# BRITISH CHEMICAL ABSTRACTS

# B.—APPLIED CHEMISTRY

FEB. 26 and MAR. 4, 1932.\*

# I.-GENERAL; PLANT; MACHINERY.

Colloidal conditioning of boiler feed water. O. E. BLINCOW (J. Inst. Brew., 1931, 37, 603-607). Ca and Mg salts in the water may be co-pptd. with added colloid in a condition that does not form scale; such a process does not introduce into the boiler equiv. quantities of Na salts. The tendency to prime and risk of caustic embrittlement are also reduced. Materials may be incorporated with the colloid to neutralise any acidity and to combine with dissolved  $O_2$ . D. K. MOORE.

Lubrication of automobile engines with olive oil. N. CHAMPSAUR (Bull. Mat. Grasses, 1931, 50, 254—260).—The retention of viscosity at high temp. and the fact that the compounds present contain unsaturated linkings, OH and  $CO_2H$  groups make olive oil a good lubricant. Its low viscosity at low temp. restricts its use to lighter engines of < 15 h.p. Satisfactory results on various automobiles are reported. The oil should contain < 4% of free fatty acids. A neutral Tunisian oil was found to have developed 4% of free acidity after a run of 1500—2000 km.

T. A. SMITH.

Tests of vacuum pan circulation. WERKSPOOR (Arch. Suikerind. Nederl.-Indië, 1931, 39, 912—917).— Using a small vac. pan which had been vertically cut through and provided with a window, observations were made of the circulation of the liquid (glucose syrup and very thick oil), using different systems of heating elements, but blowing in air in place of steam. Coils in general gave rise to less dead space than steam drums, even when the latter were provided with central circulation wells. J. P. OGILVIE.

Preparation of emulsions. W. CLAYTON (Chem. & Ind., 1932, 129—139).—A summary and discussion of published work on the prep., properties, and stability of emulsions, the functions of emulsifiers, the formation of foams, and a description of colloid mills and other technical emulsifying apparatus and methods.

# E. S. Hedges.

# PATENTS.

Rotary furnace. H. D. NEWKIRK, Assr. to ONON-DAGA STEEL Co., INC. (U.S.P. 1,809,563, 9.6.31. Appl., 18.6.29).—A furnace for making sponge Fe or otherwise needing exclusion of air is provided with a gastight closure at the burner end. Complete gastightness being impossible, the joint is subjected to a vac. applied to an outer annulus, while an inner annulus is watercooled. B. M. VENABLES.

Rotary kilns. E. RONNE (B.P. 362,361, 1.9.30).— With the object of increasing the contact between the slurry and gases at the wet end of a rotary kiln, the latter is provided with a baffle on the axis and the surrounding annular space with radial partitions and chains. Preferably the shell is made conical at the end to such an extent that the lower side becomes horizontal. B. M. VENABLES.

**Burning [calcination] of material.** A. ANDREAS (B.P. 362,517, 1.9.30. Ger., 2.9.29).—At the top of a shaft furnace in which the calcination is completed the material (*e.g.*, cement) is subjected to the action of the waste gases while on a travelling grate through which the gases pass downwardly. The fines should be removed by screening and a preliminary agglomerating process may be applied. B. M. VENABLES.

Furnaces for the supply of hot gases. L. HARDING and E. R. DEBENHAM (B.P. 362,498, 5.9.30).—The fuel is burned inside a refractory cylinder situated above a saucer-shaped firegrate which is provided with an upward extension forming an annular hopper. A hood is provided to draw off the gases, and air is passed between the hood and combustion chamber, between the combustion chamber and a surrounding metallic casing, as well as through the fire-grate. The regulation of the temp. of the gases is effected by altering the distance between the parts of the furnace.

B. M. VENABLES.

Apparatus for economising fuel and abating smoke in furnaces. G. DE BUYER-CHAILLOT (B.P. 362,429, 30.8.30. Fr., 30.8.29).—A hollow fire-bridge admitting preheated air and steam is described.

B. M. VENABLES.

Rotary dryer. G. M. BECHTEL (U.S.P. 1,808,626, 2.6.31. Appl., 13.5.29).—Baffles for insertion into the cylinder of a rotary dryer are described.

B. M. VENABLES.

Drying of crops, cut grasses, or other fibrous or flaked materials. L. HARDING and E. R. DEBENHAM (B.P. 362,487, 1.9.30. Addn. to B.P. 334,496).—The material is dried by currents of air while kept in motion by the rapid irregular vibration of perforated plates.

B. M. VENABLES.

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Drying apparatus. S. DIESEN (B.P. 362,680, 15.12.30).—In an apparatus suitable for fish, whale flesh, etc., comprising drying cylinders with interior conveyors, the cylinders are provided with access doors extending the whole length, in addition to inlet and outlet apertures. B. M. VENABLES.

Process and apparatus for catalytic reactions. L. GILLETT, ASST. to GEN. CHEM. Co. (U.S.P. 1,809,114, 9.6.31. Appl., 24.11.24).—In the oxidation of SO<sub>2</sub>, or a similar process, the gas is passed through four catalysers in series, each catalyser having an increased

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

bulk of catalyst in the same order as the gas flow. After passing through the first three catalysers, alternating with two heat exchangers, the gases are cooled and absorbed, passed through the heat exchangers, in which they are reheated to about their original temp., subjected to the action of the final catalyst, cooler, and absorber, and thence pass to waste. B. M. VENABLES.

Apparatus for effecting synthesis. R. WILLIAMS, Assr. to DU PONT AMMONIA CORP. (U.S.P. 1,809,291, 9.6.31. Appl., 28.3.30).—In an apparatus for effecting catalytic reactions which are exothermic and need a raised temp. to start them, the incoming gases are passed through one or more U-tubes embedded in the main body of the catalyst material and themselves also containing a catalyst. B. M. VENABLES.

Heat-transfer device. D. S. JACOBUS, ASST. to BABCOCK & WILCOX CO. (U.S.P. 1,808,169, 2.6.31. Appl., 20.6.29).—An apparatus suitable for use as an economiser is described. B. M. VENABLES.

Regulating and maintaining heat transfer [e.g., for vulcanisation purposes]. H. R. MINOR, Assr. to LIQUID CARBONIC CORP. (U.S.P. 1,808,429, 2.6.31. Appl., 7.2.28).—Heat and pressure for the vulcanisation of rubber articles are provided by a mixture of  $CO_2$ and steam, the former being in such quantity as to provide a partial pressure equal to the difference between the pressure desired and that of steam at the temp. desired. Air is excluded and under the damp conditions the  $CO_2$  has a preservative effect on the rubber bags used. B. M. VENABLES.

Crusher and pulveriser. H. J. SHELTON (U.S.P. 1,808,264, 2.6.31. Appl., 26.3.27).—A high-speed rotary crusher is provided with a crusher plate that is pivoted at the top and is adjustable at the lower end by tension rods outside the body of the crusher.

B. M. VENABLES.

Crushing or moulding machinery. A. W. SIZER (B.P. 362,605, 28.10.30).—An apparatus for producing pellets or cake from seeds etc. comprises a pair of hollow rolls having intercalating teeth on their surfaces and radial holes through which the material is squeezed by the action of the teeth, which are so shaped as to sweep out entirely the spaces between the opposing teeth. B. M. VENABLES.

Clarification of liquids. W. A. GILCHRIST (B.P. U.S., 9.9.29) .- A multi-decked 362,431, 1.9.30. thickener is operated with outward radial flow of both sludge and clear liquid, the separation taking place because the flow is laminar and the solids have only a short distance to settle. The rakes are provided with flexible blades, and the overflows are steeply inclined upwards to prevent clogging, their tops being adjustable in height to regulate the flows. A scum overflow is provided at the enlarged upper part of the axial feed-well. At the bottom of the apparatus the combined sludges are collected from the circumference and delivered to an axial outlet by rakes inclined in the opposite direction. B. M. VENABLES.

Filter. E. BURT (U.S.P. 1,808,917, 9.6.31. Appl., 10.12.28).—A concentric valve for air and sludge or

wash-liquor is placed in the trunnion of a filter as described in B.P. 4045/1909 (B., 1910, 404).

B. M. VENABLES.

[Double] distillation of liquids. ATLAS-WERKE A.-G. (B.P. 362,729, 15.1.31. Ger., 15.3.30).—Fresh water containing only about 10—15 mg. of residue per litre is obtained from sea-water by double distillation. The first evaporator contains a large water space and is heated by steam coils in the usual manner; the vapour from the first evaporator forms the heating medium of, and the condensate thus produced forms the liquid to be distilled in, a second evaporator of the circulating type. The latter has a vapour separator which is situated preferably above the vapour space of the first evaporator. To allow for blowing-down the second evaporator, part of the original condensed steam is admitted to it. B. M. VENABLES.

Treatment of liquids [e.g., boiler feed water]. W. S. ELLIOTT (U.S.P. 1,809,441, 9.6.31. Appl., 6.8.25). —Feed water is de-aerated by spraying and subjection to a vac. in two stages at room temp.

B. M. VENABLES.

**Production of emulsions.** P. LECHLER (B.P. 362,430, 1.9.30. Ger., 30.8.29).—The liquids are forced tangentially into a chamber having an axial outlet at the other end through which they issue as a spray. The liquids may first be heated and mixed (by known means) with emulsifying agents, *e.g.*, bitumen may have wool fat, tallow, oil, or resin, and water may have alkali, added to it. B. M. VENABLES.

Emulsifying apparatus [for gas in liquid]. R. H. HARGREAVES, ASST. (in part) to D. STEWART and A. M. RIESEN (U.S.P. 1,809,033, 9.6.31. Appl., 17.12.28).— The gas and liquid are supplied together upwardly into inverted, cup-shaped, perforated, centrifugal rotors which are superposed on the same vertical shaft.

B. M. VENABLES.

Controlling the application of reagents to solutions [e.g., of sugar]. W. C. GRAHAM, Assr. to GIL-CHRIST & Co. (U.S.P. 1,808,546, 2.6.31. Appl., 2.7.25). —The apparatus is particularly applicable to the addition of CaO to sugar solution. Both the sugar and lime-water are kept in rapid circulation, the former by a propeller and guides in a tank, the latter by a pump in a pipe circuit leading from and to a reservoir. CaO is sprayed into the sugar solution through a pipe branched off from the CaO circuit. The result of the reaction is observed in a window inserted in a by-pass or gauge attached to the tank, preferably when the circulation is momentarily stopped therein by a valve. Raw sugar solution may be continuously added to, and treated solution withdrawn from, the reaction tank.

B. M. VENABLES.

Bubble-tower cap. H. E. WIDDELL, Assr. to GASOLINE PRODUCTS CO., INC. (U.S.P. 1,808,276, 2.6.31. Appl., 21.12.23).—The caps are loose and are permitted to lift to pass an abnormal rush of vapour.

B. M. VENABLES.

Combining liquids and gases. S. G. KETTERER, Assr. to Schutte & KOERTING CO. (U.S.P. 1,808,956, 9.6.31. Appl., 17.6.30).—A pump draws liquid from an intermediate level of a vessel and delivers it to the lower part of the same vessel; on its way it entrains, by means of an ejector device, gas drawn from the upper part of the same vessel. B. M. VENABLES.

Utilising the waste gases from furnaces. B. M. JOHNSON, ASST. to CARBORUNDUM CO. (U.S.P. 1,809,628, 9.6.31. Appl., 13.8.28).—Gases from a furnace, used, e.g., for porcelain enamelling, are in part passed under a drying chamber through a flue having a false roof which is provided with adjustable apertures through which the gases are evenly distributed under the floor of the drying chamber forming the roof of the flue. The other part of the gases is used to preheat the air for combustion. B. M. VENABLES.

Apparatus for treating gases [e.g., removing naphthalene etc. from fuel gas]. F. W. SPERR, JUN., Assr. to KOPPERS Co. (U.S.P. 1,809,646, 9.6.31. Appl., 1.10.27).—A tower is filled alternately with hurdles and metallic wool or turnings. The bars of the hurdles are of rectangular cross-section and are arranged with the diagonals vertical and horizontal.

B. M. VENABLES.

Air or gas purifier. Air filter. [A, 1:] C. P. HEGAN and [A] W. B. DIECKS, ASSTS. to AMER. AIR FILTER Co., INC. (U.S.P. 1,807,983—4, 2.6.31. Appl., [A] 12.3.24, [B] 15.9.24).—In gas cleaners of the irrigated surface type, in (A) the baffles are of stream-line form having a width across the flow smaller than the depth along the flow; in (B) hollow, vertical baffles are provided, the viscous liquid being supplied to the interiors and caused to overflow down selected portions only of the outer surfaces. B. M. VENABLES.

Separation of desired liquefiable constituents from a gaseous mixture. E. H. LESLIE and E. M. BAKER (U.S.P. 1,808,420, 2.6.31. Appl., 22.11.24).— In the separation of petrol etc. from natural gas in a packed tower under pressure the heat of compression is used partly to reboil the petrol product to remove "wild gas." After further cooling (with condensation), both the gaseous and liquid products from the gas are admitted to different stages of the tower. B. M. VENABLES.

Apparatus for contacting two or more fluids. K. M. URQUHART (U.S.P. 1,808,088, 2.6.31. Appl., 29.1.29).—In order that a tower with trays may be adapted to pass upward currents of vapour of such magnitude that entrainment might be produced, the passages for upflow are in the form of cyclone separators. B. M. VENABLES.

Apparatus for metering gaseous fluids. A. E. BLAKE, ASST. to UNITED GAS IMPROVEMENT CO. (U.S.P. 1,808,709, 2.6.31. Appl., 18.10.28).—A radioactive element is placed in the conduit carrying the gas. When there is no flow the ions will lose their charges on the walls of the conduit, but in the case of a flow a number of the ions proportional to the flow will reach a discharge device comprising an insulated electrode in the conduit which is connected to the grid of a 3-electrode valve and other accessory apparatus to measure the discharge. B. M. VENABLES.

Apparatus for automatically withdrawing gas samples. J. F. AUSTIN and D. H. MCINTOSH, ASSIS. to AMER. SMELTING & REFINING Co. (U.S.P. 1,809,325, 9.6.31. Appl., 15.11.27).—An apparatus for operating valves in correct sequence is described. In the intervals between samplings the sample tube is supplied with compressed air. B. M. VENABLES.

**Evacuation of air or other gas from plastic or** viscous material. DUNLOP RUBBER Co., LTD., and H. SMITH (B.P. 362,531, 11.9.30).—The material is passed into a vac. chamber in the form of a continuously moving ribbon or other form thin enough to ensure that all the bubbles are on the surface and hence easily broken by the vac. The elongation may be effected by passing the material over a weir, through jets, or between rolls. B. M. VENABLES.

Fluid separators. D. SAMIRAN and P. MELVILLE (B.P. 362,466, 5.7.30).—A float-operated apparatus for insertion in a pipe line, *e.g.*, that supplying a pump for vending petrol, is described. On removal of the suction due to the operation of the pump the separated water drains away. B. M. VENABLES.

Hygrometers. R. G. BATESON (B.P. 362,543, 17.9.30).—A pair of wet- and dry-bulb thermometers having a powerful action, *e.g.*, of the Hg-in-steel type, are connected to a nomographic linkwork which gives a direct reading of relative humidity.

B. M. VENABLES.

Joints for resisting high pressures. S. R. BOYCE, F. S. LUNDY, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 362,689, 18.12.30).—A gasket ring is constructed of hard metal and has a U- or V-shaped cross-section with the point or bend of thinner metal than the legs. An auxiliary ring (in parts for convenience) bridges the opening, but vents are provided so that the space between the legs is always open to the pressure.

B. M. VENABLES.

Steam dryers, grit or dust extractors for air or gases, etc. F. K. O. MOYNAN (B.P. 362,723, 12.1.31. Addn. to B.P. 341,532; B., 1931, 370).

Air preheaters for furnaces. UNDERFEED STOKER Co., LTD., and W. F. HARLOW (B.P. 363,559, 2.3.31. Addn. to B.P. 346,114).

Sharp freezing apparatus. COPEMAN LABORA-TORIES Co., Assees. of L. G. COPEMAN (B.P. 363,625, 21.8.30. U.S., 30.9.29).

Drying of comminuted material. Separation of gas mixtures.—See II. Rolling-mill piercing points etc. Alloys resistant to steam. Apparatus for hydrogenations. Cylinder linings.—See X. Pptn. of particles from gases.—See XI. Boiler water.—See XXIII.

# II.-FUEL; GAS; TAR; MINERAL OILS.

Determination of moisture in coals by drying in vacuum. R. VONDRÁCEK and L. MOSENDZ (Brennstoff-Chem., 1931, 12, 445—446).—The moisture contents of coals determined by the loss of wt. on drying in vac. at 100° were greater than those obtained by drying at 105° under the ordinary pressure, e.g.,  $14\cdot35\%$  and  $17\cdot44\%$  for two brown coals and  $2\cdot85\%$  and  $1\cdot31\%$  for two bituminous coals, as compared with the corresponding vals.,  $12\cdot58\%$ ,  $16\cdot34\%$ ,  $2\cdot12\%$ , and  $0\cdot85\%$ , obtained by the second method. The losses in wt. on drying in vac. at even lower temp., e.g.,  $60^\circ$ , were still greater than the moisture contents as determined by the more usual method. It is uncertain whether these differences are due to the loss of hydrocarbons in the coal or to actual decomp. of the coal under reduced pressure.

A. B. MANNING.

Determination of moisture in fuels. C. PADOVANI and C. SINIRAMED (Atti III Cong. Naz. Chim. pura appl., 1929, 778—781; Chem. Zentr., 1931, ii, 169).—The authors' apparatus has been improved.

A. A. ELDRIDGE.

Determination of the various forms of sulphur in South African coals and shales. P. E. ROUSSEAU (Brennstoff-Chem., 1931, 12, 446-449; cf. Blom, B., 1929, 767).—Five coals and four shales were examined. The following methods were found suitable: total S (which varied from 0.15% to 1.6%) by Eschka's method, using an Fe crucible; sulphate S by extraction with 3% HCl at 60° for 40 hr.; total inorg. S by extraction with  $HNO_3$  (d 1.12) at room temp. for 72 hr. The total S in the PhOH-insol. fraction of the coal or shale was also determined, and the humin S calc. as the difference between org. S and PhOH-sol. S. With some of the coals an exact correlation was found between the pyritic S, calc. as the difference between the inorg. S and the sulphate S, and the pyritic Fe; the correlation failed with one coal and with three shales.

A. B. MANNING.

Nomograms for calculating the calorific value of coal from the elementary analysis. W. SCHREIBER (Chem. Fabr., 1931, 4, 495).—The nomograms are based on the following equations : calorific val. (for bituminous coal) =  $81C + 290(H - O/8) + 25S - 6H_2O$ ; (for lignite and peat) =  $85C + 216H + 25S - 25 \cdot 50 - 6H_2O$ . In both expressions the chemical formulæ and symbols represent the % of the constituent found by analysis. A. R. POWELL.

Calculation of the calorific value of coal. DE CAHER (Gas World, 1931, 95, 596—599).—The accuracy obtained with various types of calorimeter is discussed. Objections to the use of formulæ based on ultimate analyses are quoted and the several formulæ available for calculating the calorific val. from proximate analyses are critically examined. Nomographic methods for calculating this value of the coal as received or on the dry, ash-free basis from its proximate analysis are given. C. B. MARSON.

Thermodynamics of difference between gross and net heating values, solid and liquid fuels. L. C. LICHTY and B. L. BROWN (Ind. Eng. Chem., 1931, 23, 1419-1421).-A temp.-vol. or -pressure diagram is shown representing the burning and cooling process which illustrates the reason for, and defines the difference between, gross and net vals. For const.-vol. bomb determinations a difference of 970 B.Th.U. per lb. of H<sub>2</sub>O vapour formed is suggested. For the const.pressure combustion process in air of 100% R.H., the difference is 1070 B.Th.U. per lb. of H<sub>2</sub>O formed, regardless of the fuel or the temp. at which it is burned; below 100% R.H. the difference may vary from 1070 to 0 B.Th.U. The difference between gross and net heating vals, for the const.-pressure process per lb. of H<sub>2</sub>O vapour condensed was found to equal the latent heat

of  $H_2O$  at the final temp., and this relationship is proved thermodyamically. C. B. MARSON.

Net and gross heating values [of fuels]. H. C. PORTER (Ind. Eng. Chem., 1931, 23, 1433-1434).-In America the heating vals. of solid, liquid, and gaseous fuels are generally given as the gross vals. obtained by the laboratory calorimeter and include the heat derived from condensation of both H<sub>2</sub>O vapour and the original moisture in the fuel. The amount of heat derived from these sources, although hardly ever realised in practice, is 500-550 B.Th.U. per lb. for solid, 1000-1200 for liquid, and 2100 for gaseous fuels. The net calorific val. is lower than the gross val. by an amount resulting from one single correction factor, viz., that depending on the H<sub>2</sub>O formed in combustion. The A.S.T.M. method for determining the calorific val. of coal and coke prescribes a deduction of 1040 B.Th.U. for each lb. of H<sub>2</sub>O formed (cf. Lichty and Brown's val., 970, in preceding abstract). The use of these two terms in assessing the relative efficiency of a fuel under practical conditions is discussed. C. B. MARSON.

Effect of fine inerts on agglutinating power of Pittsburg coals. J. D. DAVIS and W. D. POHLE (Ind. Eng. Chem., 1931, 23, 1427-1431).-The addition of up to 25% of finely-ground, 100-mesh, inert materials such as pyrites, gypsum, calcite, and fusain to the coal increased the strength of the coked buttons obtained in the Marshall-Bird test (using a ratio of 10 pts. of sand to 1 pt. of coal + filler). Substitution of electrode C for the sand prescribed in the test, together with addition of the same inert materials to the coal, weakened the coke buttons. The effect of fusain was confirmed by actual coking tests on 80-lb. charges. It is concluded that further investigation of the various factors involved in the test is necessary before it can be confidently applied to the solution of practical problems relative to coke quality, and it is also suggested that several dilution ratios, in addition to the prescribed 10:1 ratio, should C. B. MARSON. be employed.

Report of test by the Director of Fuel Research on the plant of the Leicestershire (L. & N.) Coal Distillation Co., Ltd., at Newbold, near Ashby de la Zouche (Dept. Sci. Ind. Res., 1931, 27 pp.) .- The retort is a cylindrical steel shell 90 ft. long by 9 ft. in diam., inclined at an angle of 1 in 20 to the horizontal. At the lower and hotter end it is lined internally with firebrick, and the rest of the retort is lined with a heatinsulating material. When working at its nominal capacity (100 tons/day) the retort is rotated once per min. Inside the shell are packed 7 steel cylinders, each 30 in. in diam., extending a distance of 55 ft. from the upper charging end; as the retort rotates, coal is fed into these tubes in turn and from them is discharged after about 2 hr. into the outer shell. At the lower end of the retort the coke is discharged through a slide valve into an outer annular compartment, and thence, through a second slide valve, to air. The charge is heated by the products of combustion of producer gas, cooled to about 700° by admixture with recirculated mixed combustion and distillation gas. About 75,000 cu. ft. of heating gas are required to carbonise 1 ton of coal; the distillation gas is thereby so diluted that its calorific val. is reduced to 40 B.Th.U./cu. ft. The gases and vapours leaving the retort are passed through dust separators and condensers; most of the cooled gas is recirculated and the remainder is discharged to atm. A washer is provided for scrubbing the light spirit from the gas, but this was not in operation during the test. The coal used was an inferior quality slack, under  $\frac{3}{4}$  in. in size. The air-dried coal had the analysis :  $H_2O$  10.4, volatile matter 35.7, fixed C 44.6, ash 9.3%; as supplied to the retort, however, the  $H_2O$  content was  $19 \cdot 2^{\circ}$ . The test period was 125.75 hr., but owing to stoppages, due mainly to choking of the coal feed and trouble with the coke washer, the period during which coal was fed to the retort was only 96 hr. The operation of the retort itself gave little trouble. The total throughput of coal was 340.6 tons (wet), and the yields of products per ton of dry coal were coke 0.63 ton, tar 14.1 gals., and gas to atm. 39,530 cu. ft.;  $2 \cdot 0$  gals. of tar per ton of dry coal were found in the sludge from the dust tower and 0.8 gal. as uncondensed tar fog, giving a total yield of 16.9 gals., or 59.3% of the Gray-King assay yield. The yield of liquor could not be measured. The tar was deficient in constituents boiling below 170°. The heat required for carbonisation was 31 therms per ton of coal; the steam consumption was 2000 lb./hr.; the labour employed was 7 men per shift and two additional men on day shift only. The coke was separated into "fuel" and "dirt" by flotation in water; the "fuel" was briquetted with pitch, the briquettes being either used as made or subsequently baked. Both types of briquette formed a satisfactory domestic fuel. A. B. MANNING.

Influence of the coal constituents on the quantity and composition of the gas evolved on carbonisation. H. BRÜCKNER and W. LUDEWIG (Brennstoff-Chem., 1931, 12, 465—467).—The  $\alpha$ ,  $\beta$ ,  $\gamma_1$ ,  $\gamma_2$ , and  $\gamma_{3+4}$  constituents of a bituminous coal (cf. Cockram and Wheeler, B., 1927, 401) were separately carbonised by heating rapidly to 1000° *in vacuo*, and the evolved gases were analysed. The  $\beta$  constituents gave the highest yield of gas, whilst the  $\gamma_1$  and  $\gamma_2$  constituents gave much tar and but little gas. The gas yield from the original coal was greater than that calc. from the yields obtained from the individual constituents, and the yields from mixtures of the constituents were similarly greater than the calc. yields. It is concluded that some interaction of the constituents occurs during carbonisation.

A. B. MANNING. Salerni system of low-temperature carbonisation. R. V. WHEELER (Gas J., 1931, 196, 783-785).-The plant consists of a continuous, slightly inclined, rotary furnace, enclosed in an outer casing, which is hydraulically sealed, thus eliminating distortion troubles. Three separate concentric ducts serve to introduce the heating gas, to remove the distillation products, and to provide a stream of gas to cool the walls of the vessel. Mixing of these gases is prevented by regulation of their respective velocities and pressures; no mechanical seal is provided. The coal to be carbonised is blended with part of the hot semi-coke produced, ground to pass 60-mesh I.M.M., and mixed with the heavy fractions of the low-temp. oil. The semi-coke thus has a high sp. gr. and coherency, and is rich in volatile matter. The yield of light oil is increased and dust troubles are eliminated. R. N. B. D. BRUCE.

"Bertinisation" of brown coal. C. STAEMMLER (Brennstoff-Chem., 1931, 12, 449—451; cf. Seidenschnur, B., 1927, 737; Thau, B., 1931, 790).—By preheating brown coal to 300°,  $H_2O$ ,  $CO_2$ ,  $H_2S$ , etc. are evolved and the products, especially the gas, obtained by subsequently carbonising the coal at higher temp. are improved in quality. The gas yield obtained during 2 hrs.' preheating represents about 30%, and the tar yield 6-7%, of the assay yield, the gas containing 50% or more of inert material. If the preheating at 300° be carried out in a current of scavenging gases a considerable proportion of the tar, e.g., 40% of the assay yield, may be evolved during the process. This tar may be separately collected or a lower preheating temp. may be employed. A. B. MANNING.

Fuel economy and control in the basic industries, with special reference to iron and steel works. J. S. KERR (J. Inst. Fuel, 1931, 5, 148—159).—The economies to be effected by combining coke ovens, blast furnaces, steel-making plant, and rolling mills, together with the separate economies which may be made in each of the individual processes, are discussed.

C. B. MARSON. Selected problems relative to the coal industry. B. POCHOBRADSKY (J. Inst. Fuel, 1931, 5, 102—107).— Chiefly a progress report of the industry in the United Kingdom, dealing with production and reasons for variations in the consumption of coal. Attention is drawn to the necessity for cleaning and grading coal, and the economies of low-temperature carbonisation and hydrogenation are briefly considered.

C. B. MARSON.

**Purification of coal gas in a rotary drum.** P. FRITZSCHE (Brennstoff-Chem., 1931, 12, 468—469).— The gas was passed through a horizontal rotary drum, about 2 m. in length, through which at the same time a quantity of "luxmasse" was being slowly conveyed in the opposite direction by the motion of the drum. Baffles were affixed to the inner walls of the drum for continuously lifting the purifying material and allowing it to fall through the gas. The efficiency of purification was equiv. to that obtained with an ordinary purifying box of 20 times the vol. A. B. MANNING.

