HOLLINS.

Photographic reversal process. A. R. DE TARTAS (U.S.P. 1,814,788, 14.7.31. Appl., 9.4.29).—The exposed film is developed, partly fixed in an acid fixing bath, and washed. The Ag image is then bleached, and the film washed and cleared in a NaHSO<sub>3</sub>-NaHCO<sub>3</sub> bath. The remaining emulsion is redeveloped, fixed, and washed. Greater latitude in exposure and elimination of any subsequent exposure to light are thereby obtained. J. LEWKOWITSCH.

Manufacture of colloid reliefs. I. G. FARBENIND. A.-G. (B.P. 366,552, 1.10.30. Ger., 6.12.29).—The developed Ag in a gelatin emulsion is bleached with a solution of a heavy-metal (e.g., Cu, Hg) salt and KBr. The sol. salts are removed by washing. The residual metallic complex is dissolved out by a solution of a suitable alkali or acid, the colloid in these (exposed) areas being also removed, whereby a colloid relief is obtained. A pure gelatin relief is obtainable by applying 7% aq. ZnSO<sub>4</sub> where required, and dissolving out with KOH. J. LEWKOWITSCH.

[Photographic] production of coloured designs for textiles. A. HILGER, LTD., F. TWYMAN, and C. F. SMITH (B.P. 366,578, 6.11.30. Addn. to B.P. 227,072 and 314,359).

[Light source for] photographic processes. ELEKTR. GLÜHLAMPENFABR. "WATT" A.-G. (B.P. 367,474, 26.11.30. Austr., 19.2.30).

Planographic printing bases.—See V. Electrodeposition of Ag.—See X.

# XXII.—EXPLOSIVES; MATCHES.

2:4-Dinitrophenol.—See III.

See also A., April, 340, Explosion of picrates. 376, Mol. compounds of centralite.

#### PATENTS.

**Production of explosives.** E. VON HERZ (B.P. 367,713, 28.7.31. Ger., 28.7.30).—*Trinitrophenylnitro*- $\beta$ -hydroxyethylamine nitrate (m.p. 126°), either alone or mixed with other explosives and O carriers, is used as the secondary charge in percussion caps and detonators. It may be produced by the nitration of  $\beta$ -hydroxyethylaniline or of  $\beta$ -m-di- or s-tri-nitro-anilinoethyl alcohol. Its stability equals that of tetryl, and it is less sensitive to shock and friction than pentaerythritol tetranitrate. W. J. WRIGHT.

Flashless propellant powder. E. I. DU PONT DE NEMOURS & Co. (B.P. 366,786, 27.3.31. U.S., 16.5.30). About 0.5—2.0% of K<sub>2</sub>SO<sub>4</sub> is incorporated with the powder. Its use does not debar the H<sub>2</sub>O treatment for removal of solvents. W. J. WRIGHT.

Shot gun cartridge wads. IMPERIAL CHEM. INDUSTRIES, LTD., and H. W. BROWNSDON (B.P. 368,831, 11.12.30).

Manufacture of heat-evolving cartridges. N. L. MELLGREN (B.P. 367,471, 25.11.30).

Solubilised nitrocellulose.—See V.

# XXIII.—SANITATION; WATER PURIFICATION.

Action of metallic copper on the bacteria in potable water. PHOD and CODVELLE (Compt. rend., 1932, 194, 497—500).—Following the immersion of a Cu sheet (1.0 sq. cm. total surface) in 10 c.c. of H<sub>2</sub>O, the latter is considered to have acquired standard bactericidal activity when it effects destruction of 0.2 c.c. of a *B. coli* emulsion (10 million per c.c.). Light has no material effect on activation, which occurs in 4 days at 18°, whilst at 56° only 60 hr. are required. O<sub>2</sub> is essential to the process; boiled H<sub>2</sub>O is not activated in 32 days. The bactericidal power is retained for  $> 1\frac{1}{2}$  years at room temp. and is not reduced by boiling or freezing, or addition of salts (NaCl etc.). All bacteria of the paratyphoid type are similarly affected, but *B. pyocyaneus* is more resistant. Bactericidal power varies with the dilution of the  $H_2O$  and is lost on addition of 5% of peptone or 1% of egg albumin, or by filtration. P. G. MARSHALL.

Photometric micro-analysis of potable water. I. C. URBACH (Mikrochem., 1932, 10, 483–504).—A general account is given, and available methods for the determination of  $NO_3'$  in  $H_2O$  are reviewed. Full details are given of the photometric determination of  $NO_3'$  by the brucine method. H. F. GILLBE.

Determination of the hardness of water. J. LEICK (Z. anal. Chem., 1932, 87, 81-110).-Comparative tests on various methods for determining the total hardness of H<sub>2</sub>O have shown that Blacher's K palmitate method (A., 1913, ii, 153) gives the most accurate results. Fe<sup>"</sup> up to 100 mg. per litre and Al<sup>""</sup> up to 30 mg. per litre do not interfere. Mn behaves like Ca and Mg and can be determined accurately by titration with K palmitate. Colloidal SiO2 and org. substances in the proportions usually found in boiler feed H<sub>2</sub>O do not interfere, whilst Na and K salts interfere only at high concns. Ca alone may be determined by pptn. as CaC<sub>2</sub>O<sub>4</sub> from feebly acid AcOH solution followed by titration with  $KMnO_4$ ; correct results are obtained by the use of an empirical factor : 1 c.c. 0.05N-KMnO<sub>4</sub>  $\equiv$ 1.466 mg. CaO. A. R. POWELL.

Precipitation of proteins in packing-house effluents by superchlorination. H. O. HALVORSON, A. R. CADE, and W. J. FULLEN (J. Physical Chem., 1932, 36, 185—197).—Biological methods are unsuitable for the treatment of such effluents owing to the high concn. of proteins or interfering substances. Pptn. of proteins by adjustment of  $p_{\rm H}$  requires too precise control, and pptn. with Fe or Al salts forms a sludge of high ash content, but pptn. with Cl<sub>2</sub> is quite practicable and extraction of the dried sludge with Et<sub>2</sub>O yields a product which may be fed to pigs. The amount of Cl<sub>2</sub> required, which is 2—10 times the amount of N in the effluent, is independent of the protein concn., but is increased by NH<sub>2</sub>-acids and peptones, although these are not pptd. F. L. BROWNE (c).

See also A., April, 354, Determining  $SiO_2$  in  $H_2O$ . 431, Chlorinated phenol homologues as bactericides.

# PATENTS.

Treatment of sewage. J. C. MARS (B.P. 366,510, 1.9.30. U.S., 22.10.29).—The sewage is freed from suspended solids by passing it through a succession of V-shaped, upward-flow straining filters composed of any porous refractory material. This medium and the sewage solids deposited thereon are removed continuously from the base of each filter and, after a preliminary dewatering, are subjected to destructive distillation in a current of hot flue gases. The surface level of the filters is maintained either by a portion of the charred material or by fresh supplies, if the original medium is of such a type, e.g., coke, that it can be used as the source of heat for the charring process.

C. JEPSON. Purifying device [for oily  $H_2O$ ]. Multilayer filter [for gas masks].—See I. Base-exchange bodies.—See VII.