Wet desulphurisation of coal gas by the Gesellschaft für Kohlentechnik process, with simultaneous combination with ammonia and without precipitation of sulphur. W. GLUUD, W. KLEMPT, and F. BRODKORB (Ber. Ges. Kohlentech., 1931, 3, 466-484)—The S produced in the washer in the ordinary wet process is converted into (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>3</sub> by using a solution of  $(NH_4)_2SO_3$  and ammoniacal gas. If the ratio NH3: S in the crude gas is insufficient for this the excess S must be pptd. as usual. The (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>3</sub> produced is treated with H2SO4, HNO3, or H3PO4, according to the product required. The speed of dissolution of S in (NH<sub>4</sub>)<sub>2</sub>SO<sub>3</sub> is increased by rise in temp. and increase of (NH<sub>4</sub>)<sub>2</sub>SO<sub>3</sub> concn. The presence of the (NH<sub>4</sub>),SO<sub>3</sub> does not affect S removal, but increases the time of oxidation of FeS, though not very seriously. Some oxidation of sulphite to sulphate occurs. Smalland large-scale tests of this process on coke-oven gas are described. The SO<sub>2</sub> used was obtained from cylinders, no burners being available. The purified gas contained

0.02 g, of H<sub>2</sub>S, 0.01 g, of HCN, and 0.2 g, of NH<sub>3</sub> per cu. m. The solution obtained contained  $(NH_4)_2S_2O_3$ 25%,  $(NH_4)_2SO_4$  3%, and NH<sub>4</sub>CNS 1.4%. The lastnamed compound is undesirable and a washer should be put in to remove cyanides previously. The acid treatment of the  $(NH_4)_2S_2O_3$  was carried out in a covered enamel vessel with heating to 95—100°. The S pptd. was skimmed off and the liquor neutralised with conc. aq. NH<sub>3</sub> and evaporated. C. IRWIN.

Removal of sulphur from [ammonium sulphate] saturator exit gases. W. GLUUD, W. KLEMPT, and F. BRODKORB (Ber. Ges. Kohlentech., 1931, 3, 497— 504).—The saturator waste gases studied contained 30-40% H<sub>2</sub>S and  $1\cdot1\%$  HCN. After cooling and diluting with air they were treated with ammoniacal Fe(OH)<sub>3</sub> suspension in the same way as ordinary coal gas, working at  $35-40^{\circ}$ . The process worked normally, but the oxidation tower of the plant available was found to be much too small to deal with the H<sub>2</sub>S fed to it. The possibility of hastening oxidation by injection of steam was tested in the laboratory, but aggregation of the Fe(OH)<sub>3</sub> occurred, with detrimental results, at above 55-60°. This does not occur, however, with low NH<sub>3</sub> content. C. IRWIN.

Dilution of straight coal gas by producer gas. C. A. DEAS (Gas J., 1931, 196, 779–781).--29 vol.-% of producer gas of calorific val. 133 B.Th.U. per cu. ft. was needed to dil. rich coal gas (calorific val. 650) to the standard val. (500). The gases were mixed in the foul main prior to the exhauster. No difficulties were experienced in maintaining the flame temp., but the capacity of the mains decreased by 7%. The  $C_{10}H_8$  content increased owing to the smaller amount of unsaturateds present. The cost of the producer gas was 1.55d. per therm, the thermal yield per ton of coal carbonised being increased from 71 to 77 therms.

R. N. B. D. BRUCE.

Automatic control of calorific value of coal gas. W. R. D. TILDESLEY (Gas J., 1931, 196, 781-783).---A description of the apparatus claimed in B.P. 356,425 (B., 1931, 1036) is given. The pen arm of a recording calorimeter is connected to a valve, which controls the vac. exerted by a hydraulic governor attached to the exhauster inlet. Alteration in the height of the governor actuates a butterfly valve in the diluent gas main, and so adjusts the calorific val. of the gas to the standard required. R. N. B. D. BRUCE.

Thermal decomposition of low-temperature tar constituents. III. Thermal decomposition of neutral constituents of low-temperature tar. IV. Effects of filling materials on the yield of lower phenols. Y. KOSAKA (J. Soc. Chem. Ind., Japan, 1931, 34, 345—346 в, 348—349 в; cf. B., 1931, 956).— III. Neutral oil, b.p. 150—340°,  $d^{25}$  0.8945, from lowtemp. tar consists chiefly of complex unsaturated aromatic hydrocarbons, which are converted by passage over SiO<sub>2</sub> at 800° into C<sub>6</sub>H<sub>6</sub> hydrocarbons (by dealkylation) and polycyclic compounds (by condensation). Hydrogenated naphthalenes and anthracenes are probably also present.

IV. For the thermal conversion of higher into lower phenols at 700° the order of activity is : coke, Ni, Fe,

Cu, charcoal, acid clay, bone charcoal. Each has, however, an optimum temp., and adsorbent materials such as bone charcoal and acid clay are very effective in lowering the temp. of decomp. The yield of phenols increases with velocity of the vapours at 725°.

Mechanism of the formation of coal-tar constituents. Y. KOSAKA (J. Soc. Chem. Ind., Japan, 1931, 34, 347—348 B).—Acidic and neutral constituents of low-temp. tar play about equal parts in tar formation. Benzenes are derived from phenols and naphthenes by reduction, dealkylation, and dehydrogenation; PhOH and cresols from higher phenols;  $C_{10}H_8$ , anthracene, etc. by ring-condensation of complex aromatic hydrocarbons and also from their OH- and hydro-derivatives; diphenyls from benzenes and phenols. The benzenoid and polycyclic structures present in coal substance survive through low-temp. tar to coal tar.

C. HOLLINS.

Adaptation of cracking to European requirements. M. STÉMART (Chim. et Ind., 1931, 26, 521-530, 1023-1039).-The design and operation of a Dubbs cracking plant (capacity 300 barrels per day) erected for the Belgian Cracking Co., at Langerbrugge, are described. The results obtained by cracking mazout or gas oil therein show that from the economic viewpoint such units can be run successfully in Europe when imported raw material is used. The process may be so controlled as to leave either a liquid residue or coke. Yields of products obtained in tests are given in full. The crude motor spirits produced can be refined without difficulty by known methods. Distillation is effected in a pipe still. By cracking a topped low-temp. tar about 25 vol.-% of motor spirit was obtained; higher yields are to be expected if true "primary" tars are cracked. For an ordinary low-temp, tar it is recommended that the tar be distilled to hard pitch and only the oils cracked. Removal of phenols from the crude spirit can be effected by treatment with Ca(OH), in a Plauson colloid mill. A. B. MANNING.

Properties and composition of gasoline fractions of representative Japanese crude petroleums. V. M. MIZUTA (J. Soc. Chem, Ind., Japan, 1931, 34, 407 B; cf. B., 1931, 1081).—The aniline-point depression for each 1% of aromatic hydrocarbon in petrol increases with the content of aromatic hydrocarbon, and shows a sudden change at 35%. It is independent of the nature of the aromatic hydrocarbon ( $C_6H_6$ , PhMe, or xylene). C. HOLLINS.

Composition of the light distillates of Japanese petroleum. M. AKITA (Trans. II World Power Conf., 1930, 8, 21–29).—Vals. of  $d_4^{15}$ , S, and % having b.p.  $< 200^{\circ}$  are recorded. The olefine content of the fraction is negligible; the average content is : naphthenes 38, paraffins 49, aromatics 13%. CHEMICAL ABSTRACTS.

Synthetic acids from Emba solar oil. A. D. PETROV and I. Z. IVANOV (J. Appl. Chem., Russia, 1931, 4, 77—85).—The naphthenic acids are stable on distillation, the oxy-acids which decompose on heating tending to form hydrocarbons. Anhydrides can be hydrated by heating the mixtures of acids with H<sub>2</sub>O under pressure. CHEMICAL ABSTRACTS.

168

C. HOLLINS.

Isolation and determination of methylcyclopentane in a midcontinent petroleum. M. M. HICKS-BRUUN and J. H. BRUUN (Bur. Stand. J. Res., 1931, 7, 799—809).—The isolation of methylcyclopentane from petroleum by nitration of the  $C_6H_6$  and fractional distillation and melting is described. The following data for the  $(98 \cdot 7 \pm 0.2 \text{ mol.-}\%)$  substance are recorded : b.p.  $71 \cdot 8^{\circ}$ , f.p.  $-141 \cdot 9^{\circ}$  to  $-142 \cdot 3^{\circ}$ ,  $d_4^{30} 0.7487$ ,  $n_{20}^{30} 1.4098$ , crit. solution temp. with NH<sub>2</sub>Ph  $34 \cdot 7$ , latent heat of fusion  $7 \cdot 6$  g.-cal. per g. The binary eutectic mixture of methylcyclopentane and *n*-hexane contains  $95 \cdot 9 \text{ mol.-}\%$  of the former and has m.p.  $-143 \cdot 5^{\circ}$ . The infra-red absorption spectrum has also been determined. E. S. HEDGES.

Isolation of *n*-nonane from a midcontinent petroleum. J. D. WHITE and F. W. ROSE, JUN. (Bur. Stand. J. Res., 1931, 7, 907—911).—*n*-Nonane (99·9%) was isolated from the petroleum by fractional distillation and fractional crystallisation. The following data are given:  $d_{4}^{\infty}$  0.71793  $\pm$  0.00003,  $n_{2}^{\infty}$  1.4033  $\pm$  0.0001, b.p. 150.73°  $\pm$  0.01°, f.p.  $-53.65^{\circ}$ . The infra-red absorption spectrum has also been measured. These data are compared with those for synthetic *n*-nonane.

E. S. HEDGES.

Catalysis [decomposition] of Rumanian petroleum. J. POPA (Chim. et Ind., 1930, 26, 1292—1296).— Metallic catalysts produced relatively little decomp. at  $550^{\circ}$  (max. temp.), but commercial activated carbons gave good yields of catalytic decomp. products at  $500-700^{\circ}$ . The products obtained at  $600-650^{\circ}$  contained xylene and PhMe, C<sub>6</sub>H<sub>6</sub> appearing at  $700^{\circ}$ . The amount of diolefines in the product increased with rising temp. E. S. HEDGES.

Distillation of calcareous bituminous rocks for production of mineral oil. A. LA PORTA (Trans. II World Power Conf., 1930, 8, 72—90).—The oil is of asphaltic base, but contains little hard asphalt and is free from resinous substances; it is sol. in  $\text{Et}_2$ O, petroleum,  $C_6H_6$ ,  $CS_2$ , and  $\text{CCl}_4$ . The average mol. wt. is 132—145; I val.  $37 \cdot 5\%$ . CHEMICAL ABSTRACTS.

Acid and vapour-phase methods of refining cracked gasolines. I. BESPOLOV and A. DUDENKO (Azerbaid. Neft. Choz., 1931, No. 5, 70—74).—The fraction b.p. 175—200° of cracked Baku gasoline was agitated for 10 min. with 1, 3, 5, 10, and 30% of 93%  $H_2SO_4$ , washed with water, neutralised, again washed with water, and redistilled to 200° end-point. The Saybolt colours initially and after 1 month in the dark were, respectively: 2, 2; 16, 12; 17, 17; 15, below —9; 17, 3. The most stable product obtained by refining with 5% acid lost 15—20% of the aromatic substances. Colourless gasoline was obtained by vapourphase refining with floridin. A combination of acid and vapour-phase treatment is advocated.

CHEMICAL ABSTRACTS.

Gum formation in motor benzols. H. A. J. PIETERS and H. S. VISSER (Brennstoff-Chem., 1931, 12, 470-472).—The tendency of a motor benzol to form gum, as shown by the gum production on refluxing the benzol in a current of  $O_2$ , is greatest in the fractions boiling below about 86° and above 116°, and is negligible in the large intermediate fraction, although the latter

still contains unsaturated hydrocarbons and has in consequence a high anti-knock val. It is suggested therefore that the crude benzol should be fractionated, and that only the first and last fractions should be refined with conc,  $H_2SO_4$ . A. B. MANNING.

[Fuel value of] vegetable oils. GAUTHIER (Bull. Mat. Grasses, 1931, 50, 232–237).—In engine tests with arachis oil as fuel, the consumption was greater than that of gas oil, but less than might be anticipated from the lower calorific val. of the arachis oil. Similar observations were made on the thermal efficiency, thus indicating that arachis oil is a better cylinder oil than is gas oil. No mechanical difficulties were met with and the engine started easily when cold. Arachis oil may be used without preheating, thus differing from castor oil, palm oil, and karité butter. Badly deteriorated oils were found to be serviceable in engines, and oils containing up to 28% of fatty acids have been used without inconvenience. T. A. SMITH.

Relation between physico-chemical characteristics of fuels and the construction of internalcombustion engines. LESCHÈRES (Bull. Mat. Grasses, 1931, 50, 237—241).—A discussion of the influence of the nature of the fuel to be used (gas oil or petrol) on engine design. T. A. SMITH.

Use of heavy oils in lorry engines. DE GOTIRAU (Bull. Mat. Grasses, 1931, 50, 241—244).—Tests with tar as fuel for high-speed Diesel engines gave unsatisfactory results. Vegetable oils have been used, but the consumption is 25% higher than with gas oil, owing to the low calorific val., and the price precludes their use in France. T. A. SMITH.

Improved paraffin-base lubricating oils [for internal-combustion engines]. G. H. B. DAVIS and A. J. BLACKWOOD (Ind. Eng. Chem., 1931, 23, 1452-1458) .- The dewaxing of paraffin-base oils so as to obtain the necessary fluidity (pumpability and quick distribution) at low temp. affects adversely other properties of the oil. The viscosity index is decreased, with resultant difficulty in starting; the tendency to form C is increased; the volatility of the oil is increased, causing increased oil consumption; the load-carrying ability and "oiliness" are decreased; the resistance to oxidation is decreased with consequent sludging in the engine and other deleterious effects. By the addition of a very small quantity of a specially-prepared pure hydrocarbon lubricating oil "Paraflow" (Ú.S.P. 1,815,022) the pour point is reduced without the necessity for severe dewaxing and the consequent disadvantages given above. The lubricating characteristics of the low-pour oil thus produced are superior to those of the original oil. Low pour point oils can be produced more economically by the use of "Para-C. B. MARSON. flow " than by dewaxing.

Lubrication with olive oil. Vac.-pan circulation. —See I. Hydrogenation of  $C_{10}H_8$  etc.—See III. Treatment of pulp liquors.—See V.  $(NH_4)_2S_2O_3$ . CO recovery from gases.—See VII. Vaseline for smokeless powder. Gas from sludge digestion.— See XXIII.

See also A., Jan., 25, Flame temp. 27, Formation of aromatics from lower paraffins.

#### PATENTS.

Washing and draining of coal or other minerals. CLEAN COAL CO., LTD., R. LESSING, and R. H. ALLEN (B.P. 361,906, 23.6.30).—Coal etc. which has been cleaned by flotation in a solution of high sp. gr. (cf. B.P. 325,031; B., 1930, 403) is freed from the adhering solution by immersing it in an aq. liquor of lower sp. gr. than that admixed therewith, withdrawing it from the liquor up an inclined surface, whereby a substantial proportion of liquor flows back between the particles into the main body, and then passing it over a perforated surface where more drainage takes place. The process is repeated with liquors of progressively decreasing concn. A. B. MANNING.

Briquetting of fuel material. A. D. LITTLE, Assr. to A. D. LITTLE, INC. (U.S.P. 1,809,245, 9.6.31. Appl., 14.6.28).—Fine coal is mixed with cellulose hydrate, dispersed in an excess of  $H_2O$ , to a plastic mass, which is then briquetted and air-dried to a  $H_2O$  content producing irreversibility in the colloidal cellulose.

A. B. MANNING.

Fuel briquette. R. P. BOTSCH, ASST. to UNITED COAL & DOCK CO. (U.S.P. 1,808,340, 2.6.31. Appl., 26.10.29).—Pulverised semi-bituminous or anthracite coal is briquetted, preferably with a waterproof, asphaltbase binder, and, in order to prevent the production of dust during handling, the briquettes are lightly coated with a mineral oil having an affinity for the binder.

A. B. MANNING.

Briquetting of coal and other materials. J. ROGERS (B.P. 362,570, 29.9.30).—The binding agent, e.g., pitch, is incorporated with the material to be briquetted, which is preheated to 100—200°, in the form of a froth produced by projecting a jet of water on to a jet of molten pitch. A. B. MANNING.

Coking apparatus. F. PUENING (B.P. 362,522, 10.9.30. U.S., 1.10.29).—An apparatus for the lowtemp. carbonisation of coal consists of a series of narrow vertical coking chambers separated by hollow rectangular heating walls, supported from horizontal beams; the walls can be swung about vertical axes adjacent to one edge in such a manner as to enlarge the coking chambers sufficiently to facilitate the discharge of the coke. A fluid heating medium is circulated through a furnace and thence through the heating walls, the entrance and exit flues of which are given the necessary flexibility for the walls to be moved without permitting mixing of the heating and distillation gases. The mechanism for moving the walls is so designed that none of the linkage mechanism is subjected to a high temp.

#### A. B. MANNING.

Heating of coke ovens. P. E. VERPEAUX, and UNION CHIM. BELGE, Soc. ANON. (B.P. 362,427, 27.8.30).— Gas is supplied at a const. rate to the bottom of each flue, while air is supplied at two or more levels therein, the rate of supply of air at each level being automatically varied in any desired manner while the total air supply is maintained const. between two reversals of the current. Or one air supply may be provided at the bottom of the flue and gas supplied at two or more levels therein, the composition of the gas supplied at each level being automatically varied, e.g., by admix-

ing two gases in varying proportion, while maintaining a const. total rate of heat supply. A. B. MANNING.

Production of coke and water-gas in vertical chamber ovens. C. OTTO and C. W. ANDREWS (U.S.P. 1,809,421, 9.6.31. Appl., 8.5.25).—When the coal in the upper part of the chamber is coked, but before that in the lower part is also coked, steam is admitted to the top of the chamber, and the water-gas so produced, together with the distillation gas still being produced, is withdrawn at the bottom of the chamber. The water-gas may be carburetted by simultaneously supplying oil to the chamber. A. B. MANNING.

Manufacture of semi-coke. F. TOTZEK, Assee. of H. KOPPERS A.-G. (B.P. 362,040, 17.9.30. Ger., 29.11.29). —Coal is carbonised in externally heated oven chambers, the partition walls separating the chambers from the combustion spaces being of refractory material designed as to its thickness and/or its material to produce a temp. gradient across it sufficient to ensure that the temp. within the oven chamber is not above that requisite for the production of semi-coke. The wall is preferably composite, comprising a layer of CaO-bound SiO<sub>2</sub> bricks and a layer of fireclay bricks. The design of the remainder of the apparatus may follow general coke-oven practice employing regenerative heating.

A. B. MANNING.

Low-temperature distillation of coal, cannel coal, and like fuels. H. STEVEN (B.P. 362,581, 6.10.30. Belg., 5.10.29).—The coal is conveyed through an externally-heated retort, e.g., by means of scraperchains on an endless belt, and is carbonised therein at 420—500° under a partial vac., e.g., a pressure of  $\frac{1}{2}$  atm. The feeding and discharging devices, which may be of the reciprocating piston type, are so designed that both ends of the retort are sealed by a layer of the fuel sufficiently impervious to maintain the low pressure therein, A. B. MANNING.

Manufacture of carbon black. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 361,837, 21.6.30).— CO, unsaturated hydrocarbons, etc. are decomposed by heat in the presence of finely-divided solid which is maintained in suspension in an upward current of the heated gas. Metals of the Fe group, to which preferably an activator is added, form suitable catalysts.

A. B. MANNING.

Drying of carbonaceous comminuted decolorising material. L. WICKENDEN and S. A. W. OKELL (B.P. 362,112, 31.10.30).—The material is heated in a horizontal receptacle along which it is advanced by means of paddles which are rotated so slowly that substantially no dispersion of the material takes place. A regulable amount of air is admitted to the dryer. The material is cooled by means of a water-jacket surrounding the discharge end of the dryer, in order to prevent its igniting on discharge into the atm.

A. B. MANNING.

Continuous revivification of decolorising carbons and the like. L. WICKENDEN and S. A. W. OKELL (B.P. 362,609, 31.10.30. Cf. B.P. 362,112; preceding abstract).—The spent C is heated in the presence of a regulated quantity of air in a firebrick trough along which it is conveyed and simultaneously stirred by the

# British Chemical Abstracts-B.

171

slow rotation of a series of paddles. Heating is effected electrically by passing a current through the material between pairs of electrodes arranged at intervals along the inner walls of the trough. When the heating is completed the stirring is continued until the material is cool enough to permit it to be exposed freely to the air. A. B. MANNING.

Manufacture of products from carbonaceous material. H. D. ELKINGTON. From N. V. DE BATAAF-SCHE PETROLEUM MAATS. (B.P. 362,354, 25.8.30. Cf. B.P. 353,506 and 358,180; B., 1931, 873, 1130).— Coal, petroleum, or tar, etc. is heated with H<sub>2</sub> under pressure in the presence of catalysts comprising Re or a compound thereof deposited in colloidal form on a carrier, e.g., activated C, together with other metals or compounds, e.g.,  $MoO_{32}$  also similarly deposited.

A. B. MANNING.

Manufacture of water-gas. O. B. Evans, Assr. to U.G.I. CONTRACTING CO. (U.S.P. 1,808,214, 2.6.31. Appl., 3.4.24).-Two separate fuel beds are provided in two intercommunicating water-gas generators (A, B), each of which is connected to a corresponding carburettor and superheater. Air is passed through one superheater and carburettor to the centre of the fuel bed in A, part of it passing through the upper part of the bed and thence into the top of B, the remainder passing through the lower part of the bed and thence into the bottom of B. The gases leaving B (from the centre of the fuel bed) are burned with secondary air in the other carburettor and superheater. If desired, secondary air may also be admitted at a point between the generators. The air blast is followed by a steam blast in the same direction, and this by successive air and steam blasts in the opposite direction through the plant. A. B. MANNING.

Separation from gas mixtures of substances which have a high vapour pressure at their m.p. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 361,914, 20.8.30).—The gas mixture is treated with a solvent capable of dissolving the substances of high v.p., at a temp. below their triple points and, if desired, under pressure, and the solution is then slowly heated, preferably in stages, the evolved gases being collected separately. The method may be applied, *e.g.*, to the separation of  $C_2H_2$  from gas mixtures by treating them with COMe<sub>2</sub> below --81.5°. A. B. MANNING.

Apparatus for treating [removing moisture and recovering gasoline from] gas. K. M. URQUHART (U.S.P. 1,808,087, 2.6.31. Appl., 29.1.29).—The gas is passed up through a tower countercurrent to a stream of gasoline cooled sufficiently to condense most of the moisture in the gas and to remove the higher-boiling constituents of the gasoline in the crude gas. The upper part of the tower, above the gasoline inlet, is occupied by bubble plates and above these is a reflux condenser cooled by a refrigerant; in this the last low-boiling fractions of gasoline are condensed. The gases leaving the apparatus are passed through heat exchangers wherein the gasoline used for washing is precooled.

A. B. MANNING.

Gas-purification process. F. W. Sperr, JUN., Assr. to Koppers Co. (U.S.P. 1,806,370, 19.5.31. Appl., 30.7.26).

—The gas is first washed with an alkaline solution  $(Na_2CO_3)$  containing free S, or FeS, to remove HCN as NaCNS; in the presence of FeS the S is derived from the H<sub>2</sub>S of the gas. The gas is then purified from H<sub>2</sub>S etc. by washing with an alkaline solution containing 0.01-0.1% Ni. The preliminary removal of HCN effects an economy in the consumption of Ni in the second stage. Provision is made for reactivating both solutions. A. B. MANNING.

Extraction of hydrogen sulphide from gases. GES. F. KOHLENTECHNIK M.B.H. (B.P. 362,669, 10.12.30. Ger., 20.12.29).—The gas is washed with a suspension of  $Fe(OH)_3$  in an alkaline solution, and the FeS is regenerated by oxidation with air, the separation of S being prevented by the addition of SO<sub>2</sub> or sulphites to the solution. A portion of the solution is withdrawn from circulation at intervals and the thiosulphate therein is converted into sulphate and S, or other products, in known manner. A. B. MANNING.

Desulphurisation plant for use in manufacture of gas. M. and M. KLÖNNE (A. KLÖNNE) (B.P. 362,805, 19.3.31. Ger., 26.5.30).—Each purifying box of a dry gas purifier is provided at all four corners with a gas distributor, so that the stream of gas is led simultaneously horizontally and vertically diagonally through the box. A. B. MANNING.

Storage of acetylene. E. G. LUENING, ASST. to AIR REDUCTION Co., INC. (U.S.P. 1,808,900, 9.6.31. Appl., 12.5.27).— $C_2H_2$  is stored in a vessel containing a filler of suitable porosity and having a partition of solid porous material of greater density interposed between the filler and the mouth of the vessel. The partition is of such a nature and dimensions that the gas when freed will flow through it at the required max. rate, but that if the gas is dissolved in a liquid, *e.g.*, COMe<sub>2</sub>, the liquid will be retained in the main body of the highly porous material. A. B. MANNING.

Manufacture of emulsions of tars and the like. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 362,467, 5.7.30).—The tar is heated to  $100-200^{\circ}$ , mixed with finely-divided brown coal (H<sub>2</sub>O < 3%), and the mixture emulsified with H<sub>2</sub>O. A. B. MANNING.

Manufacture of aqueous dispersions of bituminous materials. COLAS PRODUCTS, LTD., A. G. TERRY, L. G. GABRIEL, and J. F. T. BLOTT (B.P. 362,577, 2.10.30).—A fine dispersion of the bitumen is first prepared and in this a further quantity of the material is more coarsely dispersed. Conc. dispersions are obtained which are sufficiently fluid when cold to flow from a 2-in. diam. bunghole. A. B. MANNING.

Manufacture of impregnating and adhesive masses. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 362,034, 15.9.30).—Asphaltic materials, e.g., petroleum pitch or "topped" coal tar, are heated with vegetable or animal fatty materials, e.g., rapeseed, sesamé, or train oil, preferably with the addition of a resin, in the presence of  $O_2$ , preferably above  $100^\circ$ , under conditions preventing the loss of org. materials, e.g., with reflux. An oxidation catalyst, e.g., Ni or Mn resinate or naphthenate, may be added if desired. The material is suitable for making sacks from paper and jute.

A. B. MANNING.

Dressing of coal and the like. HUMBOLDT-DEUTZ-MOTOREN A.-G. (B.P. 364,426, 13.3.31. Ger., 20.3.30).

Burners suitable for pulverised fuel. W. A. WHITE, and WHITE'S MARINE ENGINEERING Co., LTD. (B.P. 363,396, 20.11. and 11.9.30).

Apparatus for dry extinction of coke. M. BERTRAND (B.P. 363,366, 19.9.30. Belg., 16.8.30).

Gas burners. Gas Light & Coke Co., J. G. Clark, and A. Masterman (B.P. 364,063, 30.9.30).

Emulsions. Bubble-tower cap. Separation of mixed gases. Gas purifier. Removing  $C_{10}H_8$  etc. from gas. Fluid separators.—See I. Polymerisation of olefines. Wetting etc. agents.—See III.  $H_2SO_4$  recovery from oil treatment.—See VII. Apparatus for hydrogenations. Paraffin hydrocarbons for heat-treating metals. Colloidal Pb. Shingles. Ore coke.—See X. Treatment of emulsions.—See XI. Insecticides.—See XVI.

# III.--ORGANIC INTERMEDIATES.

New wetting agents—sulphonated abietenes. I. GUBELMANN, H. J. WEILAND, and C. O. HENKE (Ind. Eng. Chem., 1931, 23, 1462—1463).—Abietene, b.p.  $340-350^{\circ}$ , obtained by thermal decomp. of wood rosin, gives on sulphonation with 100% H<sub>2</sub>SO<sub>4</sub> at 10° abietenesulphonic acid. The Na salt is a wetting agent suitable for neutral, alkaline, or acid solutions, but salted out by conc. acids or salts. A homogeneous mixture of 50 pts. of the Na salt, 15 pts. of H<sub>2</sub>O, and 35 pts. of terpineol, which is fluid above 30—35°, is a very effective dyeing assistant. C. HOLLINS.

Hydrogenation of naphthalene, anthracene, and phenanthrene. H. WINTER and G. FREE (Brennstoff-Chem., 1931, 12, 451-453).-The hydrogenations were carried out in a gas-heated steel autoclave (capacity 1.85 litres) at a max. temp. of 455-485° (355° for anthracene), and a max. pressure of 200-250 atm. Some of the C<sub>10</sub>H<sub>8</sub> hydrogenations were carried out in the presence of AlCl<sub>3</sub> or Fe, otherwise no catalyst was used. Each of the products contained more H than the initial material and was principally the tetrahydro-derivative. Some decomp. to lower-boiling hydrocarbons, including aromatic hydrocarbons and CH4 etc., occurred. C10H8 and anthracene, but not phenanthrene, also gave highboiling pitch-like decomp. products. In an asbestosjacketed autoclave, anthracene and, in the presence of AlCl<sub>3</sub>, C<sub>10</sub>H<sub>8</sub> also were completely decomposed to gas and coke. A. B. MANNING.

Methylcyclopentane and *n*-nonane from petroleum. Phenols from tar.—See II.

See also A., Jan., 50, Prep. of *m*-nitroaniline. 51, Prep. of Me derivatives of  $\alpha$ - and  $\beta$ -naphthylamines. 60, Electrochemical oxidation of *p*-toluoquinone. Dinitroanthraquinones. 64, Formation of thionaphthindoles. 68, *S*-Substituted dithiourethanes. 92, Enzymic production of esters.

# PATENTS.

Separation of acetic anhydride, acetic acid, and water. C. F. BOEHRINGER & SÖHNE G.M.B.H. (B.P. 358,831, 19.1.31. Ger., 18.1.30).—The mixture is cooled

to  $-20^{\circ}$  to  $-40^{\circ}$  and solid AcOH and H<sub>2</sub>O are removed, leaving Ac<sub>2</sub>O and AcOH of concn. suitable for acetylations. C. HOLLINS.

Packaging of ether. E. MALLINCKRODT, JUN., H. V. FARR, and L. P. HALL, Assrs. to MALLINCKRODT CHEM. WORKS (U.S.P. 1,807,598, 2.6.31. Appl., 20.12.29).— An Al or other metallic container the interior of which is coated with  $Al_2O_3$  is suitable (cf. B., 1929, 453).

E. H. SHARPLES.

Production of ketones [acetone]. DEUTS. GOLD-U. SILBER-SCHEIDEANSTALT, VORM. ROESSLER, Assees. of HOLZVERKOHLUNGS-IND. A.-G. (B.P. 359,430, 14.7.30. Ger., 13.7.29).—Pr<sup>g</sup>OH and H<sub>2</sub>O are led over heavymetal oxides (NiO on oxidised Fe sponge) at 420—450°. Any EtOH present is also converted into COMe<sub>2</sub>. C. HOLLINS.

Production of higher aliphatic alcohols [from esters]. H. T. BÖHME A.-G. (B.P. 358,869, 20.5.31. Ger., 18.7.30).—Carboxylic acids above  $C_5$  are smoothly hydrogenated to alcohols, *e.g.*, in presence of reduced Cu on kieselguhr, if free from "ageing products," *e.g.*, if freshly prepared by hydrolysis of fats or oils.

C. HOLLINS.

Polymerisation of unsaturated hydrocarbons, in particular of the olefine series. H. D. ELKINGTON. From N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 358,068, 29.4.30 and 28.1.31).—Catalysts are prepared by treating halides of Al, Fe, Sn, Sb, Bi, As, Mo, W, V, Ti, Th, or Zr with org. or inorg. polar compounds (which may be solvents for the halides), *e.g.*, PhNO<sub>2</sub>, MeNO<sub>2</sub>, COMe<sub>2</sub>, COPhMe, COPh<sub>2</sub>, BzCl, Ph<sub>2</sub>SO<sub>2</sub>, AgCl, CuCl<sub>2</sub>, NaCl, CaCl<sub>2</sub>, BaCl<sub>2</sub>, NH<sub>4</sub>Cl, or mixtures of these, so as to form additive complexes ; compounds of the type  $C_6H_6$ , AlCl<sub>3</sub> are excluded. C. HOLLINS.

Manufacture of polymerisation products [from acrylic derivatives]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 358,534, 6.5. and 13.11.30).-Saponifiable, H<sub>2</sub>O-insol. derivatives of acrylic or  $\alpha$ -methylacrylic acid are emulsified, preferably in presence of emulsifying and/or wetting agents, before polymerisation, which may be effected with the aid of polymerising agents and irradiation and in presence of org. filmforming substances with or without plasticisers. The products may be converted into rubber-like threads, films, etc. Me acrylate is emulsified in H<sub>2</sub>O with Marseilles soap and Na iso-propyl- or -butyl-naphthalenesulphonate and polymerised with  $H_2O_2$  at 70–90°, unchanged ester is removed, and the polymeride is pptd. with HCl. Other starting materials are Et acrylate (alone or with styrene, cellulose nitrate, acrylic acid, Me acrylate, or acrylonitrile), Bua a-methylacrylate, acrylonitrile (alone or with Me acrylate, vinyl acetate, styrene, or a mixture of octadecyl alcohol, Ac<sub>2</sub>O, and acrylic acid). C. HOLLINS.

Reduction of organic compounds. H. T. BÖHME A.-G. (B.P. 358,721 and 359,188, 8.10.30. Ger., [A] 27.11.29, [B] 9.12.29. Addns. to B.P. 346,237; B., 1931, 834).—(A) In the reduction of such compounds as pyridine, ketones, lactones, aromatic compounds, etc. by Na and alcohols, the amount of Na may be theoretical or less if the reaction is performed under H<sub>2</sub> pressure, *i.e.*, in a closed vessel. (B) Excess pressure

of inert gas  $(N_2)$  is used in place of the H<sub>2</sub> pressure. A 50% yield of octadecylene glycol is obtained from castor oil with Na and BuOH at a pressure of 50 atm. of N<sub>2</sub>. C. HOLLINS.

[Manufacture of] wetting, penetrating, foaming, and dispersing agents. H. T. BÖHME A.-G. (B.P. 358,535, 20.5.30. Ger., 10.7.29).—A sulphonated dior poly-hydric aliphatic alcohol above  $C_{10}$ , e.g., octadecylenic alcohol, is added to liquid or paste preparations (cf. B.P. 308,824 and 317,039; B., 1930, 809; 1931, 12) to improve dispersion etc. C. HOLLINS.

Manufacture of wetting, cleansing, and dispersing agents. J. Y. JOHNSON. From I. G. FARB-ENIND. A.-G. (B.P. 358,583, 11.7.30).—Sulphonic acids and/or sulphuric esters, stable to boiling dil. acids and not pptd. by hard water, are obtained by treating  $\Delta^{a}$ -olefines of C<sub>10</sub>—C<sub>20</sub> with 1 mol. of CISO<sub>3</sub>H, oleum, or mixtures of H<sub>2</sub>SO<sub>4</sub> with Ac<sub>2</sub>O, AcCl, (PrCO)<sub>2</sub>O, P<sub>2</sub>O<sub>5</sub>, PCl<sub>3</sub>, B<sub>2</sub>O<sub>3</sub>, etc., or SO<sub>3</sub> in C<sub>2</sub>HCl<sub>3</sub>, EtOAc, Et<sub>2</sub>O, etc. Suitable starting materials are octadecylene, olefines from alcohols obtained by reducing coconut fat, octadecadiene. C. HOLLINS.

Manufacture of organic nitrogen compounds [wetting, cleansing, tanning, and dispersing agents]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 359,001, 7.7.30).—Aliphatic halides above C<sub>9</sub> are condensed with free primary or sec. amines or amides, or fatty amines, and the products are treated, if desired, with org. or inorg. non-oxidising acids, *e.g.*, sulphonated. Examples are : paraffin wax, m.p. 46—50°, chlorinated to 25% increase in wt., freed from HCl, heated with  $\beta$ -hydroxyethylamine at 180°, and esterified with CISO<sub>3</sub>H at 50—60° for a wetting agent ; chlorinated brown-coal tar oil (b.p. 200—300°) with NH<sub>2</sub>Bu<sup>a</sup>, for a leather softening agent or dispersing agent for dye printing pastes ; chlorinated paraffin oil (b.p. 150—300°) with *p*-aminophenol for a dispersing agent. C. HOLLINS.

Manufacture of products resembling Turkeyred oils [wetting agents]. F. B. DEHN. From DEUTS. HYDRIERWERKE A.-G. (B.P. 359,116, 26.8.30).—Naphthenic acids or esters (e.g., Bu<sup>a</sup> naphthenate) are reduced, e.g., with Na and Bu<sup>a</sup>OH, or with CO or  $HCO_2H$  at 400° over a hydrogenation catalyst, and the product, preferably freed from esters, is sulphonated, e.g., with CISO<sub>2</sub>H at 40°. C. HOLLINS.

Production of solid salts of the higher molecular alkylsulphuric acids. H. T. BÖHME A.-G. (B.P. 358,612, 18.7.30. Ger., 22.8.29).—An aliphatic unsaturated hydrocarbon or aliphatic alcohol above  $C_9$ , or a derivative which retains a double linking or OH group, is sulphonated and the crude sulphuric esters are neutralised directly by org. or inorg. bases or their weak-acid salts. Octadecyl alcohol is treated in  $C_2HCl_3$  with CISO<sub>3</sub>H at 50—60° and the product is neutralised with gaseous NH<sub>3</sub>; ricinoleic acid is treated in Et<sub>2</sub>O with CISO<sub>3</sub>H at —10° to —5°, HCl is removed by a stream of CO<sub>2</sub>, and Na<sub>2</sub>CO<sub>3</sub> is added. Snow-white salts are obtained by recrystallisation from abs. EtOH.

C. HOLLINS.

Manufacture of condensation products of formaldehyde with aromatic sulphonamides and N-aryl derivatives thereof, and their application [as plasticisers and for varnishes]. MONSANTO CHEM. WORKS, Assees. of A. L. RISPLER, M. LUTHY, and F. E. SCHILLING (B.P. 349,956, 31.12.29. U.S., 31.12.28).— Arylsulphonamides or their N-aryl derivatives are heated with CH<sub>2</sub>O in presence of an acid condensing agent (H<sub>2</sub>SO<sub>4</sub>, ZnCl<sub>2</sub>) at 100—150°, or at 60—120° if a solvent (AcOH) is used. Cryst. products are thus obtained from benzenesulphonamide (m.p. 215—220°), p-toluenesulphonamide (m.p. 160—170°), mixed o- and p-toluenesulphonamides (m.p. 135—140°).

C. HOLLINS.

Inhibiting the development of micro-organisms. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 358,279, 7.10.30). — Cyanophenols, except bis(hydroxycyanophenyl) sulphides, *e.g.*, *p*-cyanophenol, 2-chloro-4-cyanophenol, 5-cyanosalicylic acid, m.p. 216—217°, are added (0.025-0.1%) to glue, gelatin, textile finishing materials, etc., to prevent attack by micro-organisms. C. HOLLINS.

Manufacture of hydroxynitriles. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 359,262, 26.11.30). -Cyanohydrins of aliphatic or aromatic aldehydes or ketones or of alkylene oxides are prepared in presence of an org. N base (0.1-1.0%) of the HCN) instead of alkali, whereby resinification is avoided. HCN gas, e.g., passed into a mixture of MeCHO and pyridine gives a quant. yield of the cyanohydrin, b.p. 75-80°/12 mm.; or MeCHO and o-toluidine are added to a mixture of 60% H<sub>2</sub>SO<sub>4</sub> and NaCN in dioxan. The cyanohydrin from aq. COMe2, liquid HCN, Bu<sup>e</sup>OH, and pyridine is converted by HCl gas into Bu<sup>8</sup> a-hydroxyisobutyrate, b.p. 52-60°/14 mm. The prep. of cyanohydrins from CH<sub>2</sub>O, PhCHO, epichlorohydrin (b.p. 128-131°/8mm.), and ethylene oxide (b.p. 106-108°/18 mm.) is described. C. HOLLINS.

Manufacture of phenol or its homologues [from chlorobenzene etc. and steam]. DR. F. RASCHIG GES.M.B.H. (B.P. 358,903, 8.7.30. Ger., 8.7.29. Addn. to B.P. 354,948; B., 1932, 13).—In the process of the prior patent there is used as catalyst, in place of Al<sub>2</sub>O<sub>3</sub> or Al(OH)<sub>3</sub>, another compound of a metal of groups I— IV giving a basic oxide, free from Fe and other impurities. Cu, Co, Ni, Ag, Au, or a Pt metal, or compounds of these, may be added to Al<sub>2</sub>O<sub>3</sub> or Al(OH)<sub>3</sub> or to the new catalysts. Examples are : Sn(OH)<sub>4</sub> or Ti(OH)<sub>4</sub> at 450°, ZnO at 420°, CaCO<sub>3</sub> with 2% Cu at 350°, Na<sub>2</sub>CO<sub>3</sub> with 2% Ni, AlPO<sub>4</sub> at 480°, and AlCl<sub>3</sub> at 420°.

C. HOLLINS.

Manufacture of aryl[am]ides of unsymmetrical o-[4]-xylenol-[5]carboxylic acid. [Coupling components for ice colours.] W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 358,477, 30.6.30).—The PCl<sub>3</sub>-solvent method is used for the prep. (m.p. in parentheses) of o-4-xylenol-5-carboxylic anilide (178°), m-hydroxyanilide (218—219°), 7- and 8-hydroxy- $\beta$ naphthylamides (194—195°, 185°), tolidide (296—297°),  $\alpha$ - and  $\beta$ -naphthylamides (199—200°, 214—215°), o-, m-, and p-toluidides (140°, 202—203°, 194—195°), m-4xylidide (175—176°), o-, m-, and p-chloroanilides (189— 190°, 198—200°, 209—210°), o- and p-anisidides (159— 160°, 177—178°), m- and p-nitroanilides (245—246°, 267—269°), 4- and 5-chloro-o-toluidides (192—193°,

207—208°), 4- and 5-chloro-o-anisidides (220°, 170°), 2:5-dimethoxyanilide (120—121°), 4-chloro-2:5-dimethoxyanilide (180—181°), 4- and 5-nitro-o-anisidides (266—268°, 165—167°), 4- and 5-nitro-o-toluidides (191—192°, 251—252°), 2-nitro-p-toluidide (230—231°), the 2:5-dichloro-, 2-chloro-5-methoxy-, and 2:5-dimethoxy-p-phenylenediamides (250°, 310—315°, 308— 310°), and 4-chloro-2:5-tolylenediamide (288—290°).

C. HOLLINS.

Manufacture of aromatic [imino-]compounds containing nitrogen. I. G. FARBENIND. A.-G. (B.P. 359,201, 17.10.30. Ger., 17.10.29).—Malononitrile, or one of its homologues, is condensed with aromatic hydrocarbons or derivatives in presence of AlCl<sub>3</sub> etc. to give diketimines. The nitrile may be produced *in situ*, *e.g.*, from cyanoacetamide or malonamide. Acenaphthene yields *peri*acenaphthindandione. Corresponding *peri*indandiones are obtained from  $C_{10}H_8$ , 1-methylnaphthalene,  $\alpha$ - and  $\beta$ -naphthyl Me ethers with malononitrile and from  $C_{14}H_{10}$  and methylmalononitrile. PhMe and malononitrile gives a *compound*, m.p. 109°.

C. HOLLINS.

Manufacture of derivatives of 3-nitro-4-hydroxybenzamide [arylene-iminazoles, -thiazoles, -oxazoles, and -perimidines]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 359,063, 22.7.30. Addn. to B.P. 341,970; B., 1931, 434) .- A 3-nitro-4-acyloxybenzoyl halide is condensed with o- or peri-diamines, o-aminophenols, or o-aminothiophenols to give cyclic compounds having a µ-m-nitro-p-hydroxyphenyl group; the products may be reduced to amines. Examples of second components are: o-phenylenediamine (for a benziminazole, m.p. 186°), 4-nitro-o-phenylenediamine, 1:8-naphthylenediamine-3:6-disulphonic acid, 1:2naphthylenediamine-5-sulphonic acid, o-aminophenol (for 3-nitro-4: 2'-dihydroxybenzanilide, m.p. 219°, which is cyclised by ZnCl<sub>2</sub> in AcOH to a benzoxazole, m.p. 220-221°), and 4-chloro-o-aminothiophenol (for a benzthiazole, m.p. 201°). C. HOLLINS.

Production of chlorinated diaryls. FEDERAL PHOSPHORUS CO., Assees. of R. L. JENKINS (B.P. 358,703, 24.9.30. U.S., 28.9.29).—Ph<sub>2</sub> (etc.) is treated with  $Cl_2$ in presence of Fe. Preferably  $Cl_2$  enters the base of an Fe-packed tower filled with molten Ph<sub>2</sub>, cooled to 80—66° for monochlorination or allowed to reach 175—220° for polychlorination. C. HOLLINS.

**Production of diarylguanidines.** SILESIA VER. CHEM. FABR. (B.P. 359,318, 7.1.31. Ger., 27.11.30. Addn. to B.P. 258,203; B., 1926, 995).—The PbO of the prior patent is replaced by Pb salts, e.g., PbCl<sub>2</sub> or PbSO<sub>4</sub>; the PbS produced is reconverted into PbCl<sub>2</sub> or PbSO<sub>4</sub> by HCl or HNO<sub>3</sub>. C. HOLLINS.

Preparation of 1-aminoanthraquinone-2-sulphonic acid. CHEM. FABR. VORM. SANDOZ (B.P. 358,852, 9.3.31. Ger., 11.3.30).—1-Aminoanthraquinone is heated at  $210-240^{\circ}$  with at least 1 mol. of an alkali hydrogen sulphate (KHSO<sub>4</sub>), water vapour being continuously removed in vac. or by a current of gas. The yield is  $90-95^{\circ}_{0}$ . C. HOLLINS.

Vat-dye intermediates. Anthraquinone intermediates. Benzanthrone intermediates.—See IV. Barbet test.—See XVIII.

# IV.—DYESTUFFS.

Anthraquinoneazo compounds. III. Substantive disazo dyes from 1 :5-diaminoanthraquinone. V. Light absorbtion of anthraquinoneazo-Naphthol AS dyes. T. MAKI (J. Soc. Chem. Ind., Japan, 1931, 34, 392—397 B, 496—498 B).—III. Tetrazotised 1 : 5diaminoanthraquinone is coupled with 2 mols. of H-acid (dark blue on cotton direct; coppered, violet, with better fastness to washing; tetrazotised on the fibre and developed with resorcinol, dark green, or  $\beta$ -naphthol, violet-brown),  $\gamma$ -acid (dark brown on cotton; coppered, grey; developed with resorcinol, yellow-brown, or  $\beta$ -naphthol, violet-black), naphthionic acid (dark red on cotton, loose to washing and acids). Absorption spectra are described.

V. The absorption spectra of these pigments in  $C_2H_2Cl_4$  show max. vals. of about 50 Å. The bathochromic effect of either component is exactly parallel with the effect on m.p. The amount of absorption is practically identical for the three  $\alpha$ -compounds, but for the  $\beta$ -compounds the order is AS > BS > BO, although more intense shades on cotton are obtained with BO and BS than with AS. The spectra are very similar for all 6 pigments, but the  $\beta$ -compounds show more sharply selective absorption. C. HOLLINS.

Anthraquinoneazo compounds. IV. Naphthol AS dyes from 1- and 2-aminoanthraquinones. T. MAKI (J. Soc. Chem. Ind., Japan, 1931, 34, 427—432 B). —New scarlet to red *dyes*, prepared by coupling diazotised 2-aminoanthraquinone with 2:3-hydroxynaphthanilide (I), m.p. 280.6°, 2:3-hydroxynaphtho-*m*-nitroanilide (II), m.p. 315.9°, and 2:3-hydroxynaphtho-*a*naphthalide (III), m.p. 306.2°, and diazotised 1-aminoanthraquinone with (I), m.p. 300.5°, (II), m.p. 319.6°, and (III), m.p. 315.5°, are described. All m.p. are corr. H. A. PIGGOTT.

See also A., Jan., 57, Prep. of Me derivatives of  $\alpha$ - and  $\beta$ -naphthylamines. 60, Dinitroanthraquinones. 66, Dyes from pyrazine-2:3-dicarboxylic acid. 67, Dyes from phenanthraquinone and from acenaphthenequinones. Thiazoles.

#### PATENTS.

Manufacture of sulphur dyes. I. G. FARBENIND. A.-G. (B.P. 359,254, 359,273, and 359,276, [A] 19.11.30. [B] 3.12.30, [c] 4.12.30. Ger., [A] 19.11.29, [B, C] 4.12.29). -Sulphurisation of azines is effected in presence of (A) Mo compounds in addition to the usual metal compounds, or (c) W, V, U, Sb, or P compounds with or without the usual metal compounds. (B) The shade of other S dyes is similarly modified. Examples are S dyes from (A) 3-amino-7-hydroxyphenazine or its 2-Me-5-Et derivative with molybdic acid and CuSO<sub>4</sub> (red); (B) 1-amino-4-phydroxyanilinonaphthalene or 4-p-hydroxyanilino-1-toluidinonaphthalene-8-sulphonic acid with NH4 molybdate (bright blue-green); crude cresol with NH4 molybdate (bright brown); (c) 3-amino-7-hydroxy-2-methyl-5ethylphenazine with Na tungstate, or Na tungstate and CuSO<sub>4</sub>, or uranyl nitrate (bright blue-red), or with Na<sub>2</sub>HPO<sub>4</sub> or tartar emetic (bright red). C. HOLLINS.

Manufacture of acid wool dyes of the anthraquinone series. I. G. FARBENIND, A.-G. (B.P. 359,348, 20.2.31. Ger., 25.2.30).—Quinizarin-6-sulphonic acid is condensed, preferably in presence of org. solvent (AcOH),  $H_3BO_3$ , and a reducing agent (Zn powder, SnCl<sub>2</sub>), with *p*-aminoformanilide, *p*-aminoacetanilide, or derivatives alkylated in the amide group. *p*-Aminoacetanilide gives a level yellowish-green wool dye. C. HOLLINS.

Manufacture of condensation products and pigment dyes of the anthraquinone series. A. CARP-MAEL. From I. G. FARBENIND. A.-G. (B.P. 359,051, 19.7.30).—A 2-halogenoquinizarin is condensed with a phenol, preferably in presence of a Cu catalyst; the products may be sulphonated and converted into insol. salts (lakes). The product from *p*-cresol, sulphonated with 5% oleum at 30°, gives a red Al lake. Al lakes are prepared from the *p*-chlorophenol (blue-red), *p*-hydroxybenzoic acid (red), and phenol (blue-red) compounds. C. HOLLINS.

Manufacture of nitrogenous vat dyes [of the anthraquinone series]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 358,074, 31.5.30 and 21.1.31).— A 1-(3'-benzanthronylamino)anthraquinone, carrying in 4-, 5-, or 8-position a substituted NH<sub>2</sub> group, is treated with alkaline condensing agents (alcoholic KOH), whereby cyclisation by union between positions 2 and 4' occurs. Products in which the substituted NH<sub>2</sub> group survives may be further treated with acid condensing agents (H<sub>2</sub>SO<sub>4</sub>, ClSO<sub>3</sub>H, NaAlCl<sub>4</sub>). 1-(3'-Benzanthronylamino)-5-butylaminoanthraquinone gives by alkaline fusion a grey-blue vat dye (annexed formula), the levelling power



of which is improved by  $H_2SO_4$ treatment. Similar products are obtained from the 5- $\beta$ -hydroxyethylamino- (bluegrey), 4-cyclo-

hexylamino- (olive-grey), 5-cyclohexylamino- (blue-grey), 8-cyclohexylamino- (green-grey), 5-benzylamino- (grey), 4-anilino- (olive-grey; with NaAlCl<sub>4</sub> brown), 5-ptoluidino- (grey-blue), 5- $\alpha$ -anthraquinonylamino- (grey; with NaAlCl<sub>4</sub>, olive), 5- $\beta$ -anthraquinonylamino- (redgrey; with NaAlCl<sub>4</sub>, bluer grey), 5-(5'-hydroxy- $\alpha$ -anthraquinonylamino)- (reddish grey-black), and 5-dimethylamino- (grey-blue) compounds. C. Hollins.

[Manufacture of] colouring matters and intermediates of the benzanthrone series. IMPERIAL CHEM. INDUSTRIES, LTD., D. C. R. JONES, R. F. THOMSON, and J. THOMAS (B.P. 358,420, 1.4.30).—Condensation products from benzanthrones and CH<sub>2</sub>O, *e.g.*, dibenzanthronylmethane, are treated with org. acid halides or anhydrides, *e.g.*, BzCl, AcCl, PhSO<sub>2</sub>Cl, in presence of AlCl<sub>3</sub> etc., with or without oxidants (*e.g.*, O<sub>2</sub>). The products may subsequently be oxidised with NaOCl and alkylated. The BzCl product is a yellow vat dye and gives bright yellow shades on wool from faintly acid suspension. C. HOLLINS.

Production of vat dyes and intermediates [of the benzanthrone series]. R. F. THOMSON, P. G. CARTER, J. THOMAS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 358,426, 4.6.30 and 27.2.31).—Benzanthrone is condensed with an o-halogenobenzoic acid in presence of an

alkaline agent (KOH), e.g., at  $180-200^{\circ}$ , and the product is cyclised, e.g., with  $H_2SO_4$  or with  $P_2O_5$  in PhNO<sub>2</sub>, to give a yellow-orange vat dye. C. HOLLINS.

**Production and use of vat dyes and intermediates.** J. THOMAS, R. F. THOMSON, W. SMITH, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 358,078, 4.6.30).—PhCl is condensed with a chlorinated phthalic anhydride, and the product is mononitrated, reduced, and cyclised; the resulting isomeric chlorinated α- and β-aminoanthraquinones are separated by dilution of the H<sub>2</sub>SO<sub>4</sub> solution. The α-compound may be condensed directly to an indanthrone and the β-compounds after monohalogenation. 4:5-Dichlorophthalic anhydride thus gives 3:6:7-trichloro-2-amino- and 2:6:7-trichloro-1-aminoanthraquinones, from which are obtained 3:3':6:6':7:7'-hexachloro- and 6:6':7:7'-tetrachloro-indanthrones. C. HOLLINS.

[Manufacture of] colouring matters and intermediates of the anthraquinone series. IMPERIAL CHEM. INDUSTRIES, LTD., W. SMITH, J. PRIMROSE, and J. THOMAS (B.P. 358,421, 1.4.30).-A 1:2-, 2:1-, or 2:3-halogenoanthraquinonecarboxylic acid is condensed with a 2-(3'-aminobenzoyl)benzoic acid carrying in 4'-position an NH<sub>2</sub>, substituted NH<sub>2</sub>, OH, ether, SH, or thioether group, and the product is cyclised, e.g., with H<sub>2</sub>SO<sub>4</sub>. The condensation may be effected in an alkaline medium in presence of acid absorber and Cu catalyst, or in pyridine and PhNO2 with or without Cu catalyst; or the acids may be used in the form of esters in PhNO<sub>2</sub>. 2-(4'-Chloro-3'-nitrobenzoyl)benzoic acid is converted by NaOPh into the 4'-phenoxy-compound, which is reduced and heated with 1-chloroanthraquinone-2-carboxylic acid, Na2CO3, and CuSO4, and the product is cyclised to a red-brown vat dye. Dyes are similarly prepared from the 4'-anilino- (green-blue), 4'-methylamino- (blue), 4'-hydroxy- (red), 4'-thiol- (blue), 4'-a-anthraquinonylamino- (dark green), and 4'-benzamido- (blue-grey) compounds. The 4'-methylamino-, 4'-benzamido-, and 4'-anilino-derivatives of 2-(3'-aminobenzoyl)benzoic acid give with 2:3-chloroanthraquinonecarboxylic acid after cyclisation blue-green, yellow-green, and green-grey vat dyes, respectively. C. HOLLINS.

Manufacture of substitution products [vat dyes] of the dibenzpyrenequinone series. I. G. FARBENIND. A.-G. (B,P. 358,502, 4.7.30. Ger., 6.7.29. Addn. to B.P. 324,964; B., 1930, 411).—The NH<sub>2</sub> group or groups in amino-1:2:7:8-dibenzpyrene-3:6-quinones are replaced by other groups by diazo reactions. The diamine of B.P. 307,926 (B., 1930, 810) gives a dihydroxy-compound, which is methylated for a brownish-red vat dye. The monoamine of B.P. 333,882 (B., 1930, 1059) gives a chloro- or a bromo-compound (blue-red), or a cyanocompound (red-orange). C. Hollins.

Manufacture of derivatives [vat dyes] of the dibenzpyrenequinone series. I. G. FARBENIND, A.-G. (B.P. 358,509, 4.7.30. Ger., 6.7.29. Addn. to B.P. 325,222; B., 1930, 453).—Polyamino-1:2:6:7-dibenzpyrene-3:8-quinones or mono- or poly-amino-1:2:7:8dibenzpyrene-3:6-quinones are condensed with suitable negatively substituted org. compounds. Examples are : BzCl and the diamino-1:2:6:7-dibenzpyrene-3:8quinone of B.P. 307,926 (B., 1930, 810) for a yellow-red

vat dye, or the amino-1:2:7:8-dibenzpyrene-3:6quinone of B.P. 333,882 (B., 1930, 1059) for a violet; anthraquinone-2-carboxyl chloride and the diamino-1:2:6:7-dibenzpyrene-3:8-quinone for a brown-red. C. HOLLINS.

Manufacture of azo dyes [for wool]. A .CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 358,055, 2.7.30).-An o-aminodiarylsulphone, carrying a sulphonamido- or sulphonalkylamino-group, is diazotised and coupled in acid medium with a B-naphthylamine- or a 2:8-aminonaphtholsulphonic acid or an N-derivative thereof. Examples are : 2-amino-4'-hydroxy-3'-carboxydiphenylsulphone-4-sulphonmethylamide  $\rightarrow \gamma$ -acid (red); 2-amino - 4' - methyldiphenylsulphone - 4 - sulphonmethylamide  $\rightarrow$  methyl- $\beta$ -naphthylamine-7-sulphonic acid (red); 2-amino-4'-hydroxy-3'-carboxydiphenylsulphone-4-sulphoamide  $\rightarrow \gamma$ -acid (red). [Stat. ref.]

C. HOLLINS.

Manufacture of azo dyes [for wool]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 358,136, 8.7.30).-A non-sulphonated and non-carboxylated o-aminobenzenesulphonarylamide, carrying in the sulphonamide group a hydroxy- or sulphato-alkyl group as N-substituent, is diazotised and coupled in acid medium with a B-naphthylamine- or 2: 8-aminonaphthol-sulphonic acid or an N-derivative thereof. Examples are: 4-nitroaniline-2-sulphon-\$-hydroxyethylanilide, m.p. 127- $128^\circ, \rightarrow \gamma\text{-acid}$  [blue; reduced (bordeaux-red) and acetylated (blue-red)]; corresponding sulphato-compound  $\rightarrow$  methyl- $\beta$ -naphthylamine-7-sulphonic acid (redblue), or  $\rightarrow \gamma$ -acid (navy-blue; reduced, blue-red).

C. HOLLINS.

Manufacture of azo dyes [pigments and ice colours]. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 357,959, 21.6.30).-A 3-hydroxydiphenylamine-4-carboxylic arylamide, which may carry alkyl, alkoxyl, aryloxy-, or halogen substituents in positions 2'-6', is coupled in substance or on the fibre with a suitable diazo or tetrazo compound. Amongst the examples are: 3-hydroxydiphenylamine-4-carboxylic anilide with diazotised 3:5-dichloroaniline (yellowbrown), B-naphthylamide with diazotised m-chloroaniline (dark brown), and dianisidide with diazotised 5-nitro-o-toluidide (dark brown); 4'-chloro-3-hydroxydiphenylamine-4-carboxylic o-anisidide with diazotised 2:5-dichloroaniline (brown), p-nitroanilide with diazotised 4-benzamido-2:5-diethoxyaniline (red-brown) or 4-aminoazobenzene (violet-red), o-anisidide with diazotised 2:5-dichloroaniline (vellow-brown) or 4-aminoazobenzene (red), 2:5-dichloroanilide with diazotised 2:5-dichloroaniline (brown), and m-hydroxyanilide with diazotised 5-chloro-o-toluidine (yellow-brown).

C. HOLLINS.

Manufacture of [acid] monoazo dyes. A. CARP-MAEL. From I. G. FARBENIND. A.-G. (B.P. 358,483, 3.7.30).-An arylaminesulphonic acid having no o-OH group is diazotised and coupled with an *a*-naphthol-3:6- or -3:7-disulphonamide or an 8-halogeno-anaphthol-3: 6-disulphonamide. Examples are: orthanilic acid  $\rightarrow \alpha$ -naphthol-3: 6-disulphonmethylamide (orange); p-toluidine-3-sulphonic acid  $\rightarrow \alpha$ -naphthol-3:6-disulphonanilide, or m-acetamidoanilide (yellowred); or 8-chloro-α-naphthol-3: 6-disulphonanilide,

m.p. 193° (red); *p*-chloroaniline-2-sulphonic acid  $\rightarrow \alpha$  - naphthol - 3 : 6 - disulphon - ( $\beta$  - hydroxyethylanilide) (vellow-red): 2-ethoxy-Cleve acid  $\rightarrow \alpha$ -naphthol-3:6disulphon-(m-acetamidoanilide) (violet); p-aminoacetanilide-3-sulphonic acid  $\rightarrow \alpha$ -naphthol-3:7-disul-C. HOLLINS. phonanilide (blue-red).

Manufacture of disazo dyes [for dyeing and printing on wool]. J. R. GEIGY A.-G. (B.P. 359,096, Ger., 9.8.29).-A diaminoditolylmethane-11.8.30. disulphonic acid is tetrazotised and coupled with 2 mols. of a naphtholmonosulphonic acid or a pyrazolonesulphonic acid. Examples are : disulphonated 3:3'diamino-4: 4'-dimethyldiphenylmethane  $\rightarrow 2$  mols. of N.W. acid (red); disulphonated 4:4'-diamino-2:2'dimethyldiphenylmethane  $\rightarrow 2$  mols. of 1-p-sulphophenyl-3-methyl-5-pyrazolone (yellow). C. HOLLINS.

Wetting etc. agents .- See III.

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

Fibrillar structure of bast fibres. A. HERZOG (Papier-Fabr., 1931, 29, 833-837).-Most bast fibres, ramie, hemp, flax, stinging nettle, etc. show a fibrillar structure, but whereas in raw, unpurified material this is seen only with difficulty, it is readily visible in the purified fibres after a swelling treatment with NaOH, cuprammonium, etc. that also causes dissolution of the interfibrillar incrustants. Sometimes pressure alone is sufficient to cause separation into fibrils, and it is suggested that the washing, bleaching, squeezing, etc. of textile materials may cause weakening of the fibres by producing such separation. Cotton is much less easily disintegrated by mechanical means than is flax, and differences in the properties of materials made from these fibres may be due to differences in the degree of B. P. RIDGE. separation into fibrils.

Degumming of real silk fabrics containing cellulose acetate silk. ANON. (Kunstseide, 1931, 13, 434).-After scouring at 85° for 1-11 hr. in a solution containing Marseilles soap (10%) and soda ash (1%), the real silk was completely degummed, but the cellulose acetate silk was partly hydrolysed and suffered a 20-40% loss in tensile strength. A process satisfactory for both fibres consists in treating the mixed fabric for 0.5 hr. at 75-80° in a solution containing soap (10%) and pine oil (5%), then rinsing, and again treating for 1 hr. at 70-75° in a similar soap-pine oil liquor of A. J. HALL. half that concn.

Chemical researches on cellulose materials. I. Bast fibre of "Kayu Baru." M. SHIKATA and M. WATANABE. II. Bast fibre of "Kayu Selemoh." M. SHIKATA and Y. WATANABE (J. Cellulose Inst., Tokyo, 1931, 7, 261-264, 265-266).-I. An easily bleachable pulp is best obtained by digestion for 3 hr. at 160° with 25% of NaOH on the wt. of dry material. The digestion is easier than for Loblolly pine or fir wood, but more difficult than for flax straw or corn stalk. The fibre of the pulp is pure white, of silky lustre, is about 3 mm. long, and can be used as a substitute for wood pulp. Results of analysis of the digested pulp are given.

II. The fibre of Kavu Selemoh is rough and of a greyish colour. It is easily digested by the NaOH process, but, compared with Kayu Baru, gives a higher yield of difficultly bleachable pulp. The bleached pulp is pure white and can be used for making paper.

B. P. RIDGE.

True sp. gr. of ramie cellulose. U. YOSHIDA and B. TAKEI (J. Cellulose Inst., Tokyo, 1931, 7, 255—260).— Determination of the sp. gr. of samples of ramie after prolonged heating and evacuation of the glass tubes containing them gives higher vals. than several previously recorded. This is considered due to the fact that in the former cases the material contained gas and  $H_2O$  in its inner structure. The time of evacuation of the tubes is shortened by raising the temp., but the cellulose decomposes above 130°. Mean vals. obtained are : retted fibre 1.622, purified ramie 1.614, mercerised 1.610. B. P. RIDGE.

Hollow-filament [cellulose] acetate silk. H. SCHUPP (Kunstseide, 1931, 13, 424-429).-Hollow artificial silk produced by dry spinning at the usual spinning temp. a solution of cellulose acetate containing a suitable amount of a non-solvent for the acetate gives a product  $(d \ 0.8)$  having much greater covering power than ordinary acetate  $(d \ 1 \cdot 3)$ , viscose  $(d \ 1 \cdot 5)$ , or hollow viscose (d 1.37) silk. Although its normal lustre is slightly less than that of ordinary acetate silk, the conditions of spinning may be adjusted so that it is perfectly white and opaque; this latter matt appearance resists scouring, bleaching, and dyeing processes. 100-denier yarn of the new silk has a covering power in textile materials equal to 150-denier yarn of ordinary acetate silk, and is much warmer in handle. The new silk has a tensile strength of 1.2-1.3 g. per denier and suffers a 25-30% loss of strength when wetted; it is usually spun so that each filament is of 2-6 denier, and has been found satisfactory in the manufacture of crêpe and plush materials. A. J. HALL.

Recent advances in the chemistry of cellulose in relation to pulp and paper. C. J. J. Fox (Proc. Tech. Sect. Paper Makers' Assoc., 1931, 12, 123–137).— A lecture, outlining the relation of such advances and the micellar theory of fibre structure to the pulp and paper industry. T. T. Ports.

Separation of fibrous cellulose from plant substances. J. M. ARNOT (Proc. Tech. Sect. Paper Makers' Assoc., 1931, 12, 147—154).—A résumé of recent methods for the digestion of fibrous materials, particularly those involving the use of  $NH_4$  compounds. Pulps of varying bleachability are produced, but cost of reagents and difficulty of recovery make industrial application difficult. The results of trial digestions are given.

T. T. Potts.

Highly purified wood fibres as papermaking material. R. H. RASCH, M. B. SHAW, and G. W. BICKING (Bur. Stand. J. Res., 1931, 7, 765—782).— The mechanical properties, chemical purity, and ageing effect of high-grade bond papers made from highly purified wood fibres have been determined. The stability of resin-sized papers in the accelerated ageing test (heating at 100° for 72 hr.) was seriously impaired by the presence of excess alum, but a stable, well-sized paper was produced by maintaining the  $p_{\rm H}$  at 5.0. Starch did not reduce the stability in the ageing test, and the resistance to ageing was improved by surface-sizing with glue or starch. Moderate amounts of alum, used as preservative in the surface-sizing baths, were not deleterious. E. S. HEDGES.

Sulphur, pyrites, and sulphite pulp. R. E. COOPER (Pulp and Paper Mag., 1931, 31, 1190—1193).— S and pyrites as sources of SO<sub>2</sub> for the manufacture of sulphite pulp are compared. D. K. MOORE.

Hydrolysis of pulp. II, III. M. S. FILOSOFOV (Nauk. Zapiski Tzuk. Prom., 1931, 12, 361-373).— HCl is first removed from the hydrolysate with superheated air ( $71\cdot2\%$  recovery); the aniline acetate, phloroglucinol, and phenylhydrazine methods for determining furfuraldehyde gave 2, 5, and 3%, respectively.  $10\cdot75\%$  of furfuraldehyde can be obtained from dry pulp. CHEMICAL ABSTRACTS.

Volumetric composition of paper. I. Permeability of paper to air. T. T. Porrs (Proc. Tech. Sect. Paper Makers' Assoc., 1931, 12, 91-122).-The factors controlling the rate of flow of air through paper have been investigated. Rate of flow is directly proportional to area of sample and to pressure, but is not inversely proportional to thickness, the relation between rate of flow and thickness being different for each of the samples examined. Poiseuille's formula does not hold for paper as the capillaries are short relative to diam. and are of irregular form and direction. Rate of flow of air through paper is a function of interstitial space, the pore-space of the fibres playing no part therein. Air permeability is measured as the rate of flow of air through a standard area under standard and const. difference of pressure. Examples of the use of the Standard Sheet Machine in investigating the effect of thickness on permeability of sheets made from the T. T. Potts. same pulp are given.

Survey of storage conditions in libraries relative to the preservations of records. A. E. KIMBERLY and J. F. G. HICKS, JUN. (U.S. Bur. Stand., Misc. Publ. No. 128, 1931, 8 pp.).-Daylight, polluted air, variations of temp. and humidity, and pests are all causes of the deterioration of paper. The elimination of dust is very important and the removal of acid ingredients  $(SO_2 \text{ etc.})$  from the atm. by scrubbing with slightly alkaline solutions is advisable. If the paper contains Fe the rate of formation of H<sub>2</sub>SO<sub>4</sub> from the SO<sub>2</sub> is greatly accelerated, and the effect of this acid depends on the moisture content of the surrounding atm. Rosin sizing is one cause of the yellowing of records with age. Ten days' exposure to air polluted with SO<sub>2</sub> (5-10 p.p.m.) may cause losses in physical strength up to 40% and increases in the acidity of the paper of 90-400%. Daylight should be rigidly excluded. The temp. and R.H. of the atm. should be maintained at 18-24° and 45-55%, respectively. B. P. RIDGE.

Electrometric analysis in paper mills. T. T. Ports (Proc. Tech. Sect. Paper Makers' Assoc., 1931, 12, 156—161).—Conductometric titrations are employed in the analysis of spent caustic liquors and in the examination of sizing solutions. Low-tension a.c. is obtained from the mains by means of step-down transformers, output voltage being controlled by a potentiometer. Rectifier-type a.-c. galvanometers are used as

indicators, and where the dilution is sufficiently high sharp end-points are obtained. Complete freedom from polarisation is attained by the use of a tuning-fork oscillator giving 1000 cycles per sec. of almost perfect sine-wave form. Titration curves are given for representative conductometric and potentiometric titrations, the latter obtained with apparatus built from radio components. T. T. Ports.

Determination of free and combined sulphurous acid in bisulphite liquors from sulphite-cellulose manufacture. Use of visual conductometric titration in operation control. G. JANDER and K. F. JAHR (Z. angew. Chem., 1931, 44, 977–980).—The liquor is treated with neutral  $H_2O_2$ , whereby each mol. of Ca(HSO<sub>3</sub>)<sub>2</sub> gives 1 mol. each of CaSO<sub>4</sub> and  $H_2SO_4$ , whilst free  $H_2SO_3$  gives  $H_2SO_4$ . Conductometric titration with standard Ba(OH)<sub>2</sub> gives a curve consisting of 3 straight lines, from the points of intersection of which the total  $SO_3''$ , free  $H_2SO_3$ , and Ca<sup>--</sup> are obtained by a simple calculation. The method is rapid and accurate.

F. L. USHER.

Soda recovery from [pulp] black liquors. E. HÄGGLUND, M. TUOMINEN, and K. LINDBLOM (World's Paper Trade Rev., 1931, 96, 405–406).—The org. matter is pptd. by heating the liquors under pressure after addition of alkali. Rise of temp. from  $310^{\circ}$  to  $350^{\circ}$ increases the speed of decarbonisation about 10 times and increases the pressure from 100 to 200 atm. The yield of tar is max. and that of AcOH improved by addition of 5 g. of NaOH per 100 g. of wood. Scotch pine gave : tar 25 g., MeOH 2.5 g., AcOH 5.6 g., gas 4.5 litres per 100 g. ; aspen wood gave : tar 16.00, MeOH 2.80, COMe<sub>2</sub> 0.45, AcOH 9.25 g., gas 6 litres per 100 g. CHEMICAL ABSTRACTS.

Bleaching wood pulp. Colouring paper.—See VI. Causticising.—See VII. Concn. control.—See XI. Sulphite waste liquor as fungicide.—See XVI.

See also A., Jan., 20, Adsorption of salts by cotton. Morphology of cellulose fibres. 47, Prep. of cryst. cellulose acetate. 48, Conversion of cellulose acetates. 71, Hydrolysis of tussah-silk fibroin.

#### PATENTS.

Manufacture of fur for use in the manufacture of fur pile fabrics. J. MORTON (B.P. 363,731, 1.10.30).

Spinning machines for artificial silk. VEREIN. GLANZSTOFF-FABR. A.-G. (B.P. 363,529 and Addn. B.P. 363,556 and 363,566, [A] 20.1.31, [B] 24.2.31, [C] 10.3.31. Ger., [A] 20.1.30, [B, C] 10.3.30).

Washing and after-treating freshly spun cakes of artificial silk, VEREIN, GLANZSTOFF, FABR. A.-G. (B.P. 364,489, 20.5.31, Ger., 5.6.30).

Manufacture of paper articles such as handkerchiefs, towels, etc. CELLULOSE PRODUCTS, INC., Assees. of K. WANDEL (B.P. 364,115, 26.9.30, U.S., 27.6.30).

Drying of fibres etc. Drying of crops etc.— See I. Adhesive masses [for sacks].—See II. Wetting etc. agents.—See III.

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

Use of liquid chlorine in preparation of bleach liquors. H. R. J. FEENY (Proc. Tech. Sect. Paper Makers' Assoc., 1931, 12, 163—169).—A brief description of the plant, operation, and methods of control necessary for the chlorination of bleaching powder solutions and of milk-of-lime. The Golding-Pritchard plant for rapid chlorination of CaO is illustrated, 200 gals. of aq. Ca(OCl)<sub>2</sub> (3% available Cl) being prepared in 5 min. T. T. Ports.

Step-bleaching [of wood pulp] with elementary chlorine. R. RUNKEL (Papier-Fabr., 1931, 29, 830-833).—A process is described in which, by the addition of a washing drum, a rotary pump, and an injector to the hollander plant, suitable quantities of liquor are continuously withdrawn from the washing drum, controlled amounts of Cl, are injected into them, and they are forced back into the bottom of the hollander. In this way no increase in the quantity of liquor in the latter takes place. The bleaching process, which gives a saving in the cost of Cl, of 18% and of chemicals of about 35%, is carried out according to the scheme chlorination-wash-treatment with alkali-wash-final bleach, and the acidity or alkalinity of the liquor can be satisfactorily controlled. Washing must be thorough and takes a long time unless presses are used for removing excess liquor, but this time is compensated for by the rapidity of the bleaching. B. P. RIDGE.

Treatment [desizing, dyeing, etc.] of [cellulose] acetate silk materials. Anon. (Textilber., 1932, 13, 26-27).-Fabric having a Rhodiaseta acetate silk warp and a wool crêpe weft is desized preparatory to dyeing by entering at 40° into a bath containing 10 g. of soap per litre, the temp. then being maintained for 2 hr. at 70-75°; at higher temp. the wool will felt. Fabric having a Rhodiaseta acetate silk warp and a real silk crêpe weft is desized at 92-95° in a bath containing 20 g. each of soap and anhyd. Na2SO4 per litre. Hyposulphite solutions are usually not satisfactory for stripping dyed cellulose acetate silk since many of the dyes used are derived from anthraquinone. Stripping is better effected with dil. aq. NaOCl, especially if a swelling agent, e.g., HCO2H, is added, but a disadvantage of this method is that the tone of the colour remaining on the stripped silk is usually quite different from that of the original colour. This defect may be avoided by stripping at 85° in a bath containing 2 g. of activated C and 5 g. of soap per litre, to which should be added (if the activated C is coarse) 1-2 g. per litre of a protective colloid, e.g., dextrin, glucose, or gelatin. A. J. HALL.

Dyeing and colouring of paper. F. HAMILTON (Proc. Tech. Sect. Paper Makers' Assoc., 1931, 12, 175—190).—The range of natural and synthetic colouring matters applicable to the colouring of paper is reviewed, details of manipulation of individual classes being given. T. T. Ports.

Wetting agents.—See III. Hollow acetate silk. —See V. Determination of active O [in bleaching cotton].—See VII. Al apparatus.—See X. Ethanolamine soaps.—See XII. Lacquer and undercoatings.—See XIII.

#### PATENTS.

Dyeing [of cellulose esters and ethers]. IMPERIAL CHEM. INDUSTRIES, LTD., W. W. TATUM, W. M. TODD, L. J. HOOLEY, and J. THOMAS (B.P. 358,033, 28.3.30).— Acetate silk etc. is dyed, printed, etc. in green to bluegreen shades by application of dihydroxydithiolanthraquinones in which all the OH and SH groups occupy  $\alpha$ -positions, or of the compounds obtained by alkali hyposulphite reduction of Solway-emerald or by interaction of alkali polysulphide and a dihalogenodihydroxyanthraquinone in which both halogens and hydroxyls are in  $\alpha$ -positions. C. HOLLINS.

Production of textile or the like material coated with rubber or the like. DUNLOF RUBBER Co., LTD., and H. WILLSHAW (B.P. 361,921, 23.8.30).—A sheet or web of weftless material, formed from individual cords, is coated with rubber and divided into strips which are then spooled. D. F. TWISS.

Impregnation of textile fabrics [with rubber]. J. H. FENNER & Co., LTD., and J. H. FENNER (B.P. 361,999, 4.9.30).—During the weaving of a textile fabric, an impregnating material, such as rubber latex, is sprayed on to the warp alone, or on the warp and weft. D. F. Twiss.

Washing, dyeing, rinsing, and drying machine. C. FLORI (B.P. 363,927, 1.4.31).

Drying apparatus [for knitted goods] and forms therefor. E. J. BERGER, H. W. MATTHEWS, and W. BOCK (B.P. 362,708, 30.12.30).

Salts of alkylsulphuric acids. Wetting etc. agents. Inhibiting development of micro-organisms.—See III.

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

Ammonia synthesis as a physico-chemical, technical, and economic problem. R. PODHORSKY (Arh. Hemiju, 1931, 5, 314—340).—A short historical survey of the development of synthetic NH<sub>3</sub> plants is followed by a detailed discussion of the theoretical principles involved in the synthesis and a discussion of the various methods which have been devised to solve the problems of manufacturing NH<sub>3</sub> from N<sub>2</sub> and H<sub>2</sub> on a commercial scale. A. R. POWELL.

Ammonia-soda process. N. F. JUSCHKEVITSCH, A. L. URAZOV, and L. S. SOLOVIEVA (J. Chem. Ind., Russia, 1930, 7, 1728-1734, 1889-1900; 1931, 8, 478-485, 581-596).-Empirical equations are given for calculating the concns. of Na, Cl, NH4, alkali, CO2, and H<sub>2</sub>O at 15°, 20°, and 30°, and at vapour pressures of 0.5, 1, 2, and 3 atm. Whatever the original composition of the solution, the solid phase in contact with the equilibrium solution always contains only NH<sub>4</sub>Cl and NaHCO<sub>3</sub>. The [NH<sub>4</sub>] increases with decrease of pressure and rise of temp. and [Cl']. In solution with saturated NaHCO3 and NH4Cl it decreases with increase in [Cl'], but pressure has little effect. In saturated solutions of  $NaHCO_3$  and  $NH_4Cl$  the [Na] rises with increase of [Cl']. The optimal yield of Na<sub>2</sub>CO<sub>3</sub> is obtained when the NH4HCO3, NaHCO3, and NH4Cl are in equilibrium in the solution ([Cl'] 5.5 equiv. per. litre). At higher temp. or pressure the yield is increased (80%). The optimal yield of NH<sub>3</sub> (94%) is obtained when the solution is saturated with NaCl, NH<sub>4</sub>Cl, and NaHCO<sub>3</sub>, but at [Cl'] optimal for yield of Na<sub>2</sub>CO<sub>3</sub>, that of NH<sub>3</sub> is also about 80%. The ratio [NH4] : [Cl'] increases with rise of temp. and decrease of pressure. The % carbonisation (100  $\times$  [CO<sub>2</sub>]:[alkali]) increases in equilibrium solution with increase in pressure and fall in temp., but the change in % carbonisation with pressure has little effect on the yield of Na<sub>2</sub>CO<sub>3</sub>. Solid NaCl may be added to the solution during addition of CO<sub>2</sub>, and dissolves as the NaHCO<sub>3</sub> ppts. The yield of Na<sub>2</sub>CO<sub>3</sub> is improved by lowering the temp. at the end of the addition of CO2 and increasing the amount of NH4 in the solution. CHEMICAL ABSTRACTS.

Continuous causticising by the lime-soda process. R. F STEWART and R. J. VENN (Proc. Tech. Sect. Paper Makers' Assoc., 1931, 12, 210-230).-The Dorr processes for the continuous causticising of Na<sub>2</sub>CO<sub>3</sub> solutions from the recovery plants of pulp and paper mills are considered in detail. Continuous slaking of CaO, with the addition of part of the dil. NaOH liquor, is followed by classification of the suspension to remove grit. A coarse suspension is desirable for controlling the settling of the final CaCO<sub>3</sub> ppt. The "green liquor" meets the CaO at 70-80° in steam-heated, air-agitated reaction vessels. Dorr thickeners in series are used to decant the caustic liquor and wash the sludge. Many arrangements of plant are possible and representaive flow diagrams are given. The effects of various factors on time of causticising and rate of settling are illustrated graphically. Economic advantage over batchcausticising is claimed where 10 tons or more of NaOH per day are used. Heat and power requirements are small and control is simple. T. T. Potts.

M. O. CHAR-Preparation of active silica gel. MANDARYAN and S. L. KAPELLEVICH (J. Chem. Ind., Russia, 1930, 7, 1484-1490).-SiO<sub>2</sub> gel prepared from commercial Na2SiO3 solution and the min. quantity of acid required for gelatinisation produced the lowest yield of a gel of max. activity ; the activity is greatly reduced by the use of a slight excess of acid. Washing the gel after drying greatly reduces its adsorbing power for C<sub>6</sub>H<sub>6</sub>. The activity of a gel is increased by elimination of Cl and other electrolytes by washing. Washing with dil. aq. NH3 increases the activity of gels pptd. from slightly acid media. The effects of concn. of reactants on the activity were examined. Substitution of H<sub>2</sub>SO<sub>4</sub>, FeCl<sub>3</sub>, or AlCl<sub>3</sub> for HCl gave satisfactory CHEMICAL ABSTRACTS. results.

Ammonium thiosulphate. W. KLEMPT, F. BROD-KORB, and H. ERLBACH (Ber. Ges. Kohlentech., 1931, 3, 493—496).—A solution of  $(NH_4)_2S_2O_3$  in  $H_2O$  cannot be cryst. by evaporation without decomp., except *in* vacuo at 40—45°. This method gives very fine crystals which do not filter well. Good crystals are obtained by injection of a finely-divided current of air free from  $CO_2$  at 40—50°. The solubility is 60.3 g. per 100 c.c. at —10° or 69.4 g. at 60°. In presence of  $(NH_4)_2SO_4$ it is difficult to crystallise fractionally with good yields, neither is it satisfactory to remove sulphate as  $CaSO_4$ or  $BaSO_4$ , owing to filtration difficulties. The thermal decomp. of  $(NH_4)_2S_2O_3$  is incomplete, part subliming unaltered. With HCN (in coke-oven gas etc.) the reaction  $(NH_4)_2S_2O_3 + HCN + NH_3 = NH_4CNS + (NH_4)_2SO_3$  proceeds, but as a method of HCN washing it is unsuitable, owing to the difficulty of separating the products. C. IRWIN.

Conversion of ammonium thiosulphate into sulphate and mixed fertilisers. W. KLEMPT and F. BRODKORB (Ber. Ges. Kohlentech., 1931, 3, 484-492). -If  $H_2SO_4$  (d 1.84) is added to conc. aq.  $(NH_4)_2S_2O_3$ the reaction  $H_2S_2O_3 = H_2O + S + SO_2$  is instantaneous, but practically no SO2 is evolved as it reacts with further  $H_2S_2O_3$  to form polythionates. The decomp. of the latter is much slower and requires heat. Decomp. with HNO3 proceeds similarly and there is no loss of N. The pptd. S can readily be filtered. KH<sub>2</sub>PO<sub>4</sub> is not capable of decomposing thiosulphate, but H<sub>3</sub>PO<sub>4</sub> acts similarly to the other acids tried. An equimol. mixture of HNO3 and H3PO4 can also be used. Addition of liquid H<sub>2</sub>SO<sub>4</sub> and gaseous SO<sub>2</sub> is also practicable, but the quantity of the latter used must be adjusted to the thiosulphate present. If the (NH4)2S2O3 is added to the H<sub>2</sub>SO<sub>4</sub> with air-blowing to remove the SO2 the first reaction occurs approximately, but polythionates are formed in the solution. If it is added to HNO<sub>3</sub> a violent reaction occurs with loss of gaseous N<sub>2</sub>. C. IRWIN.

Conversion of chromite into chromium salts. A. S. SOFIANOPOULOU (Praktika, 1928, 3, 385–391; Chem. Zentr., 1931, ii, 102).—100 kg.  $Cr_2O_3$  requires 140 kg. CaCO<sub>3</sub> and 136 kg. Na<sub>2</sub>CO<sub>3</sub>. The mixture is heated at 760° for 12 hr. in a current of air (50 cu. m. per min.); the charge is 6.97 kg.  $Cr_2O_3$  per cu. m. A. A. ELDRIDGE.

Carbon monoxide recovery from industrial gases. I. Using non-cuprous or alkaline cuprous salt solutions. W. GLUUD and W. KLEMPT [with H. ERLBACH and H. RITTER]. II. Using acid cuprous salt solutions. W. GLUUD, W. KLEMPT, and H. RITTER. III. Apparatus for above and results. R. BESTEHORN and E. CURLAND (Ber. Ges. Kohlentech., 1931, 3, 505-524, 525-533, 534-543).-I. A cheap method of recovering CO from blast-furnace gas and the like would be of great advantage to industry. The compounds which CO forms with Na3Fe(CN)5 or the corresponding Ni or Co compounds are not suitable for the purpose. The remaining absorbents studied are essentially Cu<sub>2</sub>Cl<sub>2</sub> in either aq. NH<sub>3</sub> or dil. HCl. The aq. NH<sub>3</sub> of corresponding molar content absorbs CO more rapidly than does dil. HCl. It has only an absorption capacity of 15 times its own vol., however, which limits its usefulness, and is corrosive to Fe. No benefit is obtained by replacing Cl by other anions, or NH<sub>3</sub> by org. bases, of which a large number were tried. More promising results were given by an hyd. Cu<sub>2</sub>Cl<sub>2</sub> in non-aq. org. solutions, e.g., a 1.6% solution of Cu<sub>2</sub>Cl<sub>2</sub> in pyridine to which a 3:1 C<sub>6</sub>H<sub>6</sub>-NH<sub>2</sub>Ph mixture was added was as efficient as the ordinary cuprammonium solution, though rather slower acting. A mixture of 2 g. of Cu<sub>2</sub>Cl<sub>2</sub>, 3 c.c. of cresol oil, 2 c.c. of PhNO<sub>2</sub>, and 3 c.c. of pyridine is still better, taking up 35 vols. of CO. The gas is recovered by warming to 60-70°. The solution does not deteriorate with use, though it is

sensitive to  $O_2$ . A small, continuous CO washing apparatus was designed, which worked well.

II. No other acid can replace HCl with advantage in acid  $\text{Cu}_2\text{Cl}_2$  solutions. A method of procedure based on the formation of the solid compound  $\text{CuCl},\text{CO},2\text{H}_2\text{O}$ is as follows. The gas is washed with a suspension of  $\text{Cu}_2\text{Cl}_2$  in its neutral or acid solution until the solid is pptd. The gas is compressed or the solution cooled so that the partial pressure of the gaseous CO is higher than the v.p. of the CO in solution. The solid is heated to 35° or to lower temp. under reduced pressure. Absorption is reasonably rapid. A mixture of Cu,  $\text{CuSO}_4$ , and NaCl may replace the  $\text{Cu}_2\text{Cl}_2$  as  $\text{Na}_2\text{SO}_4$ does not interfere and the reaction  $\text{Cu} + \text{CuCl}_2 = \text{Cu}_2\text{Cl}_2$ is catalysed by CO. In presence of Cu the solution is much less sensitive to  $\text{O}_2$ . Good agitation of the solid  $\text{Cu}_2\text{Cl}_2$  is necessary for efficient absorption.

III. The only suitable material for apparatus for operating the above-described  $\operatorname{acid}-\operatorname{Cu_2Cl_2}$  process is Cu, and construction is simplified if CO evolution is effected by pressure reduction alone without heating. Stripping is best performed in several shallow vessels in series. A small works-scale plant is described. The CO washer is jacketed. The gas is previously freed from O<sub>2</sub> in a vessel containing Cu<sub>2</sub>Cl<sub>2</sub> and Cu, and the treated gas is passed through NaOH to remove HCl vapours. Gas was passed at the rate of 5 cu. m./hr. and the initial stripping efficiency was 94%. This gradually fell as saturation was approached. Two washers in series give better results. The CO recovered was of 98.7% purity. C. IRWIN.

Manufacture of carbon disulphide in Spain. J. BOHLE (Chem.-Ztg., 1931, 55, 981—982).—A description of Spanish practice. E. S. HEDGES.

Determination of active oxygen by oxidation with hydrogen peroxide. W. SCHRAMEK and H. GIEHLER (Z. anal. Chem., 1931, 87, 1-7).—An apparatus is described for determining the amount of  $H_2O_2$  used up in autocatalytic decomp., as compared with that employed in useful oxidation, when  $H_2O_2$  is used as an oxidising agent. Data are given for various alkaline  $H_2O_2$  bleaching liquids for cotton. M. S. BURR.

S from gases.—See II. Soda from pulp liquors. S etc. for sulphite pulp.—See V. Bleach liquors.— See VI. Al apparatus.  $H_3PO_4$ -resistant alloys. Analysis of Ag-plating solutions.—See X. Determining Fe in red lead.—See XIII.

See also A., Jan., 11, Phosphorescent ZnS. 16, Occlusion of H<sub>2</sub> by Pt-black, and by Pd-Ag alloys. 19, Pptn. of Th(OH)<sub>4</sub> from aq. Th(NO<sub>3</sub>)<sub>4</sub>. 28, Catalytic properties of ThO<sub>2</sub>. 30, Prep. of pure salts by means of zeolites. 33–35, Determinations of ClO<sub>3</sub>' and F', of PO<sub>4</sub>''' in phosphorites and apatites, and of permanganate in presence of manganate, respectively.

#### PATENTS.

Recovery of sulphuric acid from waste acids produced in the refining of mineral or tar oils. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 363,205, 31.1. and 19.10.31).—Sludges, *e.g.*, from the treatment of benzine or benzol, are diluted with 1—2 pts. of water with the addition, if desired, of about 3% of lignite coke, active C, bentonite, etc., and dil.  $H_2SO_4$  is recovered by centrifuging or filtering; residual org. matter may be used for waterproofing building materials. L. A. COLES.

**Production of nitric acid.** E. COLLETT (B.P. 362,908, 8.9.30).—N<sub>2</sub>O<sub>4</sub> obtained, e.g., by the expulsion of oxides of N from solution in H<sub>2</sub>SO<sub>4</sub> and subsequent oxidation of the gases, is absorbed in 90% HNO<sub>3</sub>, and the mixture, after dilution to < 80% HNO<sub>3</sub>, is treated with O<sub>2</sub> under pressure to convert the N<sub>2</sub>O<sub>4</sub> into HNO<sub>3</sub>; part of the acid leaving the absorption apparatus is returned to the process, and the remainder, corresponding to that formed from the N<sub>2</sub>O<sub>4</sub>, is distilled, after removal of any unoxidised N<sub>2</sub>O<sub>4</sub>, to yield highly conc. acid and a fraction of about 70% conc., the latter being used to dil. the absorption acid.

L. A. Coles.

**Decomposition of ammonia.** DUPONT AMMONIA CORP. (B.P. 363,300, 31.7.31. U.S., 1.8.30).—Liquid NH<sub>3</sub> is vaporised and the gas passed through a decomposer consisting of an annular catalyst chamber (A) surrounding and spaced apart from a central electric heater (B). The decomp. products pass through the annular space between A and B and are then led to the apparatus for vaporising the NH<sub>3</sub> before being collected. F. YEATES.

Manufacture of sal ammoniac and soda. A. JACOB (B.P. 363,218, 13.2.31).—Liquid  $NH_3$  is used as the reaction medium when making  $NH_4Cl$  and  $Na_2CO_3$  by the reaction of NaCl with  $NH_4HCO_3$  or  $(NH_4)_2CO_3$ , with a consequent higher % conversion of the raw materials. F. YEATES.

Manufacture of sodium cyanide. A. MENTZEL (B.P. 363,149, 29.12.30. Ger., 3.1.30).—Preheated  $N_2$ acts at a high temp. on briquettes made of mixtures of C with Na<sub>2</sub>CO<sub>3</sub> or the like, to which NaCl (as rock salt) has been added in an amount up to  $\frac{1}{4}$ — $\frac{1}{5}$  of the Na in the mixture. The N<sub>2</sub> is preheated to a temp. above that of the solid reaction mixture. F. YEATES.

Recovery of values from cyanide solutions. H. T. DURANT, H. L. SULMAN, and H. F. K. PICARD (B.P. 362,861, 8.9.30 and 9.4.31).—The solutions, after use for the extraction of Au or Ag, are treated at  $p_{\rm H}$  7.5—8 with ZnSO<sub>4</sub> to ppt. Zn(CN)<sub>2</sub>, which is removed, and the liquor after slight acidification is treated with Zn to ppt. Au or Ag. Alternatively, these metals and Cu are pptd. together with the Zn(CN)<sub>2</sub> and are subsequently recovered from the ppt. The Zn(CN)<sub>2</sub> is treated with H<sub>2</sub>SO<sub>4</sub> to yield HCN and to recover ZnSO<sub>4</sub> for re-use. L. A. COLES.

**Production of sulphates.** METALLGES. A.-G., C. (BARON) VON GIRSEWALD, and E. STAHL (B.P. 363,215, 10.2.31. Addn. to B.P. 350,050; B., 1931, 757).—A mixture of  $(NH_4)_2SO_3$ ,  $NH_4HSO_3$ , and  $(NH_4)_2SO_4$  prepared by the action of  $SO_2$  (0.5—1 mol.) on  $NH_3$  (1 mol.) in the presence of  $H_2O$  and air and preferably deposited by the action of an electrostatic field, is used for the production of  $(NH_4)_2SO_4$  by the prior process, sufficient  $NH_3$  being added before or during oxidation to convert the  $NH_4HSO_3$  into  $(NH_4)_2SO_4$ . Alternatively, the gases are passed into conc.  $(NH_4)_2SO_4$  solution and the liquor, after removal of the pptd. salt, is neutralised and oxidised as before. L. A. Colles.

Burning of barium carbonate to oxide. E. L. RINMAN (B.P. 363,299, 25.7.31).—BaCO<sub>3</sub> is intimately mixed with at least the equiv. quantity of CaCO<sub>3</sub>, and preferably with such proportions of CaCO<sub>3</sub> that for each unit of BaO (by wt.) there is I unit of CaO. The mixture is then burned during a slow rise of temp. up to approx. 1400°. F. YEATES.

Manufacture of solidified carbon dioxide and the like. J. GRISON (B.P. 363,870, 28.1.31).

Catalytic apparatus. Apparatus for effecting synthesis.—See I.  $H_2S$  from gases.—See II. Acidresistant steels. Cu extraction. Recovery of Zn. Sn ores. Mn from ores.—See X.  $H_2S_2O_8$  or its salts by electrolysis. Pb(OH)<sub>2</sub> by electrolysis.—See XI. Zn white.—See XIII. Fertilisers.—See XVI. Cacarbohydrate compound.—See XX. Gas filters.— See XXIII.

#### VIII.—GLASS; CERAMICS.

Making glass from blast-furnace slag. C. A. BASORE (Chem. and Met. Eng., 1931, 38, 701).—Nearly colourless glass, of high tensile strength, good thermal endurance, and high resistance to hot 2N-NaOH and boiling HCl and H<sub>2</sub>O can be made by melting at 1370— 1420° for 100—180 min. a mixture of 7 pts. of granulated blast-furnace slag, 8 pts. of sand, 1.76 pts. of Na<sub>2</sub>CO<sub>3</sub>, and 2.1 pts. of Na<sub>2</sub>SO<sub>4</sub>,10H<sub>2</sub>O; 1% of As<sub>2</sub>O<sub>3</sub> is added to eliminate S as As<sub>2</sub>S<sub>3</sub> and As<sub>2</sub>S<sub>5</sub> and oxidise Fe<sup>--</sup> to Fe<sup>--</sup>. D. K. MOORE.

Casting and tempering of silicate melts, with special reference to mill clinkers. R. GRÜN and H. MANECKE (Z. angew. Chem., 1931, 44, 985—990).— The physical properties of cast silicates have been examined. The castings can be obtained either in glassy or coarsely-cryst. form. Tempering is best carried out at a temp. at which the melt is still a viscous liquid. E. S. HEDGES.

Interpolation formulæ for the refractive indices of glass. (MRS.) E. GIFFORD (Trans. Optical Soc., 1930— 31, 32, 69—72).—Simple formulæ for 5 lines are given, making use only of the measured indices for the C, D, and F lines. N. H. HARTSHORNE.

Silvering and evacuating pyrex Dewar flasks. R. B. Scorr, J. W. Cook, and F. G. BRICKWEDDE (Bur. Stand. J. Res., 1931, 7, 935-943).—The process includes (1) modification of the concn. of the reagents in the Brashear formula for silvering, (2) methods for preserving the reflective properties of the Ag coat when heated at high temp. during evacuation, (3) baking out in 2 stages of falling temp., (4) pumping off the gases evolved during scaling. In the improved flasks, liquid air changes to the gaseous phase entirely by surface evaporation. E. S. HEDGES.

Progress report on investigation of saggar clays. VI. Life of the saggar as affected by varying certain properties. R. A. HEINDL and L. E. MONG (J. Amer. Ceram. Soc., 1931, 14, 867—876; cf. B., 1929, 1044).—The effects of composition, grog content, and firing temp. on the structure, porosity, thermal expansion, strength (hot and cold), elasticity, resistance to

thermal shock, etc. in a series of 10 commercial and 35 laboratory-prepared bodies were determined. It is not possible to prepare from the usual saggar clays a body having all the desired characteristics, but with a knowledge of the properties of the ingredients, the most desired may be developed at the expense of the less desired characteristics. E.g., a body containing coarse grog is resistant to thermal shock but has a high plastic flow, whilst fine grog gives low plastic flow but poor resistance to thermal shock. J. A. SUGDEN.

Effect of grog on pressure transmission in drypressing. C. M. DODD and M. E. HOLMES (J. Amer. Ceram. Soc., 1931, 14, 899–905).—The transmission of pressure (as judged from the porosity) in the drypressing of a fine-grained clay is far from uniform, and the interior may differ from the exterior porosity by 15% of the former. The degree of pressure transmission is increased by the addition of grog with a max. effect at 50% of grog. J. A. SUGDEN.

Thermal-shock effect on the transverse strength of clay bodies. W. R. MORGAN (J. Amer. Ceram. Soc., 1931, 14, 913—923).—As the initial strength of bodies with a softening point of cone 7—30 and fired at cones 04—6 increased, the strength after, and reduction in strength due to, thermal shock increased to a max. and then declined. J. A. SUGDEN.

Proportioning the grain size in clay refractories. S. M. SWAIN and S. M. PHELPS (J. Amer. Ceram. Soc., 1931, 14, 884-898).-A large number of bodies were prepared from flint-clay particles bonded with plastic clay to study the effect of varying the proportions of the fine, medium, and coarse particles and the amount of bond clay. The max. density of the unfired body is given by equal parts of fine and coarse sizes free from medium sizes. The bulk density of an unfired body is not necessarily an index to that of the fired body. The firing shrinkage can be varied over a wide range by proportioning the particle sizes, and increases with the content of fines. Increasing the plastic clay content has less effect than proportioning the particle sizes. The porosity and texture (as affecting spalling, slag penetration, resistance to load, etc.) are greatly influ-J. A. SUGDEN. enced by the particle sizes.

Sillimanite kiln refractories made from an andalusite base. F. H. RIDDLE and R. TWELLS (J. Amer. Ceram. Soc., 1931, 14, 877–883).—Good andalusite materials have a refractoriness of above cone 40, show no deformation at 1700° under 25 lb. per sq. in., and at 1525° under 50 lb. per sq. in. are resistant to thermal shock, unaffected by kiln conditions, and can easily be moulded and fired. Plant trials in zones of particular severity (flame shields, saggars, etc.) have shown the high qualities of andalusite as a high-temp. refractory.

SiO<sub>2</sub> mortars.—See IX. Glass for medicaments. —See XX.

See also A., Jan., 14, Vitreous state [for glass]. 98, Winter sunlight [and ultra-violet light-transmitting glasses],

#### PATENTS.

Glass furnaces. UNITED GLASS BOTTLE MANUFRS., LTD., T. C. MOORSHEAD, and F. A. HURLBUT (B.P. 351,636, 22.4.30).—A furnace consisting of a rotary, annular trough for the glass batch, over about half of which are disposed a number of heating chambers with their lower ends open, is claimed. The chambers are preferably electrically heated and can be independently controlled. F. SALT.

Manufacture of [white] glass. J. M. WILSKER (B.P. 362,938, 8.9.30).—Sufficient red sand is included in the ingredients of soda-lime glass to yield a product containing equal proportions of FeO and  $Fe_2O_3$ ; other decolorising and colour-correcting agents (Se, Co, NaNO<sub>3</sub>, As) may also be added. L. A. COLES.

Colouring of enamel glazings, glass, and the like. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (B.P. 361,898, 26.8.30. Ger., 24.9.29).—The stability and uniformity of glass coloured red by means of Se or its compounds is improved by the addition of small quantities of rare-earth oxides, e.g., of Ce or La. (Cf. B.P. 354,566; B., 1931, 972.) C. A. KING.

Decoration of ceramic objects [with dust gold]. DEUTS. GOLD- U. SILBER-SCHEIDEANSTALT VORM. ROESS-LER (B.P. 351,531, 25.3.30. Ger., 30.3.29).—Relatively small quantities of Rh, Cr, or U are added to the dust Au, or to the lacquer, oil, or other carrier, in order to prevent or minimise volatilisation of the Au in the glost oven. The usual colouring effects may be obtained by the addition of Ag, Cu, Bi, etc. F. SALT.

Manufacture of refractory ceramic ware. (MISS) E. LUX (B.P. 361,858, 25.7.30).—Non-plastic filling material (grog) is treated with  $H_2O$  containing a deflocculating agent before mixing with the raw clay, to produce a plastic mass. C. A. KING.

Decoration of porcelain and the like. R. H. SCHLINDENBUCH (B.P. 362,253, 24.2.31. Czechoslov., 17.5.30).—The entire surface of an article is coated with paint, the design sketched by hand, and paint removed where desired with a stylus. C. A. KING.

Manufacture of bricks. H. NIELSON (B.P. 362,102, 21.10.30).—The moisture in raw clay is reduced or adjusted to 5—10% and the clay granulated to about 2.5 mm. in diam. Bricks are pressed from the material under a pressure of 300 kg. per sq. cm. and burned.

C. A. KING. Manufacture of articles from basalt. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of H. L. WATSON (B.P. 363,291, 15.6.31. U.S., 16.6.30).—Basalt, after melting under controlled reducing or oxidising conditions, is cast in moulds, and the articles, set in saggers containing sand and graphite, are heated uniformly to 725° and then at 900° for about 1 hr. L. A. COLES.

Mechanical production of compound or flashed sheet glass. B. ENGELS (B.P. 363,385, 13.9.30. Ger., 23.9.29).

Manufacture of rings, or other hollow bodies, of fuzed quartz. DEUTSCH-ENGLISCHE QUARZ-SCHMELZE G.M.B.H. (B.P. 363,610, 8.7.31, Ger., 10.9.30).

[Installation for] producing pottery ware. W. J. MILLER (B.P. 364,181, 7.7.30).

Waste gases from furnaces.—See I. Wetting etc. agents.—See III.

J. A. SUGDEN.

Long slurry ring in a rotary [cement] kiln. O. FREY (Cement, 1931, 4, 1341—1343).—A ring 49 ft. long occurred in a 207-ft. rotary kiln when using a raw mixture of rather high silicate modulus (3.5). It was caused by the high CaSO<sub>4</sub> (9.76%) content of the coal ash which must have fallen on the material in the form of a liquid spray, thus making it sticky. The clinker ring was completely free from sulphates. C. A. KING.

Recent developments in the wet process of cement manufacture. E. SCHIRM (Cement, 1931, 4, 1355— 1366).—The object of the various methods described is to provide a "wet process " with heat economy approximating to that of the modern dry process. The processes include drying the slurry by filter-pressing, spraying the slurry on to chains hung in the path of the gases, and the Lellep process which is suitable only for a reasonably dry raw material. C. A. KING.

Mineralogy of cement. A. C. DAVIS (Cement, 1931, 4, 1351—1354).—The preponderating minerals recognised in cement clinker are alite and celite together with smaller amounts of belite and felite, the latter two occurring in disintegrated cement of low strength. The importance of cement "glass" is not agreed. X-Ray study indicates that the most abundant constituents are Ca<sub>3</sub> silicate and  $\beta$ -Ca<sub>2</sub> silicate together with Ca<sub>3</sub> aluminate, Ca<sub>4</sub> aluminoferrite, and MgO. Free CaO is normally <2.5%. C. A. KING.

Effect of gypsum on the soundness of high-limed clinker. O. GOFFIN and G. MUSSGNUG (Cement, 1931, 4, 1344-1350).-The limiting CaO contents given by Guttmann's lime modulus of 3 or Kühl's lime-saturation factor of 1 can be exceeded without giving expansion as shown by the usual soundness test pieces. In more accurate observations all clinkers exceeding the above CaO figures showed a serious expansion when ground without or with up to 2% of gypsum, but with >3%the boiling and kiln tests showed no indication of expansion. The Heintzel ignited-sphere test and the Prüssing compressed-pat kiln test continued to indicate a degree of expansion harmless in practice, and both tests are too stringent probably because the cement is tested before it has set. It is possible to increase the soundness of cement by increasing the amount of added gypsum, but as the standard specification limits the quantity of added material to 3%, the improvement can be only theoretical. No appreciable effect was observed by merely adding an indifferent material, e.g., limestone or C. A. KING. sand.

Relation between physico-chemical and technically important properties of concrete. D. WERNER and S. GIERTZ-HEDSTRÖM (Tek. Tidskr., 1931, 61, No. 17, 45—53; Chem. Zentr., 1931, ii, 108).—Results of the determination of mechanical properties are recorded and correlated mathematically. Technically important properties depend chiefly on the manner of combination of  $H_2O$  in the system cement- $H_2O$ . A. A. ELDRIDGE.

Thermal expansion of silica mortars after firing at 950°, 1200°, and 1500°. S. S. COLE and D. C. LYNN (J. Amer. Ceram. Soc., 1931, 14, 906—912).—Although, in general, the expansion of a SiO<sub>2</sub> mortar tends to decrease on firing, it undergoes a marked change only when fired above  $1200^{\circ}$ ; such change is due to the formation of tridymite. With rise in firing temp. the expansion approaches that of a SiO<sub>2</sub> brick. J. A. SUGDEN.

Surface conditions of wall plaster as they affect paint. F. C. ATWOOD (Amer. Paint & Varnish Manufrs.' Assoc., Dec., 1931, Circ. No. 404, 501—521).—A general account is given of the materials used in plastering and their application, stress being laid on inequalities in the surface that lead to paint failure, actual and virtual. S. S. WOOLF.

Treatment of plaster surfaces for painting. BIDLACK (Amer. Paint & Varnish Manufrs.' Assoc., Dec., 1931, Circ. No. 404, 561—562).—A preliminary account is given of an investigation into the alkalinity of plaster surfaces and the effect of treatment with chemicals, e.g., ZnSO<sub>4</sub>, solutions of glue, gums, oils, etc. S. S. WOOLF.

Relation between durability and the extractives of the cypress pines (Callitris, spp.). I. W. DADSWELL and H. E. DADSWELL (J. Counc. Sci. Ind. Res., Australia, 1931, 4, 208—216).—Durability is due to the existence in the medullary ray cells and the parenchyma tissue of a volatile acid identified as *l*-citronellic acid. The material is highly toxic towards the wood-destroying fungus *Fomes annosus*, and repellent to termites, *Eutermes exitiosus*. When isolated and used alone as a preservative it is slowly volatilised or decomposed on exposure to air and is consequently presumed to be protected in its natural state by the associated resinous substances. W. G. EGGLETON.

Composition of wood of Trochodendron aralioides. J. WIERTELAK (J. Forestry, 1931, 29, 64-67).—The dry wood contains 20.46% of pentosan and 6.7% of OMe, and gives 7.42% of volatile acid on hydrolysis. The coloration given by Mäule's reagent (1% KMnO<sub>4</sub> followed by HCl and aq. NH<sub>3</sub>) is due to the Cl<sub>2</sub> liberated from the HCl by MnO<sub>2</sub> derived from the KMnO<sub>4</sub>. The reaction is similar to Vincent's Cl<sub>2</sub>-NH<sub>3</sub> test and to Cross and Bevan's Cl<sub>2</sub>-Na<sub>2</sub>SO<sub>3</sub> reaction.

CHEMICAL ABSTRACTS.

Painting new wood. Wall plaster and paint. Painting wood.—See XIII.

See also A., Jan., 23, Thermochemistry of Portland cement.

#### PATENTS.

Manufacture of cement. W. W. TRIGGS. From EDDYSTONE CEMENT CORP. (B.P. 361,901, 21.5.30).— Hydraulic cement is prepared by grinding together a mixture of CaO, burned to 900—1200°, and clay or shale calcined at 600—900°. The ground product is then agitated with a definite quantity of  $H_2O$  or steam to liberate active SiO<sub>2</sub>. C. A. KING.

Manufacture of concrete aggregates. J. E. GREENAWALT (B.P. 351,689, 23.5.30).—Finely-crushed clay is mixed with 25—50% of finely-crushed furnace cinders, the mixture moistened slightly with water, and the charge sintered by down-draught ignition (in an apparatus described) in such a way as to preserve the porous nature of the final product. F. SALT.

aa 3

Sintering of clay [for artificial concrete]. J. E. GREENAWALT (B.P. 351,690, 23.5.30).—Finely-ground clay is made into a slurry with water, the slurry converted by heat into a homogeneous mass of particles about the size of a pea, and these are mixed with fuel and ignited on a perforated grate through which air is drawn. Combustion of the fuel proceeds through the mass, and a hard, porous aggregate is produced suitable for concrete etc. The fuel may be added to the clay before the mixture is heated. F. SALT.

Priming or surfacing absorbent foundations [of stone, wood, etc.]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 362,479, 27.8.30).—Compositions for the purpose comprise aq. emulsions of condensation products from polyhydric alcohols (e.g., glycerol, pentaerythritol), polybasic acid anhydrides (e.g., of phthalic, malic, and maleic acid), and highly unsaturated monobasic carboxylic acids (e.g., linoleic, elæostearic, adipic acids); emulsifying agents, drying agents, and fillers may also be added. [Stat. ref.] L. A. COLES.

Production of agglomerates [for road-surfacing material]. H. R. SEABROOK (B.P. 351,385, 17.12.29, 31.3.30, and 26.9.30).—A bituminous binder is mixed with damp soil under the application of heat. The mixing and heating are prolonged until the soil is reduced to fine particles and the agglomerate commences to granulate. F. SALT.

Composition for building and road-making purposes. (FRAU) F. REIMANN (B.P. 351,680, 21.5.30. Ger., 21.5.29).—The composition consists of cumarone resin containing lithopone, preferably rich in sulphide, as filler, Hard org. substances and small amounts of caoutchouc may also be added. F. SALT.

Manufacture and casting of cement products. W. GARTNER (B.P. 363,637, 19.9.30).

Manufacture of [moulded hollow] articles from hydraulic cement material. U. ISSMANN (B.P. 363,873, 30.1.31. It., 3.2.30).

Production of fireproof floors. Concrete, Ltd., J. G. Ambrose, and C. B. Matthews (B.P. 364,182, 22.4.31).

Screening plant [for stones, gravel, etc.]. A. SHARP and H. A. ASHTON (B.P. 363,994, 18.9.30).

Rotary kilns. Calcination of [cement] material. —See I. Tar emulsions.—See II. Oil treatment [residues].—See VII. Shingles.—See X. Articles resembling wood.—See XIV.

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

Energising action of various chemicals in the carburisation of steel with solid carburising agents. R. A. RAGATZ and O. L. KOWALKE (Met. & Alloys, 1931, 2, 290—296).—The increase in wt. of steel (0.2% C) and the depth of the carburised layer after heating for 4 hr. at 954° in a 25:1 mixture of wood charcoal and the chemical under test as an energiser (the ratio given is C:wt. of metal in energiser) has been determined. Among the activating compounds Na<sub>2</sub>CO<sub>3</sub>, NaOH, NaOAc, Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub>, NaCN, Na<sub>2</sub>CrO<sub>4</sub>, and Na<sub>2</sub>WO<sub>4</sub> are all equally effective; similar results are obtained

with the corresponding K salts and with  $BaCO_3$ , BaO,  $Ba(OH)_2$ ,  $Ba(OAc)_2$ , and  $Ba(NO_3)_2$ , indicating that in all these compounds the metal is the energising agent whereas the anion is inert. Fluorides and S compounds of these metals are, however, either inert or actually inhibit carburisation. Contrary to many claims in the patent literature, all Ca and Mg compounds are free from energising activity.  $SnO_2$  prevents carburisation completely and has little effect on the steel surface, Bi and Sb compounds prevent absorption of C and the steel surface becomes coated with the metal, As compounds coat the surface with As, but have little effect on the depth of case, and Pb compounds exhibit a behaviour intermediate between those of Bi and As. From these results it appears that  $Na_2CO_3$  and  $BaCO_3$  are the cheapest and best energisers.

A. R. POWELL.

Mechanism of energiser action in carburisation [of steel]. R. A. RAGATZ and O. L. KOWALKE (Met. & Alloys, 1931, 2, 343-348).-Previous theories on the mechanism of carburisation are not supported and it is suggested that the energising action exerted by certain chemicals is due primarily to the catalytic effect produced on the C-CO<sub>2</sub>-CO reaction. Using Na<sub>2</sub>CO<sub>3</sub> as the energiser, it is reduced at the surface of the C to Na<sub>2</sub>O and Na, the latter being formed with considerable pressure effecting dispersion of the reaction products. The CO<sub>2</sub> formed at the steel-gas interface by the reaction of CO with the steel combines with the Na to form  $Na_2O + CO$  or, later,  $Na_2CO_3$  which completes the cycle of reaction. High concn. gradients of CO will result in a greatly accelerated transfer rate of CO to the steel surface and hence carburisation will be speeded up. Unlike K<sub>2</sub>O and Na<sub>2</sub>O, BaO will not be reduced to metal, but the partial pressure of CO<sub>2</sub> in equilibrium with BaO is very low. The inactivity of CaCO<sub>3</sub> and MgCO<sub>3</sub> is explained by the decomp. pressure of CO<sub>2</sub> being greater than that demanded by the  $C-CO_2-CO$  equilibrium. The C surfaces become coated with CaO, which can neither react to form CQ nor absorb CO<sub>2</sub> from the gaseous phase by reason of high decomp. pressure of CaCO<sub>3</sub>. Other energising compounds are discussed from the same basis. C. A. KING.

Nitrogenisation of iron and iron alloys. П. O. MEYER and R. HOBROCK (Arch. Eisenhüttenw., 1931-2, 5, 251-260).-With a const. time of nitrogenisation the concn. (c) of N in a layer x mm. from the surface of pure Fe is given by  $c = ae^{-x^2/b}$ , where a and b are consts. The hardness of the surface layers of electrolytic Fe heated in NH<sub>3</sub> at 550° for various periods decreases slightly at first with the distance of the layer from the surface, then increases rapidly to a sharp "peak hardness" of about 190 Brinell at a distance from the surface which increases slowly with the time of nitrogenisation. When nitrided Fe is heated in vac. the position of this peak-hardness zone moves outwards towards the surface at a rate which depends on the temp. The depth of the peak-hardness zone (x) increases with time (t) of nitrogenisation according to the expression  $k_1t + t \log t = k_2x^2$  when the temp. is const. Pptn. of visible needles of nitride occurs only in zones beyond the peak-hardness zone and is accompanied by a decided fall in hardness; hence it is suggested that

the peak hardness is caused by lattice distortion resulting from the presence of uncoagulated nitride. In nitrided Al-Fe alloys the hardness falls sharply with distance from the surface, and in alloys with a high Al content rhythmic pptn. of AlN can be observed; diffusion of N into these alloys is slower than is the case with pure Fe. The hardness of nitrided Al–Fe alloys is attributed mainly to blocking of the slip planes by AlN (an effect which is reduced by increasing the Al above 1%) and to a slight extent to deformation of the Fe lattice by AlN, to nitriding of the Fe ground mass, and, in alloys with high Al, to rhythmic pptn. layers.

A. R. POWELL.

Hardening phenomena in boron-iron alloys, with especial reference to precipitation hardening. R. WASMUHT (Arch. Eisenhüttenw., 1931-2, 5, 261-266).-Binary B-Fe alloys cannot be hardened by heattreatment; in the normalised condition the hardness increases linearly with the B content from 98 with 0% to 350 with 2.5% B. On addition of Mn to the binary alloys considerable increases in hardness may be obtained by quenching and also by age-hardening. The alloy with 2% Mn and 1.26% B has a Brinell hardness of 210 in the normalised condition and 350 when quenched in H<sub>2</sub>O from 1000°; subsequent ageing at 100° further increases the hardness to 420° in 4 hr., but at higher temp. or after more prolonged heating lower hardness vals. are obtained. Addition of B to Ni-Cr steels, e.g., V2A steel, causes a considerable increase in hardness after quenching from 1230° in H<sub>2</sub>O and then annealing at 700-800°; with 1.14% B the hardness rises to 450° in 8 hr. at 800°. Cast Ni-Cr-B-Fe alloys contain segregations of a eutectic resembling ledeburite around the crystal grains; this partly dissolves after forging and quenching from 1130° and on subsequent ageing at 800° is pptd. in a finelydispersed form throughout the crystals. This behaviour is attributed to the greater solubility of the borides of Mn, Cr, and Ni in  $\gamma$ -Fe than in  $\alpha$ -Fe. The tensile strength of V2A steel with 0.55% B after quenching from 1150° and ageing at 800° is 126 kg. per sq. mm. and the yield point > 100 kg. per sq. mm.

#### A. R. POWELL.

Strength at high temperatures of a cast and a forged steel as used for turbine construction. H. J. TAPSELL and A. E. JOHNSON (Dept. Sci. Ind. Res., Eng. Res., Spec. Rept. No. 17, 1931, 33 pp.).—In view of the desire to increase the working temp. of turbine plant, the creep properties of suitable steels were determined. The mechanical properties at 400° and at 450-680° are shown in tabulated form, and, judging from the creep curves for both forged and cast steel, it would be impossible to estimate the precise deformation C. A. KING. at the end of a lengthy period.

Preparation of electrolytic sheet iron. C. KADOTA (Proc. World Eng. Cong., Tokyo, 1931, 33, 127-131).-The electrolyte contained FeCl<sub>2</sub> (10-15%) Fe), NaCl (12%), and HCl (0.005-0.008N); c.d. 3-7 amp. per sq. dm. at about 85°. Heat-treatment at 700° renders the sheet ductile.

CHEMICAL ABSTRACTS. Investigation of special chromium-vanadium steels by means of potentiometric analysis. T. Несско (Z. angew. Chem., 1931, 44, 992—995).—Ап apparatus for potentiometric analysis is described. Directions are given for preparing solutions of special steels. Cr is determined by titrating the oxidised solution with FeSO<sub>4</sub>, and V by potentiometric titration with KMnO4. E. S. HEDGES.

Determination of tungsten in quick-drawn steel as tungstic acid in a Gooch crucible. H. WDOWIS-ZEWSKI (Z. anal. Chem., 1931, 87, 36-38).-By the ordinary methods of determination of W in W- or W-Cr-steels the metal is dissolved in dil. HCl, oxidised with conc. HNO<sub>3</sub>, and weighed either as WO<sub>3</sub> or H<sub>2</sub>WO<sub>4</sub>. If a very exact determination is required the WO<sub>3</sub> must be treated with HF to remove SiO<sub>2</sub> and fused with Na2CO3 or K2S2O7 to remove Fe2O3 or Cr2O3. To purify the  $H_2WO_4$  it is dissolved in hot aq.  $NH_3$  (1:3) on the Gooch crucible and filtered, leaving SiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, and Cr2O3 behind in the crucible. The clear filtrate is neutralised with a slight excess of H<sub>2</sub>SO<sub>4</sub>, and treated with benzidine. The compound of  $H_2^WO_4$  and benzid-ine is collected, washed, dried, and calcined, and the pure WO3 weighed. M. S. BURR.

Corrosion-inhibiting calcium carbonate protective layers in water-pipes. L. W. HAASE (Z. angew. Chem., 1931, 44, 990-992).-A layer of cryst. CaCO, protects the underlying metal from corrosion, but a layer of amorphous CaCO<sub>3</sub> has little or no protective effect, by reason of its porosity. A slow stream of H<sub>2</sub>O through pipes favours the formation of the cryst., protective deposit. E. S. HEDGES.

Destructive action of phosphoric acid on alloys. V. K. PERSHKE, M. P. KAPOSTUIN, and E. V. YUSHMANOV (J. Chem. Ind., Russia, 1931, 7, 875-877).-The resistance of cast Fe and Cu alloys containing Sn, Zn, Al, Pb, Sb, and Ni to the action of  $84 \cdot 7\%$  H<sub>3</sub>PO<sub>4</sub> at 20—150° was studied. Alloys containing 78% Cu are resistant; those containing 63-80% Cu are satisfactory. Alloys with the same Cu content, and nearly equal percentages of Pb, Al, or Sb, have the same resistance. Alloys containing much Pb and Zn are not resistant. Brass with a Cu content of 85-90% and Cu-Ni alloys containing 80% Cu are recommended for apparatus coming into contact with conc.  $H_3PO_4$  at 150°. CHEMICAL ABSTRACTS.

Effect of temperature on some of the physical properties of metals. F. C. LEA and C. F. PARKER (Engineering, 1932, 133, 23-26, 54-55).-The methods and apparatus for determining creep at const. temp. are described. Results showed that creep may take place at high temp. which in time may produce fracture or distortion in vessels or machine parts. Thus a mild steel under a stress of 5 tons per sq. in. at 460° might be expected to have an initial extension of 0.0005 in. per in. and then a slow extension for 10-15 days, after which the creep may become  $< 1/10^7$  in. per in. per hr., so that in 2 years the extension should not exceed 0.0025 in. per in. Mild steels show not only a low "limiting creep stress"  $> 500^{\circ}$ , but also considerable tendency to intercryst. corrosion and oxidation of the pearlitic areas. C. A. KING.

Properties of aluminium [apparatus]. T. Hoff-MANN (Chem. Apparatur, 1931, 18, No. 8; Chem. Zentr., 1931, ii, 307).—Cu salts, even in dil. solution, cause corrosion in Al vessels for  $H_2O$ . Agar agar or gum arabic (0.75%) affords a better protection than Na<sub>2</sub>SiO<sub>3</sub> against NaOH, but against Na<sub>2</sub>CO<sub>3</sub> the reverse holds. Bleaching liquor is rendered harmless by addition of  $H_2O_2$ . Duralumin particles, introduced during rolling, cause corrosion. A. A. ELDRIDGE.

Aluminium alloy resistant to corrosion. H. WOLF (Chem. Fabr., 1931, 4, 493-494).—An alloy of Al with 2% Mn is much more resistant to corrosion than is commercial Al. In the thermal HCl test of Mylius it has a reaction number of 0.13 in the hardworked and 0.48 in the annealed state, compared with 0.7 and 0.93 for 99.5% Al and 2.27 and 3.74 for 99% Al. Hard-drawn sheet of the alloy has a tensile strength of 26 kg./sq. mm., an elongation of 3%, and a Brinell hardness of 59; the corresponding vals. after annealing at 500° are 11.8, 27%, and 32. The alloy can be readily worked by deep-drawing, stamping, and pressing, can be welded as easily as Al, and electroplated in a similar manner to Al. A. R. POWELL.

Heat-treating and forging some light alloys. W. C. DEVEREUX (Metallurgia, 1931, 5, 45-50).-The "impurities," Fe and Si, in Duralumin "Y" and Hiduminium RR alloys are necessary to produce hardening alloys, but attempts to increase the two elements are associated with foundry and machining difficulties. Ingots should be soaked for 6-8 hr. before working, the "Y" alloy at 470-480° and RR 56 at 480-500°, and the first forging should be at  $\lt$  400°. Stamping operations may follow after a 50% reduction, and particular attention should be given to the correctness of the dummy to prevent splitting or lines of weakness. A common defect in stamping from the extruded bar is due to the slipping of the core of the ingot during extension, and the extensive crystal growth due to the maintenance of the plastic-deformation temp. for a prolonged period. Especial care should be observed to prevent internal strains in large work with heavy changes of section by controlling the rate of heating, and overheating should be avoided at every stage of the process. C. A. KING.

Preferred orientation produced by cold-rolling in the surface of sheets of aluminium, nickel, copper, and silver. C. B. HOLLABAUGH and W. P. DAVEY (Met. & Alloys, 1931, 2, 246-250, 302-312; cf. A., 1931, 1115).-The orientation of the crystallites in thick rolled sheets of Ni, Cu, Ag, and Al is such that one face diagonal of the cube always lies in a plane parallel to the direction of rolling and perpendicular to the rolling surface. With Cu, Ni, and Ag the number of passes through the rolls has no effect on the limits of the preferred ranges of positions, and in each case there is a range of preferred positions, symmetrical to the surface of the sheet. With Ag there are two symmetrical preferred ranges about the transverse axis with no limitations about the longitudinal axis, whilst with Ni and Cu there is only one symmetrical preferred range about the transverse axis with symmetrical limits about the longitudinal axis as well. With Al the limits of the preferred positions vary with the degree of rolling; there are two preferred but unsymmetrical ranges

about the transverse axis and the limitations about the longitudinal axis are variable. The differences between these results and those of other investigators is ascribed to the greater thickness of the metal used in this work (0.32-0.05 cm.): this results in the production of an oriented layer on each side of a less strained layer which acts as a cushion so that the straining of each surface layer takes place from only one side; these results should therefore more nearly represent what occurs in actual practice than the results obtained with thin foil. A. R. POWELL.

Silver solders and their use. R. H. LEACH (Met. & Alloys, 1931, 2, 278–283).—The microstructure of Ag-soldered joints in brass, Ni, nickel-silver, monel metal, and Fe soldered with various types of Ag solder are illustrated; all the joints show the presence of a considerable zone of diffusion at the boundaries between metal and solder, the size of the zone varying with the temp. used and the time of heating. Borax or  $B_2O_3$  fluxes should be used on all work except where a refractory oxide is formed, in which latter case alkali bifluoride fluxes are preferable.  $O_2-C_2H_2$  torches are preferable to gas-air or gas- $O_2$  torches, owing to their great flexibility and the wider temp. range in different parts of the flame. A. R. POWELL.

Sherardising. F. L. WOLF (Met. & Alloys, 1931, 2, 341-342).—Examination of several tons of castings which had been exposed to the atm. showed that at least 50-75% treated by the sherardising process were partial or total failures and were much inferior to castings galvanised by the hot process, which showed no failure. If properly and completely covered a sherardised article is resistant to corrosion, but it is stated that such a condition cannot be attained in commercial practice. C. A. KING.

Adherence of zinc coatings. W. H. FINKELDEY (Met. & Alloys, 1931, 2, 266-271). - As a general rule, thin Zn coatings (0.3-0.4 oz. per sq. ft.) adhere better and are more malleable and less prone to crack on deformation than thick coatings (1-3 oz. per sq. ft.). High speed of deformation reduces the adherence of the coating and increases its tendency to crack. The presence of Cd in the bath accelerates the growth of the brittle Zn-Fe compounds in the coating and hence reduces the adherence; the addition of Sn to the bath to improve the brightness of the deposits has a slight adverse effect on the ductility, whereas the addition of 2.5-8% Al is claimed to improve the bending qualities of the galvanised sheets. The composition of the steel base has quite a marked effect on the adherence of Zn, the best adherence being obtained with a 0.6% C steel and the poorest on pure Fe. Heating of galvanised steel causes peeling of the outer layer of pure zinc, but this may be prevented by heating the material for a short time at just above the m.p. of Zn immediately after it has been withdrawn from the galvanising bath. A. R. POWELL.

Lead-zinc eutectic. J. M. HODGE and R. H. HEYER (Met. & Alloys, 1931, 2, 297–301, 313).—Thermal analysis and micrographical examination of Pb alloys containing up to 1% Zn has established the eutectic composition as 0.5% Zn, 99.5% Pb, which agrees closely with the val., 0.49% Zn, calc. from van't Hoff's law for the lowering of the f.p. The solubility of Zn in Pb at the eutectic temp.  $(318 \cdot 2^{\circ})$  is 0.05%. Hypereutectic alloys have a microstructure consisting of very large plates of primary Zn in a eutectic ground mass; on slow cooling considerable segregation of these plates towards the top of the ingot takes place.

#### A. R. POWELL.

Soft solders and their application. G. O. HIERS (Met. & Alloys, 1931, 2, 257-261, 277).—A review of recent work on the properties of solders and soldered joints and a discussion of the mechanism of the soldering process. A. R. POWELL.

Fusion of tin and white-metal residues in a reverberatory furnace. E. T. RICHARDS (Metallbörse, 1931, 21, 627—628, 726—727; Chem. Zentr., 1931, ii, 115).—Sn residues together with stanniferous white-metal residues are worked up in a reverberatory or a smelting furnace; for the production of Sn–Pb alloys containing >45-50% Sn the reverberatory furnace is preferred. The pretreatment of the residues is discussed. A. A. ELDRIDGE.

Principles for the economic comparison of blastfurnace gas purification plants. F. LÜTH (Arch. Eisenhüttenw., 1931—2, 5, 223—230).—The nature of the gas under treatment and the arrangement and method of working the various units of the plant vary so much in different installations that a comparison of the efficiency and economics of the wet-purification, drypurification, and electro-filter types of gas purifiers is possible only by reducing the various factors to a common basis. The method of making these calculations is described and comparative operating and cost data for the three types of purifiers are tabulated.

A. R. POWELL.

Atomic hydrogen arc welding. J. T. CATLETT (Met. & Alloys, 1931, 2, 272-276).—The welding tool comprises two W-rod electrodes inclined at 60° to one another and surrounded by tubes through which  $H_2$  is passed; on striking an arc between the rods a fanshaped zone of at. H is formed in which recombination of the H atoms produces a temp. of about 4000°, using an a.c. of 50 amp. at 100 volts. The fringe of the flame is applied to the parts to be welded so that the welding is made in a bath of  $H_2$  which reduces oxides and produces strong, ductile, and non-porous welds in steel, Fe, Zn, Pt, Al, brass, and monel metal.

A. R. POWELL.

Analysis of cyanide silver-plating solutions. R. M. WICK (Bur. Stand. J. Res., 1931, 7, 913—933).— The accuracy of the existing methods of analysis has been examined. Total CN may be determined by distilling with  $H_2SO_4$ , and total effective CN by titrating with I. Ag may be separated as  $Ag_2S$ , or pptd. as metal by Zn or as AgCN by adding dil.  $H_2SO_4$ , and subsequently determined by any standard method.  $CO_3$ " may be determined by pptg. as BaCO<sub>3</sub> and titrating with acid, or by titrating the original sample with acid after adding AgNO<sub>3</sub> sufficient to combine with the free CN. Cl' may be determined by the usual methods, after removing Ag as  $Ag_2S$  and Fe by pptg. with  $Mn_2Fe(CN)_6$ . The usual methods may be applied to the determination

of Fe and Cu, after decomposing the plating solution with dil.  $H_2SO_4$ . Hg is pptd. as HgS and is extracted from the mixture with  $Ag_2S$  by means of NaOH, repptd., and weighed. Determination of  $NH_3$  by direct distillation is not satisfactory, but may be carried out after all the free CN has been removed by adding  $AgNO_3$ .

E. S. HEDGES.

187

Surface coatings for aluminium alloys. W. H. MUTCHLER (Met. & Alloys, 1931, 2, 324-330).-The naturally produced oxide coating on Al may be made more resistant artificially by chemical or electrochemical means, and the Bengough, Commentz, "Alumilite," Stafford-O'Brien, Jirotka, and McCulloch processes are described. Most of the film surfaces produced are suitable for, and improved by, the application of paints or other org. protective films. Metallic coatings, of which Zn and Cd are probably the best, may be applied by electrodeposition or spraying, but for aeroplane construction any increase in wt. must be a consideration. For severe conditions of exposure the choice of protective coatings is limited, but special material of the Alclad sheet or Duralplat type ensures protection for relatively long periods. C. A. KING.

Plating on zinc die castings. E. A. ANDERSON and E. J. WILHELM (Met. & Alloys, 1931, 2, 337-340).-Following the chemical cleaning of the Zn base, Ni forms the most desirable primary coating irrespective of the ultimate finish. A flash coating of Cu often causes failure due to the diffusion of the Cu film into the Zn. Plated coatings for outdoor exposure should be at least 0.0003 in. thick, and in order to obtain bright deposits a relatively high c.d. and a low [Ni] must be used to prevent the formation of a non-adherent immersion deposit. Ionisation may be depressed by the addition of other salts; a satisfactory bath contains (oz./gal.) Ni salts 10, Na<sub>2</sub>SO<sub>4</sub> 10-15, NH<sub>4</sub>Cl 2-3, H<sub>3</sub>BO<sub>3</sub> 2. The c.d. should be controlled at 12-18 amp. per sq. ft., and the bath is operated best at  $p_{\rm H}$  5.8-6.2 and at room temp. Atm. exposure is regarded as forming the only reliable means of testing the resistance of a plated surface. C. A. KING.

Preparation and some physical properties of strontium-cadmium alloys. H. C. HODGE [with others] (Met. & Alloys, 1931, 2, 355-357).—Alloys were prepared by the electrolysis of a fused eutectic mixture of NaCl and SrCl<sub>2</sub> over molten Cd at the lowest temp. at which the bath was molten, 12—14 amp. at 6 volts proving the most suitable conditions. It was impracticable to obtain alloys containing > 25% Sr. Thermal diagrams gave no precise results, but Cd<sub>12</sub>Sr and CdSr were indicated. In all slowly-cooled specimens with < 6% Sr considerable Cd was present. The alloys exhibited the unusual combination of softness and brittleness, and the hardness increased irregularly with increase in Sr. C. A. KING.

Fuel economy in steel works.—See II. Glass from slag. Silvering pyrex flasks.—See VIII. Metal lacquers. Japanned steel.—See XIII.

See also A., Jan., 15, Heat-treatment and physical properties of alloys (various). 16, Occlusion of  $H_2$  by Pt-black. Effect of  $H_2$  on Pd-Ag alloys. 27, Corrosion of Fe and mild steel. 33, Pptn. of

Pt-black. 35, Spectral analysis [of metals and alloys]. 36, Determination of Cr, V, and Mn in a mixture, and analysis of steel. 37, Electro-magnetic micro-balance in analysis.

#### PATENTS.

Rotary [poppet-]valve furnace. W. E. SANDERS, Assr. to GEN. MOTORS CORP. (U.S.P. 1,809,727, 9.6.31. Appl., 12.8.29).—A furnace with rotary hearth for heating only the ends of metallic rods is described.

B. M. VENABLES. Heat-treatment [of metals]. C. I. HAYES (U.S.P. 1,808,721, 2.6.31. Appl., 25.5.29).—Solid, gaseous, or liquid fuels are burned with sufficient air to yield a gas mixture relatively free from  $O_2$  and containing a predetermined proportion of CO, and this mixture is passed into an electric annealing furnace so as to envelop the metal under treatment; part of the gas is passed at high pressure through a long slot in front of the opening of the furnace so as to provide a screen for preventing ingress of air to the furnace when loading or discharging. A. R. POWELL.

Utilisation of paraffin hydrocarbons to create a non-oxidising and non-sulphurous atmosphere in the heat-treatment of solids [annealing and casehardening of metals]. F. J. HARLOW and G. B. BENZ, Assrs. to PHILLIPS PETROLEUM Co. (U.S.P. 1,806,853, 26.5.31. Appl., 25.5.28).—Natural gas or liquid hydrocarbons of low b.p. are admitted to the annealing chamber to maintain a reducing atm. in the heattreatment of non-ferrous metals; they may also be used for carburising ferrous metals. A. R. POWELL.

Chemico-thermal welding process. B. BURNS, H. B. LEWIS, and R. M. OTIS, ASSTS. to HUGHES DEVELOP-MENT Co., LTD. (U.S.P. 1,806,738, 26.5.31. Appl., 2.1.29).—Thin strips of two dissimilar metals, e.g., Ni and Fe, may be welded together by heating in a closed chamber containing a small quantity of a halide or similar salt which forms a volatile compound with one of the metals, e.g., Fe, this compound being decomposed by the other metal. Suitable salts are  $\rm NH_4Br, NH_4I$ ,  $\rm NH_4Cl, FeCl_3$ . A. R. POWELL.

Preventing the solvent action of sulphuric acid on iron and steel. J. G. SCHMIDT, Assr. to E. F. HOUGHTON & CO. (U.S.P. 1,807,711, 2.6.31. Appl., 21.3.28. Renewed 23.4.30).—An inhibitor for  $H_2SO_4$ pickling baths comprises the reaction product of an arylthiccarbamide and an aldehyde, *e.g.*, the oily product obtained by prolonged heating under reflux of a mixture of CH<sub>2</sub>O solution and di-o-tolylthiocarbamide. A. R. POWELL.

Inhibitor [to prevent corrosion of iron and steel by phosphoric acid]. R. L. JENKINS, J. H. KARRH, and C. R. McCullough, Assrs. to SWANN RESEARCH, INC. (U.S.P. 1,809,041, 9.6.31. Appl., 3.2.30).— $H_3PO_4$ (75%) can be stored in Fe vessels if about 0.1% of furfuraldehyde and 0.003% of KI are dissolved in the acid. A. R. POWELL.

Preventing metal [iron or steel] from corroding. J. H. GRAVELL, ASST. to AMER. CHEM. PAINT CO. (U.S.P. 1,805,982, 19.5.31. Appl., 4.2.30).—Sheet Fe or steel is protected by coating it with a mixture of an org. phosphate, e.g.  $(NH_2Ph)_2HPO_4$ , and a metal oxide, e.g.,  $Fe_2O_3$ , which will combine with the  $H_3PO_4$  liberated by the decomp. of the phosphate. Varnishes or resins may be added to produce a paint-like mixture. The coated metal is finally heated at 230° for 30 min. to dry and bake the coating on to the metal. A preferred mixture comprises  $(NH_2Ph)_2HPO_4$  (6.44 lb.),  $Fe_2O_3$  (1.6 lb.), kerosene (6.87 gals.), stearine pitch (8.69 lb.), and gilsonite (9.81 lb.). A. R. POWELL.

[Inhibitor for] preventing the dissolution of metals [iron or steel] in pickling baths. L. B. SEBRELL, ASST. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,805,052, 12.5.31. Appl., 21.10.27).—Products obtained by treating aliphatic aldehydes with  $H_2S$ ,  $K_2S$ , or Na<sub>2</sub>S are claimed. Trithioformaldehyde is specifically mentioned. A. R. POWELL.

[Inhibitors for use in the] cleaning and preserving [pickling] of metal surfaces. (A—E) D. H. TOMP-KINS, (F) T. W. BARTRAM and D. H. TOMPKINS, Assr(s). to RUBBER SERVICE LABS. Co. (U.S.P. 1,808,184—8 and 1,808,200, 2.6.31. Appl., [A] 30.6.30, [B] 9.10.30, [C] 11.10.30, [D] 16.10.30, [E] 13.11.30, [F] 20.3.30. Renewed [F] 4.12.30).—Inhibitors for  $H_2SO_4$  pickling baths comprise the substances obtained by treating the reaction product of  $SO_2$  (0.5 mol.) with a COMe<sub>2</sub> solution of the condensation product of MeCHO (3 mols.) and NH<sub>2</sub>Ph (2 mols.) with (A) 75% H<sub>2</sub>SO<sub>4</sub>, (B) HCl, (C) H<sub>3</sub>PO<sub>4</sub>, (D) H<sub>2</sub>CrO<sub>4</sub> and (E) AcOH. In (F) a sulphonated aldehyde– NH<sub>2</sub>Ph condensation product is claimed.

A. R. POWELL.

Aluminothermic preparation of air-quenching steel. C. L. DELACHAUX (B.P. 361,390, 18.7.30. Fr., 6.1.30).—Steel containing  $0.3 \rightarrow 0.4\%$  C,  $3 \rightarrow 3.5\%$  Ni,  $0.8 \rightarrow 1.2\%$  Cr,  $0.3 \rightarrow 0.6\%$  Mn, < 0.5% Si, < 0.1% Ti, and 0.0075% Mo is prepared by igniting a mixture of 23.7% Al and 76.3% Fe<sub>3</sub>O<sub>4</sub> to which have been added Ni, Cr, Ti, and Mo, preferably in the form of ferro-alloy, and sufficient C, preferably as cast Fe grains (2-4% C), to bring the resulting steel to the required composition. When the reaction ceases a ferro-alloy with 75% Mn or Si or (Si + Al) is added to deoxidise the molten metal. A. R. POWELL.

Nitrification [nitrogen case-hardening of steel]. A. B. KINZEL, ASST. to ELECTRO METALLURG. Co. (U.S.P. 1,808,355, 2.6.31. Appl., 11.5.29).—Fe alloys, preferably containing up to 1% Al, are embedded in MgO, SiO<sub>2</sub>, asbestos, or C in a closed container which is heated at  $460-580^{\circ}$  while NH<sub>3</sub> is passed into it. A. R. POWELL.

Manufacture of rolling-mill piercing point, plug, and guide, and method of using such points. H. F. TIELKE (U.S.P. 1,807,453—4, 26.5.31. Appl., 23.8.29).—(A) The point is made of a steel containing 0.75—1.5% C, 16—18% Cr, and 2—4% Co, with or without 0.4—2% Ni and/or up to 2% W. After casting, the steel is heated for 5 min. at 1250°, air-cooled, reheated at 760—880°, quenched in oil, and soaked at 650° for 6 hr. (B) The point is inserted in the pass of the rolling mill after preheating to 500° and kept there until the temp. rises well above 650°, when it is removed and cooled to 500° while immersed in CaO. The life of the point in piercing sheet metal is thus considerably prolonged.

A. R. POWELL.

Heat treatment of rails or other massive steel bodies. BETHLEHEM STEEL Co., Assees of E. F. KENNEY (B.P. 362,439, 4.6.30. U.S., 12.7.29).—A rail is quenched to below the crit. range and then soaked in a heat-conducting medium at a temp. above the blue range. Thus immersion in water for 30 sec. may be followed immediately by immersing the rail for 4—5 min. in a Pb bath maintained at 540°, and then annealing at the same temp. for 1—1 $\frac{1}{2}$  hr. [Stat. ref.] C. A. KING.

Manufacture of semi-finished or finished [rolled] steel products. TIMKEN HOLDING & DEVELOPMENT Co. (B.P. 362,715, 5.1.31. U.S., 17.5.30).—Scrap metal is refined in an electric furnace and poured into a rotating annular mould of a centrifugal casting machine. The severed ring is straightened and rolled, and it is claimed that 95% of the metal tapped is sound. C. A. KING.

Surfacing of refractory bodies [e.g., cylinder linings]. DOHERTY RESEARCH CO., Assees. of J. D. MORGAN (B.P. 361,964, 28.8.30. U.S., 25.10.29).—A film of metal is electrodeposited on to the inner face of an engine lining of refractory insulating material. For higher working temp. Ni–Cr is preferred, and the film may be formed by depositing a layer of Cr between two layers of Ni and then heating the lining until the metals alloy. C. A. KING.

Purification of metals [steel]. Production of a stainless chromium [steel] alloy. C. W. CARMAN, Assr. to STAINLESS STEEL CORP. (U.S.P. 1,809,436-7, 9.6.31. Appl., [A] 26.2.27, [B] 15.9.27).-(A) Molten steel in an electric furnace is purified by blowing it with  $O_2$  and adding a mixture of granulated Al (4 lb./ton) and PdO (0.3 oz./ton). (B) Steel scrap is melted in an electric furnace, refined in the usual way, and, after removal of slag, treated with high-C ferrochromium and blown with  $O_2$  at low pressure. Ni is then added and the alloy blown with  $O_2$  under a higher pressure while Al and PdO are sprinkled over the surface. Castings of this steel are claimed to be resistant to corrosion without heat-treatment. A. R. POWELL.

Manufacture of a magnetic product [iron-nickel alloy]. T. D. YENSEN, ASST. to WESTINGHOUSE ELECTRIC & MANUFG. Co. (U.S.P. 1,807,021, 26.5.31. Appl., 29.3.24).—An alloy of 60—40% Fe and 40—60% Ni when rolled into sheet 0.014 in. thick, annealed for 2—8 hr. at 1000° in H<sub>2</sub>, and allowed to cool slowly has a max. permeability of >40,000 and a hysteresis loss of <400 ergs per c.c. per cycle for B = 10,000 gauss.

A. R. POWELL.

Iron-nickel-copper alloy. N. B. PILLING, Assr. to INTERNAT. NICKEL Co., INC. (U.S.P. 1,805,049, 12.5.31. Appl., 16.11.28).—Alloys containing 26-74% Fe, 60-25% Ni, 1-26% Cu, and 0.4-2% C are claimed. The Ni is at least 1.85 times the Cu content to ensure homogeneity, and when Ni : (Ni + Fe) is < 0.27: 1 the C% is > 6(0.27 - % Ni/[% Ni + % Fe]). The alloys are soft, malleable, and resistant to corrosion.

A. R. POWELL.

Steel alloys [with a high creep limit]. F. KRUPP A.-G. (B.P. 361,397, 22,7.30. Ger., 1.8.29).—A high creep limit is imparted to non-austenitic steel alloys containing <0.19% C, <7.5% Cr, and <1.9% Si by including in the alloy 0.3—2% of V and/or Mo, the sum of these two constituents being at least twice the C content, so as to leave a substantial amount of them dissolved in the ferrite after the C has been completely converted into V and/or Mo carbides. A. R. POWELL.

Stain-resisting wrought [manganese-chromium steel] articles. ELECTRO METALLURGICAL CO., Assees. of F. M. BECKET (B.P. 361,916, 22,8.30. U.S., 14.3.30). —Steel containing  $\langle 0.3\%$  C, 16—22% Cr, >5 (8—12)% Mn, and substantially free from Ni is claimed. Resistance to staining is induced simply by severely cold-working the alloy. [Stat. ref.] A. R. POWELL.

[Steel apparatus for] carrying out reactions in the presence of hydrogen. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 361,856, 25.7.30).—In the catalytic hydrogenation of crude petroleum the parts which come into contact with oil at above 300° are made of steel free from Ni and containing 3—18 (6)% Cr, 0.2—3 (0.5)% Mo, about 0.3% V, and 0.1% C. A. R. POWELL.

Corrosion-resistant aluminium alloy article. F. C. FRARY, Assr. to ALUMINUM Co. of AMERICA (U.S.P. 1,805,448, 12.5.31. Appl., 30.11.29).—Sheet composed of an alloy of Al with  $3-5\cdot5\%$  Cu,  $0\cdot5\%$  Mg, and  $0\cdot6\%$  Mn is coated with a thinner sheet of an alloy of Al with  $0\cdot7\%$  Mg and  $0\cdot9\%$  Si, and the composite material is quenched from 500° and aged at room temp., whereby the first alloy age-hardens spontaneously whereas the second is unaffected. The coated alloy has a resistance to corrosion in the quenched state nearly equal to, and a tenacity much greater than, that of pure Al. A. R. POWELL.

Production of beryllium and other metals. H. S. COOPER, Assr. to BERYLLIUM DEVELOPMENT CORP. (U.S.P. 1,805,567, 19.5.31. Appl., 6.12.24).— BeO is converted into BeCl<sub>2</sub> by heating at 1000° in a current of Cl<sub>2</sub> and CCl<sub>4</sub>, and a mixture of the BeCl<sub>2</sub> and NaCl is electrolysed at 825° in a Cr-Fe alloy vessel which forms the cathode and is filled with H<sub>2</sub> during the electrolysis, so as to keep a reducing atm. and to combine with the Cl<sub>2</sub> evolved. Pure Be is deposited as spangles on the sides of the vessel; these are detached, washed, dried, and pressed into a bar, which is sintered at 1200—1450° under a layer of fused NaCl and/or BaCl<sub>2</sub>. A. R. POWELL.

Production of magnesium from magnesium oxide. HIRSCH, KUPFER- U. MESSINGWERKE A.-G. (B.P. 361,957, 26.8.30. Ger., 26.8.29).—A mixture of MgO, W powder, and 50% of the C required to reduce the MgO to Mg is heated at a temp. sufficient to reduce the MgO and sublime the resulting Mg. The lower W oxides formed are reduced by H<sub>2</sub> to W powder for use again. A. R. POWELL.

Amalgamator. R. LUCKENBACH (U.S.P. 1,807,162, 26.5.31. Appl., 23.8.28).—The apparatus comprises a centrifugal machine containing an inner chamber provided with a spreading disc at some distance from the bottom and with a conical cover which serves as a feed hopper. Ore pulp and activated Hg are fed into the chamber on to the disc, which distributes the mixture against the sides, from which it falls through a central hole in the chamber on to the flat distributor disc in the bottom of the centrifuge basket, and from thence

is thrown against the inner walls of the basket. The discharge from the top of these walls is made to flow downwards over a stationary amalgamated plate into an annular chamber, from which it is thrown against a vertical column of Hg maintained on the walls by the centrifugal force. A. R. POWELL.

[Promoter for flotation] concentration of ores, minerals, and the like. J. L. STEVENS, ASST. to RACONITE CHEM. Co. (U.S.P. 1,805,855, 19.5.31. Appl., 15.1.29).—For the flotation of Cu ores substances of the type  $(RO \cdot CS \cdot S)_2S_2$ , formed by the interaction of K or Na xanthate with  $S_2Cl_2$ , are claimed as promoters. Flotation is effected in CaO circuits with pine oil as a frother; the reagent has a selective action on Cu sulphide minerals. A. R. POWELL.

**Production of tin from its ores.** H. E. COLEY (B.P. 361,402, 18.8.30).—The ore is heated at 950— 1000° in a reducing atm. and a jet of crude oil containing 5—7% S is sprayed into the furnace from a water-cooled pipe so that decomp. of the oil occurs only when it comes into contact with the hot ore. The  $\text{SnO}_2$  is thus reduced to Sn which is immediately sulphidised by the S in the oil and volatilised as SnS together with any As which may be present. Extraction of the mixed sulphides with HCl affords a solution of  $\text{SnCl}_2$  free from As from which pure  $\text{SnO}_2$  may be obtained by treatment with an alkali. A. R. POWELL.

Recovery of manganese [from low-grade ores]. C. S. VADNER (U.S.P. 1,807,642, 2.6.31. Appl., 25.6.28). —Finely-divided, oxidised Mn minerals are agitated with a SO<sub>2</sub> solution and the resulting impure solution of MnSO<sub>4</sub> and MnS<sub>2</sub>O<sub>6</sub> is purified by agitation with further quantities of MnO<sub>2</sub> and air to oxidise and ppt. the Fe, As, P, etc. The purified solution is evaporated to dryness and the residue heated at 200° to convert MnS<sub>2</sub>O<sub>6</sub> into MnSO<sub>4</sub> and SO<sub>2</sub> which is used again.

A. R. POWELL.

Copper extraction process. W. E. GREENAWALT (U.S.P. 1,808,547, 2.6.31. Appl., 7.1.27. Renewed 28.10.30).—The ore is separated by gravity into (A) a high-grade (50 %Cu) and (B) a low-grade (5-10% Cu) concentrate and (C) a tailing. Product A is roasted to sulphate and leached first with H<sub>2</sub>O and then with  $H_2SO_4$ , and the residue is smelted to obtain crude Cu containing Au and Ag. Product B is roasted and leached first with H<sub>2</sub>O and then with the acid leachings of A and the solution is electrolysed with insol. anodes to obtain pure Cu and an acid solution which is returned to the leaching vats. The residue from leaching B is cyanided to recover Au and Ag and the crude metals recovered from the cyanide solution are added to the smelting charge together with any scrap Cu produced in the electrolytic tanks. The crude Cu from smelting is refined in the pure  $CuSO_4$  obtained in leaching A with H<sub>2</sub>O. A. R. POWELL.

Production of metal [copper] powders. E. FITZ-PATRICK, C. W. CLARK, and P. A. TIERNAN, Assrs. to NICHOLS COPPER CO. (U.S.P. 1,804,924, 12.5.31. Appl., 20.6.28).—CuSO<sub>4</sub> solution is electrolysed at a high c.d. between a Cu-sheet cathode and a Cu-rod anode so that the deposited Cu will have a loose fluffy structure and be readily detachable from the cathode. The deposit is washed thoroughly by decantation without exposure to the air, and dried in vac. at 55°. A. R. POWELL.

Manufacture of [copper] alloys suitable for production of metal fittings which come into contact with superheated steam. BERNDORFER METALL-WARENFABR. A. KRUPP A.-G. (B.P. 361,727, 3.2.31. Austr., 3.2.30).—The alloys comprise Cu with about 2% Fe and 2-4.5% Si, all of the Si and part of the Fe being introduced into the molten Cu by the addition of ferrosilicon. A. R. POWELL.

Manufacture of copper-plated shingles. F. MUSCHLER (U.S.P. 1,805,920, 19.5.31. Appl., 7.11.27).— Wooden shingles or paper felts are impregnated with a waterproofing material comprising molten asphalt and mineral oil or tar, then coated on one side and round the edges with a 1:1 mixture of  $Pb_3O_4$  and shellac made into an emulsion with EtOH, and finally, after drying, sprayed on the same side with a 1:1:3 mixture of lacquer, powdered Sn, and powdered Cu suspended in 6 times its wt. of thinners. A finishing coat of Cu is then deposited electrolytically on the treated surface, this coat being eventually tinted as desired.

A. R. POWELL.

Sulphuric acid fuming and decomposition process for recovery of zinc. H. H. MAYER and R. G. LA MOTTE (U.S.P. 1,808,929, 9.6.31. Appl., 29.9.26).—Oxidised ores, slags, or metallurgical products containing Zn are finely ground, mixed with sufficient  $H_2SO_4$  to convert the whole of their metal content into sulphate, and heated to expel excess of acid and to dehydrate SiO<sub>2</sub>. The dry charge is then heated more strongly to decompose Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> and other unstable sulphates and finally at a term, at which part of the ZnSO<sub>4</sub> decomposes. The cold mass is then extracted with very dil.  $H_2SO_4$  to recover ZnSO<sub>4</sub> solution relatively free from impurities. A. R. POWELL.

Preparation of colloidal metal [lead for antiknock oil fuels]. G. ALLEMAN, ASST. to SUN OIL CO. (U.S.P. 1,805,199, 12.5.31. Appl., 11.12.29).—Amyl derivatives of Pb are decomposed by heating above  $188^{\circ}$ , preferably when in solution in Ph<sub>2</sub> or in aromatic or aliphatic hydrocarbon which does not coagulate the colloidal Pb. A. R. POWELL.

**Production of pulverised lead.** Y. TAKATA (B.P. 362,129, 13.11.30).—Molten Pb is sprayed by means of compressed air against the blades of a rapidly revolving fan in a closed chamber in such a way that the temp. of the Pb is just below the m.p. at the moment of impact, so that the fan breaks up the particles when they are in their most brittle condition. A. R. POWELL.

[Lead] bearing alloy. J. V. O. PALM and E. C. KNUTH, Assrs. to CLEVELAND GRAPHITE BRONZE CO. (U.S.P. 1,807,670, 2.6.31. Appl., 19.12.28).—The alloy contains 82% Pb, 7.5% Cd, 7.5% Sb, and 3% Zn; it retains its hardness to a higher temp. than do the common Babbitt alloys. A. R. POWELL,

[Lead] bearing metal. R. J. SHOEMAKER, Assr. to S. &. T. METAL CO. (U.S.P. 1,808,793, 9.6.31. Appl., 2.8.28).—Bearing metal with a low coeff. of friction comprises Pb with 0.3-1 (0.5)% Cu, 0.5-2 (1)% Sn,

0·1-0·5 (0·25)% Hg, 0·02-0·1 (0·05)% Al, and 0·02-0·22% Li, K, or Mg (0·075% Mg). A. R. Powell.

Lead-antimony alloy. P. KEMP (U.S.P. 1,807,788, 2.6.31. Appl., 30.9.29. Austr., 27.7.29).—A hard Pb alloy contains up to 10% Sb, 0.2-2% As, and 0.1-2% Sn, the Sn being about one fifth of the Sb content.

A. R. POWELL.

[Lead] alloy metal for bearings. W. MATHESIUS and M. W. NEUFELD (U.S.P. 1,804,883, 12.5.31. Appl., 24.2.27. Ger., 17.5.26).—The alloy comprises Pb with 0.6 - 0.65% Na, 0.75 - 1% Ca, 0.2 - 0.25% Mg, and 0.1% Al, the Mg being 0.2 - 0.25 times the Ca content and the Al about half the Mg content. The alloy has a Brinell hardness > 40 and can be heated above 600° without loss of alloying constituents by oxidation.

A. R. POWELL.

Producing metallic articles [of tungsten]. C. L. GEBAUER, ASST. to OHIO INSTRUMENT MANUFG. Co. (U.S.P. 1,809,780, 9.6.31. Appl., 4.2.29).—W or other metal with a m.p. above 2000° is intimately mixed with up to 50% of Cu or other metal which has a m.p. well below 2000°, and the mixture is pressed into shape, heated in  $H_2$  until compacted, and forged into the desired final shape. A. R. POWELL.

[Tungsten-molybdenum carbide] tool alloy. P. SCHWARZKOPF, and METALLWERK PLANSEE G.M.B.H. (B.P. 361,363, 16.5.30. Ger., 16.5.29).—Hard tool alloys comprise a mixture of 50—70 (63)% WC, 40—20 (27)% Mo<sub>2</sub>C, and 10% Co. The constituents are ground together to a fine powder which is pressed into bars and sintered at 1400—1600° until the Mo<sub>2</sub>C and WC have formed a hard solid solution embedded in a eutectiferous Co cement. Sintering is preferably effected in a highfrequency induction furnace in vac. A. R. POWELL.

Manufacture of metal [thoriated molybdenum] having high emissivity. C. G. FINK (U.S.P. 1,808,889, 9.6.31. Appl., 21.6.24).—A solution of  $(NH_4)_2MoO_4$ (1 kg.), Th $(NO_3)_4$  (75 g), NaCl (10 g.), and CdCl<sub>2</sub> (10—15 g.) is evaporated to dryness and the residue heated to expel H<sub>2</sub>O of hydration. The dry mass is pulverised and reduced in H<sub>2</sub> at 1200°, the resulting Mo powder is pressed into bars, which are sintered in H<sub>2</sub> at just below the m.p. of Mo, swaged, and drawn into wire, and the wire is heated electrically to incandescence in dry H<sub>2</sub> saturated with vapours of NH<sub>2</sub>Ph. A. R. POWELL.

Preparation of alloys of precious metals [gold or silver] capable of being age-hardened. DEUTS. GOLD- U. SILBER-SCHEIDEANSTALT VORM. ROESSLER (B.P. 361,893, 19.8.30. Ger., 4.9.29).—Alloys of Au and/or Ag with Pt, Pd, or Ir within the solid solution range are rendered capable of being age-hardened by the addition of 0.02-5% of a metal or metals of the Fe group and/or 1-8% of Zn, Cd, or Cr. *E.g.*, an alloy of  $89\cdot87\%$  Au,  $9\cdot92\%$  Pd, and  $0\cdot21\%$  Fe has a hardness of 40 after quenching from 900° and of 120 after subsequent annealing at 550° for 2 hr. Under similar conditions an alloy of 10% Pt,  $88\cdot5\%$  Au, and  $1\cdot5\%$  Zn can be given a max. hardness of 170. A. R. POWELL.

Separation of platinum from the other precious metals. G. A. L. R. Collard, Assr. to Comptoir LYON - ALEMAND (U.S.P. 1,808,594, 2.6.31. Appl., 28.1.28. Fr., 4.2.27).—Jeweller's waste containing Au, Ag, and Pt is smelted with PbO, matte, and fluxes together with an excess of Cu, e.g., 1% on the wt. of Pb in the charge, the slag and matte are removed, and the Pb is allowed to cool, whereby a pasty mass of Cu containing Pt and an excess of Pb separates above the fluid Pb, which latter retains most of the Au and Ag. The paste is skimmed off and cupelled with more Pb to obtain a Au-Ag-Pt bullion with a higher Pt: (Au + Ag) ratio than that in the original material.

A. R. POWELL.

[Palladium] dental alloy for swaged dentures. F. MAULEN, ASST. to BAKER & CO., INC. (U.S.P. 1,807,068, 26.5.31. Appl., 18.11.29).—An alloy of 99—95% Pd and 1—5% Rh in claimed. A. R. POWELL.

**Production of ore coke.** METALLGES. A.-G. (B.P. 362,796, 9.3.31. Ger., 2.4.30).—Finely-divided metallic ore is intimately mixed with a coking coal and the mixture is damped and placed on the grate of a Dwight–Lloyd or other sintering apparatus. The charge is superficially ignited and is then subjected to an "up" or "down" blast with gases low in  $O_2$ , e.g., hot waste gases from the process itself, until it is coked. An air blast may be used, in which case a layer of coal is interposed between the blast and the charge. A. B. MANNING.

Electroplating. J. W. HUGHES, Assr. to BUDD WHEEL CO. (U.S.P. 1,807,157, 26.5.31. Appl., 23.11.26). —In the plating of small articles a tank is used containing a number of article carriers which are alternately made anodes and cathodes. In each plating operation alternate carriers are loaded with the articles to be plated, while the intermediate carriers are used as anodes so that the metal deposited on them in the preceding operation when they acted as cathodes is stripped off again. A. R. POWELL.

Pickling apparatus [for metals]. PHOSPHOR BRONZE CO., LTD., and Z. PAVNE (B.P. 363,928, 1.4.31).

Coating metal pipe surfaces [with cement etc.]. G. B. ELLIS. From NAT. TUBE Co. (B.P. 363,703, 16.9.30).

Rotary furnace.—See I. Values from cyanides. —See VII. Al paint for pipes.—See XIII.

#### XI.—ELECTROTECHNICS.

[Electrical] automatic methods of concentration control. W. N. GREER (Pulp and Paper Mag., 1931, 31, 1374—1376).—An automatic conductivity control is described which diverts black-liquor diffuser washings from evaporators to waste in soda- or sulphate-pulp mills when the concn. of Na<sub>2</sub>O falls below a predetermined val. An automatic  $p_{\rm H}$  control of alum dosage, suitable for industrial waters, is also described.

N. H. HARTSHORNE.

Electrolyte pastes. Application of ultra-filtration in analytical practice. H. GRIMM (Chem.-Ztg., 1932, 56, 31—32).—Electrolytes in colloidal pastes (e.g., in dry batteries) are best determined after ultrafiltration. E. S. HEDGES.

Analysis in paper mills.—See V. Arc-welding. Sheet Fe. Analysis of Cr-V steels. Sr-Cd alloys.

Plating on Zn. Surface-coating Al. Gas purification.—See X.

See also A., Jan., 8, Photo-cells. 37, Electromagnetic micro-balance. 60, Electrochemical oxidation of *p*-toluoquinone.

#### PATENTS.

Electrodes for electrolytic apparatus. KABUSHIKI KAISHA HITACHI SEISAKUSHO (B.P. 363,289, 9.6.31. Jap., 9.6.30).—Welds between electrodes and conducting terminals are arranged within gas pockets below the level of electrolyte. J. S. G. THOMAS.

Electrodes for electrolytic processes. E. and O. CONRADTY (C. CONRADTY) (B.P. 362,755—6, 3.2.31. Ger., [A] 23.7.30).—(A) Vertical electrodes of trapezoidal or rhomboidal cross-section are arranged with the sides of adjacent anode members completely overlapping one another so that an uninterrupted anode surface is presented to the cathode surface. (B) Interstices at joints between plates and connecting rods, bars, etc. are filled with plastic, fusible material, e.g., a mixture of wax and powdered graphite, which is impenetrable to, and insol. in, the electrolyte. J. S. G. THOMAS.

Electrodes for use in electrolysis. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 362,988, 12.9.30).— Single electrodes are clamped together to form a larger unit by a device arranged out of contact with electrolyte on the upper side of the electrode. J. S. G. THOMAS.

Carrying out electrolytic processes, more particularly those with decomposable end-products, *e.g.*, manufacture of persulphuric acid or its salts by electrolysis. OESTERR. CHEM. WERKE G.M.B.H. (B.P. 362,579, 4.10.30. Austr., 20.2.30).—A thin stream of anolyte flows with high velocity through the anode space containing a perforated anode, while the catholyte flows slowly through the cathode space which is separated from the anode space by a diaphragm connected to the anode or arranged close thereto. J. S. G. THOMAS.

Electrolytic process for manufacture of chemical compounds. A. L. CLARIANA (B.P. 363,054, 27.10.30. Spain, 24.1.30).—Spongy metallic cathodes, *e.g.*, of Pb, are alternately oxidised by exposure to air and reduced during electrolysis. The process is applicable, *e.g.*, to the manufacture of Pb(OH)<sub>2</sub>, using Pb electrodes and an electrolyte composed of NaClO<sub>3</sub>. J. S. G. THOMAS.

Gaseous electric-discharge devices. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of T. E. FOULKE (B.P. 362,346-7, [A] 22.7.30, [B] 23.7.30. U.S., [A, B] 27.7.29).—(A) An electrode coated with an alkali or alkaline-earth compound which is decomposable to the oxide, e.g., BaCO<sub>3</sub>, is heated *in vacuo* to drive off occluded gas, and after reduction of the alkali compound to oxide is bombarded at high temp. by a high-voltage discharge in a gas until the surface of oxide is reduced to metal. (B) One of the electrodes consists of an open metal cone, e.g., of Ni, the interior surface of which has been treated as in (A). J. S. G. THOMAS.

[Electrodes for] photoelectric cells. N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 362,838, 5.8.31. Holl., 30.9.30).—One or more electrodes are coated, at least partly, with material, *e.g.*, ZnS or ZnSiO<sub>8</sub>, which fluoresces when bombarded by electrons emitted by the photoelectric electrode. J. S. G. THOMAS.

Manufacture of electron emission tubes. A. FEVRE (B.P. 362,984, 12.9.30).—The foot of the bulb is coated internally with material, e.g.,  $BaO_2$ , which under the influence of a high-tension electric discharge between the leads and foot of the bulb reacts with any metal deposited thereat during process of manufacture, to form a non-conducting substance. J. S. G. THOMAS.

Electrical dehydrators for treatment of petroleum emulsions. PETROLEUM RECTIFYING CO. OF CALIFORNIA, Assees. of H. C. and W. O. EDDY, H. F. FISHER, S. G. GASSAWAY, F. W. HARRIS, L. LAWRASON, W. F. VAN LOENEN, J. T. WORTHINGTON, F. D. MAHONE, and C. W. GIRVIN (U.S.P. 1,796,750, 17.3.31, 1,807,833, 2.6.31, and 1,838,374—6, 1,838,386, 1,838,822, 1,838,828, 1,838,847—50, 1,838,889—90, 1,838,909—34, 1,838,937— 8, and 1,838,976—80, 29.12.31).—Various forms of apparatus are described.

Apparatus for electrical precipitation of suspended particles from gaseous fluids. Lodge-COTTRELL, LTD. From METALLGES. A.-G. (B.P. 363,298, 24.7.31).—The bent surfaces of zig-zag collecting electrodes are arranged parallel to one another so that the peaks of one project into the depressions of the next, and catch spaces at the bends are provided with catchstrips or troughs. J. S. G. THOMAS.

Metallic-vapour rectifiers. INTERNAT. GEN. ELECTRIC Co., INC., Assees of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 363,976, 21.8.31. Ger., 21.8.30).

[Water-cooled] mercury arc rectifiers. BRIT. THOMSON-HOUSTON CO., LTD., and F. P. WHITAKER (B.P. 363,984, 16.7.30).

Vacuum electric tube devices. E. Y. ROBINSON, and Associated Electrical Industries, Ltd. (B.P. 364,098, 26.9.30).

[Four-electrode] electric-discharge tubes. Egyesült Izzólampá és Villamossági Résvénytársaság (B.P. 364,411, 28.2.31. Hung., 17.10.30).

Electric-discharge tubes for emission of [ultraviolet] rays. N. V. Philips' GLOEILAMPENFABR. (B.P. 363,914, 16.3.31. Holl., 4.4.30).

X-Ray installations. N. V. PHILIPS' GLOEILAMPEN-FABR. (B.P. 363,595 and 363,602, [A] 23.4.31, [B] 9.5.31. Holl., [A] 30.4.30, [B] 21.5.30. Addn. [A] to B.P. 293,731).

[Mounting of] ozonising apparatus. BAMAG-MEGUIN A.-G. (B.P. 362,827, 11.5.31. Ger., 4.5.31).

Electric incandescence lamps [with heat-insulated cap], BRIT. ELECTRICAL & ALLIED INDUSTRIES RESEARCH ASSOC., H. G. TAYLOR, W. LETHERSICH, and E. B. WEDMORE (B.P. 363,775, 6.11.30).

[Conveyor for] manufacture of electric incandescence lamps or like articles. Gen. Electric Co., Ltd., Assees. of Patent Treuhand Ges. F. Elektr. Glühlampen M.B.H. (B.P. 364,501, 4.6.31. Ger., 17.7.30).

Electric insulated wires and cables [with ribbed india-rubber covering]. BRIT. INSULATED CABLES, LTD., W. WHITELEY, and C. WILKINSON (B.P. 363,440, 3.10.30). Metering gaseous fluids.—See I. Cylinder linings. Cu extraction. Cu powder. Be etc. Fe-Ni. Thoriated Mo. Electroplating. Shingles.—See X. X-Ray photographs.—See XXI.

# XII.—FATS; OILS; WAXES.

Circulating [fat-]rendering process. O. H. WURSTER (Soap, 1931, 7, No. 12, 79–82, 93).—The comminuted material is pumped up through vertical tubes (external diam. 4 in.) heated by exhaust steam, passes into a large flash chamber to remove vapour under vac. (up to 28 in. in the condenser), is recirculated (approx. every 3 min.) until fully rendered (2 hr.), and is then discharged into settling tanks; the max. temp. of the charge averages  $85^{\circ}$  ( $\geq 94^{\circ}$ ). The product is a high-quality fat. E. LEWKOWITSCH.

Ethanolamine soaps. P. I. SMITH (Soap, 1931, 7, No. 12, 27–29).—A review of properties and industrial applications (as emulsifier for fat-liquoring, for drycleaning, etc.). E. LEWKOWITSCH.

Isolation of fatty acids of unknown fat or oil. S. R. JAMELLIER (Chemist Analyst, 1931, 20, No. 5, 4-5). -The fat (100 g.) is saponified with KOH in EtOH; hot  $H_2O$  (2 litres) is added and the solution is acidified with H<sub>o</sub>SO<sub>4</sub>. The fatty acids are collected, drained, refluxed with Et<sub>2</sub>O, and filtered through paper which has been treated with warm Et<sub>2</sub>O. The Et<sub>2</sub>O is removed and the wt., m.p., acid val., I val., sp. rotation, and hydroxy-acid content of the acids are determined after drying in a vac. over paraffin. An aliquot part of the acids is dissolved in hot 95% EtOH, crystals which separate on cooling being identified. The motherliquor is treated with a little H<sub>2</sub>O, heated 5°, and cooled, the crystals being identified. The remaining acids are pptd. with H<sub>2</sub>O from Et<sub>2</sub>O solution and pressed to cake and oil. Acids in the oil phase are separated by way of the Pb soaps and fractional distillation.

CHEMICAL ABSTRACTS.

Influence of some antioxidants on soaps of the higher unsaturated fatty acids. K. OGAWA (J. Soc. Chem. Ind., Japan, 1931, 34, 449—451 B).—The effects of 11 org. antioxidants on the keeping properties of soaps prepared from cotton-seed, soya-bean, linseed, and chrysalis oils, and from oleic acid were studied; estimates of the extent of oxidation by determinations of the I vals. and  $n_D$  of the fatty acids, colour, hydrolysis, and scouring properties of the soaps and "oxidised acids" were in good agreement. Thymol, phenol,  $\beta$ - and  $\alpha$ -naphthols were the most effective antioxidants.

#### E. LEWKOWITSCH.

Relation of m.p. and f.p. of completely hydrogenated waxes, oils, and their fatty acids. S. UENO, G. INAGAKI, and H. TSUCHIKAWA (J. SOC. Chem. Ind., Japan, 1931, 34, 445—447 B).—Several samples of fully hydrogenated (I vals. 1—16) sperm oil (A), herring oil (B), sardine oil (C) (m.p. of the oils about  $52^{\circ}$ ,  $56^{\circ}$ , and 52— $57^{\circ}$ , respectively), and the fatty acids derived from them were examined. For (A) the m.p. and f.p. were the same and about 7—8° higher than the f.p. (titer) of the corresponding fatty acids; the m.p. of the acids was 1— $2^{\circ}$ higher than the f.p. The f.p. of (B) was 6—7° lower than the m.p., whilst the m.p. and f.p. of the fatty acids differed only by  $0.5-0.1^{\circ}$ . The m.p. of the hardened oil was about  $1.7^{\circ}$  higher than its titer. In the case of (c), the m.p. and titer differed by  $3-3.5^{\circ}$  when the titer was  $53.5-54^{\circ}$ , and by  $2.3-3.5^{\circ}$  when the titer was 50°. E. LEWKOWITSCH.

Purification of linseed oil. EBERT (Chem.-Ztg., 1931, 55, 983).—Slimy substances can be removed from linseed oil by stirring the oil with fuller's earth at 90° for about 30 min. and filtering. E. S. HEDGES.

Is washing of neutralised oils necessary? H. H. LEMMEL (Allgem. Oel- u. Fett-Ztg., 1931, 28, 394-395). --In general (except when the oil is required for hardening etc.), washing after neutralisation may be omitted if the oil is allowed to settle for about 6 hr. and strained through a coarse cloth before bleaching and deodorisation.

E. LEWKOWITSCH.

Improvement of linseed oil drying. Drying properties of the most unsaturated glycerides of linseed oils. A. EIBNER, F. BROSEL, and V. JUNG (Chem. Umschau, 1931, 38, 325-331) .- The activation of firseed oils on exposure to light is compared with the observations of Bauer (B., 1931, 551) on the insolation of linseed oil: isomerisation of  $\beta$ - to  $\alpha$ -acids is suggested as an explanation. Earlier work on the isolation of bromoglycerides from linseed oil has been revised. The m.p. of di-a-linolenic-mono-oleic bromide (Widenmayer, 72°) is 78-80° (corr.), and the presence of oleic acid was confirmed; the m.p. agrees with that given by Suzuki and Yokoyama (A., 1928, 152) for a product termed by them bromodi-a-linoleo-monolinolenic glyceride (the Br content is the same). Brosel's bromide (B., 1928, 761), m.p. 143-149°, could not be produced and is considered to have been probably a mixture of bromodi-a-linolenic-a-linoleic glyceride (m.p. 155-156°) and the bromide, m.p. 118-120°, now separated, which may be of di-a-linolenic-B-linoleic glyceride (Brosel, Suzuki) or of di-a-linolenic-B-linolenic glyceride. A fourth bromoglyceride, insol. in EtOH, was also isolated. The drying properties of the unsaturated glycerides recovered from these bromo-compounds were examined. Their films dried more rapidly, but yellowed more, were more sol. in EtOH and much more hydrophilic than those of linseed oil, sintered or melted on heating, and in general behaved like very fresh linseed oil films. On ageing (2 months) the films improved slightly in behaviour.

# E. LEWKOWITSCH.

Physical and chemical characteristics of drying oils under various heat treatments. F. R. MESSER (Amer. Paint & Varnish Manufrs.' Assoc., Dec., 1931, Circ. No. 404, 630—634).—100-gal. batches of linseed oil were heated for 1, 2, 3, 4, and 5 hr. at 274°, 288°, 302°, and 316° and of tung oil for  $\frac{1}{2}$ , 1,  $1\frac{1}{2}$ , and 2 hr. at 204°. Colour, viscosity, sp. gr., and the acid, I, and sap. vals. for all samples are tabulated. No conclusions are drawn and the work is proceeding with other varnish oils.

S. S. WOOLF.

Absorption of oxygen by unsaturated [fatty] oils. II. Influence of catalysts. S. UENO and T. SAIDA (J. Soc. Chem. Ind., Japan, 1931, 34, 448—449 B; cf. B., 1931, 551).—The vol. of  $O_2$  absorbed by "small herring" oil dissolved in AcOH was measured directly; V linoleate was the most powerful oxidation catalyst.

Co linoleate had some action, whilst Ni, Fe, Mn, Pb, and Cu linoleates had very much smaller effects.

E. LEWKOWITSCH.

**Po-yoak oil.** J. VAN LOON (Farben-Ztg., 1932, 37, 483—484).—This oil (from *Parinarium Sherbroense*) compares closely with oiticica oil, a sample of which was tested alongside it. Po-yoak oil is viscous and changes to a buttery mass. It has  $d_4^{78}$  0.925, acid val. 1.35, sap. val. 192, true I val. 232,  $n_{10}^{16}$  (supercooled) 1.5209,  $n_{10}^{70}$  1.5018, and contains unsaponifiable matter 0.59%, glycerol residue 4.30%, total fatty acids 93.5% (11.8% saturated, 79.55% unsaturated, 2.15% oxidised insol. acids), volatile matter 1.61%. Its use as a tung oil substitute is discussed. S. S. WOOLF.

Utilisation of castor oil. E. ANDRÉ (Bull. Mat. Grasses, 1931, 50, 247—251).—Pharmaceutical castor oil is the best for lubrication, but a good grade is obtained by steaming first pressings to coagulate the proteins. The second pressings carried out in the presence of tepid water contains up to 7.8% of fatty acids and can be also used for lubrication purposes and for the prep. of sulphonated oils. T. A. SMITH.

Lubrication with olive oil.—See I. Vegetable oils as fuel.—See II. Soya bean [as oil seed].—See XIX.

See also A., Jan., 43, Pure Na ricinoleate. Composition of linseed oil. 93, Decomposition of fats by micro-organisms. 97, Vitamin-A in cod-liver oil. 99, Plant waxes. 101, Fatty acids from oil of *Cantharis*.

#### PATENTS.

Emulsions.—See I. Adhesive masses.—See II. Alcohols from esters. Wetting etc. agents.—See III.

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

Priming-coat reductions for painting new wood surfaces. J. C. GEHANT (Amer. Paint & Varnish Manufrs.' Assoc., Dec., 1931, Circ. No. 404, 596—618).— A preliminary account is given of a series of exposures begun in Aug., 1931, of two- and three-coat work on 4 species of wood, the priming coats being based on white lead, white lead–ZnO–inert, and  $\text{TiO}_2$ –ZnO–inert, respectively, each thinned with various proportions of linseed oil and turpentine; the second and third coats were normal mixtures based on the same three pigment combinations. Other paints were included for comparison, e.g., primers based on synthetic resin vehicles. Full details of composition, application, and spreading powers are quoted. S. S. WOOLF.

Durability of paints over woods treated with preservatives repellent to termites. W. F. HAR-RISON, D. BUCHANAN, R. M. BAIRD, and T. C. DOODY (Amer. Paint & Varnish Manufrs.' Assoc., Dec., 1931, Circ. No. 404, 407—460).—Three types of wood (redwood, Douglas fir, and yellow pine) treated with ZnCl<sub>2</sub>, Zn(AsO<sub>2</sub>)<sub>2</sub>, and six proprietary preservatives (of various types, but excluding any creosote types) were painted with a standard Pb–Zn paint system over each of three different primers and exposed in quadruplicate alongside painted panels that had had no pretreatment with preservative. Full reports of all panels including ratings for appearance, integrity, and protection after 6 and 12 months, days to failure, final inspection report, and computations of the collected results are given.  $ZnCl_2$  and "Bruce 5 A" (an org. chemical dissolved in petroleum oil) alone showed deteriorating effects on paint. The other variables had little or no effect. The need for painting treated woods is, however, established. S. S. WOOLF.

Two-coat spray painting on different woods. M. R. COLE (Amer. Paint & Varnish Manufrs.' Assoc., Dec., 1931, Circ. No. 404, 461—472).—The results of  $1\frac{1}{2}$  years' exposure of 4 white linseed oil paints based on TiO<sub>2</sub>-ZnO, Pb-Zn, lithopone-ZnO, lithopone-leaded ZnO pigments, respectively, applied in 2 thin spraycoats on 4 types of wood are given in detail. Spruce gave the best and pine the poorest results, whilst the lithopone-leaded ZnO pigment combination was the most successful. S. S. WOOLF.

Glycerol phthalate resins in quick-drying house paints. L. K. Scorr (Amer. Paint & Varnish Manufrs.' Assoc., Dec., 1931, Circ. No. 404, 522-541).-A series of exposures designed to determine the optimum vol.ratios of pigment and resin in the dried film is fully detailed. The results confirm earlier ones (B., 1931, 33) as to the superior durability of the glyptal coatings when compared with linseed oil paints. The pigmentresin ratios in satisfactory paints ranged from 24.8:  $75 \cdot 2$  to  $36 \cdot 5 : 63 \cdot 5$  by vol., and TiO<sub>2</sub>-ZnO was the best pigment combination. For const. vol.-ratios durability increases with opacity of pigment. In a further exposure test, clear linseed fatty acid-glycerol phthalate resins proved much more durable than albertol- and ester S. S. WOOLF. gum-spar varnishes.

Paints for reflecting biologically-important ultraviolet radiation. M. LUCKIESH and L. L. HOLLADAY (J. Franklin Inst., 1931, 212, 787—795).—Casein and nitrocellulose lacquer can be used as binders for ultraviolet-reflecting paints, which should preferably contain a large proportion of MgO, MgCO<sub>3</sub>,  $AI_2O_3$ , or a mixture of these. Moderate amounts of SiO<sub>2</sub>, CaCO<sub>3</sub>, china clay, and asbestine may be added without greatly reducing the reflecting power, which is about 0.50-0.65.

J. W. SMITH. "Critical oil content" of paints. H. WOLFF (Farben-Ztg., 1931, 37, 374-376).—The author's previous work on the derivation on empirical grounds of a "critical oil absorption" of paint pigments and "critical viscosity" of paints so formed is summarised. It is shown from the behaviour of paints of oil contents higher or lower than the crit. val. that the latter is actually an optimal val. It is also shown that at the crit. point the distance between pigment particles depends on their size and activity, and a theory postulating an adsorbed layer of oil around the pigment particles, the space between the "sheathed" particles being filled by "free oil," is developed. At the crit. point the sheathed particles are closely packed and the "free oil" just fills the interstices, the "free oil' content of a "critical" paint being approx. 50%. S. S. WOOLF.

Comparison of laboratory and factory batches [of paint, varnish, etc.]. CHAMBERLIN (Amer. Paint & Varnish Manufrs.' Assoc., Dec., 1931, Circ. No. 404, 643—652).—A summary is given of various observers' experiences in transferring processes from laboratory to factory scale. Factors accounting for differences in varnish manufacture are ratio of vol. to exposed surface and to surface to which heat is applied, extent of agitation, uniformity of heat distribution, and rate of temp. exchange, whilst in grinding paints and enamels heat generated before and during grinding, ratio of peripheral speeds of ball mills, degree of dispersion, etc. are functioning variables. S. S. WOOLF.

Improvements in [paint-]drier technology. G. H. PICKARD (Amer. Paint. & Varnish Manufrs.' Assoc., Dec., 1931, Circ. No. 404, 542—549).—The significant properties of driers for use in paints, varnishes, etc. are uniformity, solubility, degree and permanence of activity, extent of yellowing of the product, and effect on skinning. Naphthenate driers are very desirable from all these aspects. The functions of Pb, Mn, and Co in driers and suitable ratios of the metals for use in combination are discussed. S. S. WOOLF.

[Paint-]drier standardisation—the "Aridyne." II. C. F. CARRIER (Amer. Paint & Varnish Manufrs.' Assoc., Dec., 1931, Circ. No. 404, 635—641).—Tests of the drying power of Pb naphthenate solutions of various concns. on linseed oil suggest the possible presence of an anti-drying material in the oil that has to be offset before positive drying occurs. Deterioration of drying power in mixtures which are set aside is also indicated. Comparative tests with equiv. amounts of Pb in different forms, *e.g.*, as linoleate, gave inconclusive results. The use of the "aridyne solution" (cf. B., 1931, 33) to standardise a Pb–Co mixed drier, which would be a more convenient standard than Pb alone, is considered desirable. S. S. WOOLF.

Accelerated weathering [of paints]. R. B. SHURTS (Amer. Paint & Varnish Manufrs.' Assoc., Dec., 1931, Circ. No. 404, 473-478).—The shortcomings of an earlier series of artificial weathering experiments are discussed, and errors to be avoided in such trials are indicated. Four paints of graded durability were submitted to 25 different accelerated weathering cycles. General conclusions are drawn as to the influences of the various weathering agencies, but no optimum cycle is suggested. S. S. WOOLF.

Code for designating and evaluating exposure tests on paint, varnish, and lacquer coatings. J. A. FREDRICKSON (Amer. Paint & Varnish Manufrs.' Assoc., Dec., 1931, Circ. No. 404, 563-594).—A questionnaire on terminology used and standards of rating and a digest of the replies thereto are given. From consideration of these, definitions of general appearance, discoloration, gloss, checking, cracking, chalking, flaking, scaling, blistering, and peeling are developed. Suitable ratings for the various properties are also laid down and characteristic photographs illustrating the most controversial items are included. S. S. WOOLF.

Hardness of films. R. S. DANTUMA (Farben-Ztg., 1932, 37, 481-483).—An apparatus on the lines of

Toeldte's needle penetrometer (B., 1931, 851) is described, in which, however, complete penetration only of the film is recorded (instead of progressive penetration), the load on the needle being gradually increased. A spherometer device for measuring film thickness is included. The "swinging beam" method is also discussed. Sand abrasion tests are accelerated by using compressed air or centrifugal force in place of gravity for applying the sand to the surface to be tested, and the apparatus described permits the testing of several panels at one time. S. S. WOOLF.

Causes of floating pigments in paints and enamels. E. V. LADD (Amer. Paint & Varnish Manufrs.' Assoc., Dec., 1931, Circ. No. 404, 479-498; cf. B., 1931, 33).—The nature of the electrical charges on paint pigment particles was studied by examining under the microscope the motion of particles in oil or varnish in which an electrostatic field was set up between two wires. Larger-scale experiments where the field was set up between two plates proved unsatisfactory. It was shown that a definite charge exists on the particles of some pigments at low concn., but in a paint film of normal concn. these charges have no appreciable effect on the floating of pigment. A greater factor in this connexion is solvent evaporation, since floating was shown to be reduced in a paint drying in an atm. of S. S. WOOLF. solvent vapour.

Dispersometric analysis applied to paint pigments. R. SALVADORI (Giorn. Chim. Ind. Appl., 1931, 13, 515-519).-When a system consisting of an inert liquid and a very fine powder in sedimentation equilibrium is treated with a third substance which is sol. in the dispersing liquid and cannot be incorporated with the powder, the sedimentation vol. undergoes contraction varying in degree with the nature and amount of the third substance. The disperse vol. of 100 g. of a powder is termed the "strato-volume," this varying with the chemical and physical constitution of the powder. Experiments made with "trieline" as dispersing liquid, boiled or raw linseed oil, olive oil, or vaseline oil as third substance, and a number of powdered pigments give results indicating the value of this method of studying the action of drying oils on pigments.

T. H. POPE.

Oil absorption [of pigments] and acid value. K. CENTNER (Farben-Ztg., 1931, 37, 340-341).—Oils of acid val. ranging from 0.5 to 20 were prepared by adding the requisite amount of linseed oil fatty acids to cold-pressed linseed oil, freed from fatty acids, mucilage, etc. The oil-absorption vals. of a range of pigments for these oils show a rapid decrease up to acid val. 5, the figure remaining practically const. with more acidic oils, or rising slightly owing to soap formation with consequent stiffening of the mix. The experiments were repeated, using mineral oils, and similar results were obtained except that the min. acid val. was 7. It is considered that linseed oil of acid val. 5 should be specified for use in oil-absorption determinations.

S. S. WOOLF.

Transparency of iron and bismuth thiocyanate solutions in regard to the determination of iron in red lead. H. HEINRICHS (Glastech. Ber., 1927,

5, 351-354; Chem. Zentr., 1931, i, 2511-2512).— Photograms and light-transparency curves of solutions of Fe(CNS)<sub>3</sub> and Bi(CNS)<sub>3</sub> show that a disturbance of the Fe(CNS)<sub>3</sub> colour need not necessarily result from the presence of Bi; on the other hand, a disturbance is possible with red lead poor in Fe. L. S. THEOBALD.

Lacquer and under-coatings. U. RETZOW (Farbe u. Lack, 1932, No. 1, 5—6).—Sheet steel subjected to Ni-Cd and Cd-Cu treatments, respectively, showed satisfactory durability under cellulose lacquer, whereas without the lacquer the "galvanising" coats weathered in 6 months. The lacquer did not protect a similar sheet of steel that had had no metallic treatment. A further series of tests on "doped" linen is described, the beneficial effects on the mechanical strength of the linen produced by coatings of clear lacquer being much enhanced by further coatings of pigmented lacquers. The difference is noticeable on storage in the shade and is accentuated on exposure to sunlight. Warp and weft behaved similarly in these trials. S. S. WOOLF.

Pigmented nitrocellulose lacquers. W. TOELDTE (Farben-Ztg., 1931, 37, 448—449).—A summary is given of details and results of an investigation in which more than 100 lacquers designed to show variations in type of nitrocellulose, resin, plasticiser, and pigments used were tested for colour, gloss, opacity, adhesion, elasticity, and resistance to abrasion, heat, cold, ultra-violet light, H<sub>2</sub>O, artificial and normal weathering.

S. S. WOOLF.

Plasticisers for nitrocellulose lacquers. K. STICKDORN (Farben-Ztg., 1931, 37, 341-342).—The softeners at present used are described and recent developments are outlined. The commercial production of hexyl, octyl, and decyl alcohols affords a new range of fatty acid alcohols, esters, and ethers, the b.p., sap. value, sp. gr., and  $n_{\rm D}$  of which are tabulated.

#### S. S. WOOLF.

Evaluation of clear nitrocellulose metal lacquers. G. C. SHAKOUR and L. W. MUNCHMEYER (Met. & Alloys, 1931, 2, 331---336).—The most widely used metal lacquers have nitrocellulose as a base, though lacquers of this type are not so durable as those manufactured from the condensation products of phenols with polyhydric alcohols or polybasic acids. The testing of a lacquer is concerned chiefly with the exposure of a bright Cu strip coated with the lacquer to the atm., H<sub>2</sub>S, NH<sub>4</sub>HS, and a salt spray. The adhesion of the film may be determined by a bending test. In the application of lacquers chemical cleaning of the metal is essential.

C. A. KING.

Japanning of steel. W. J. MISKELLA (Met. & Alloys, 1931, 2, 318-323).—Japanning is the cheapest and the most durable metal finish, not excepting Ni plating. The basis is gilsonite and the process is differentiated from painting in that no "drier" is used. True Japans are finished by baking at about 300° and cannot be applied to any surface, e.g., wood, which will be affected at that temp. Dull Japans, "Japan" varnishes, and plant suitable for the process are described. C. A. KING.

Soaps. III. Cooking procedure of a limed rosin varnish. E. C. HAINES (Amer. Paint & Varnish

Manufrs.' Assoc., Dec., 1931, Circ. No. 404, 619-629).-Twenty-one varnishes were made on the formula: rosin 100 lb., linseed oil 40 lb., tung oil 160 lb., petroleum thinner 300 lb., CaO 6 lb., the procedure being varied in each case. The amount of limed rosin cooked with the oil was progressively increased, the remainder of the rosin being added in one series as gloss oil and in another as solid rosinate. A further test was carried out by cooking all the "non-volatile" ingredients together and periodically extracting samples for thinning out. These varnishes were tested for viscosity, colour, drying time, resistance to hot and cold H<sub>2</sub>O and to lacquer thinner, accelerated and normal weathering, kauri reduction, and behaviour when made into enamel, and the results are reported. The properties are shown to alter progressively in series 1, improvements generally being noted with increase in the amount of rosin cooked with the oil up to half the total quantity of rosin.

S. S. WOOLF.

Exposure tests [on varnishes etc.]. BOLME (Amer. Paint & Varnish Manufrs.' Assoc., Dec., 1931, Circ. No. 404, 550—560).—Lacquers containing varying amounts of nitrocellulose and plasticiser were exposed in parallel in Florida, in Detroit, and to an accelerated weathering cycle. The results show that the last-named caused undue chalking, whilst the differing conditions in the two localities mentioned account for striking divergences in behaviour of paint. Exposure racks should not be protected from the wind, since mechanical as well as chemical weathering is shown to exert influence. S. S. WOOLF.

Resins. IX. Sandalwood resin. X. Malayan dammar. E. STOCK (Farben-Ztg., 1931, 37, 446– 448; cf. B., 1931, 768).—The following properties of a sample of sandalwood resin and of four Malayan dammar samples are quoted : acid val., sap. val., ash content, colour reactions, solubility in a large number of solvents and ultra-violet fluorescence in solution in many of these, and capillary-absorption tests. The possible use of these resins in cellulose lacquers is indicated. S. S. Woolr.

Recovery of volatile solvents for resin production. C. R. MAHANEY (Chem. and Met. Eng., 1931, 38, 698-701).-Air containing 0.45% EtOH and some fine solids from a plant manufacturing "Micarta "-a laminated moulded material-is cooled and the solids are removed by spraying in a wooden hurdle tower with liquor which is cooled and recirculated. The air is then scrubbed countercurrently with water and the 2-3% aq. EtOH obtained is distilled, giving a 1:3 mixture of EtOH vapour and steam, which after rectification yields a vapour containing 5% of steam. Furfural, PhOH, and cresol are withdrawn from certain decks of the still and condensed, and the lighter aq. EtOH is separated from the heavier layer of furfural, PhOH, cresol, and a little water by decantation and returned to the still. The steam and H<sub>2</sub>O consumptions are 60 lb. and 100 gals., respectively, per gal. of EtOH. The overall EtOH recovery is 85-95% (with cooling water at 5°) in winter and 70–85% (24°) in summer. D. K. MOORE.

Gum "running." E. STOCK (Farben-Ztg., 1932, 37, 484-485).---A stream of CO<sub>2</sub> is passed through the

hollow stem of a latticed stirrer which revolves in the heated gum mass. Decrease in time of running, diminution of frothing, and improved colour of the "run" gum are claimed for this device (which is patented). S. S. WOOLF.

Abietene.—See III. Extractives of cypress pines. —See IX. Surface-coating A1.—See X.

See also A., Jan., 61, Separation of terpineols. Constituents of Indian turpentine from *Pinus longifolia*.

#### PATENTS.

Coating [pipes with aluminium paint]. G. A. JOHNSON, ASST. to AMER. STEEL PIPE Co., INC. (U.S.P. 1,804,991, 12.5.31. Appl., 20.12.28).—The pipes are degreased, pickled, dried, and dipped in a paint comprising finely-divided Al suspended in a mixture of linseed oil, tung oil, a resinous gum, and a Pb, Mn, or Co drier. After draining, the pipes are baked at 150° for 1 hr., again coated, and finally baked at 165° for 2 hr. A. R. POWELL.

Manufacture of zinc white and apparatus therefor. V. SZIDON (B.P. 362,812, 1.4.31. Fr., 18.12.30).— The vapours and gases leaving the known Wetherilltype furnace for conversion of Zn ores and residues into ZnO pass without appreciable change of speed through a second furnace where they are raised to 1000°, thence to an expansion chamber where they meet hot air at similar temp., and are finally cooled and condensed. The burning of combustible matter and the oxidation of the Zn vapour are completed by this process; the product needs no further refining. S. S. WOOLF.

Production of varnishes, lacquers, and the like. "HEROLD" A.-G. (B.P. 362,728, 13.1.31. Ger., 20.2.30). —A hard material, e.g., SiC, of grain size 40—100  $\mu$ , is incorporated with synthetic resin solutions, to produce coatings resistant to shocks and blows. S. S. WOOLF.

Manufacture of pyroxylin solutions [of low viscosity]. A. E. WHITE. From HAMMERMILL PAPER Co. (B.P. 363,648, 19.6.30).—See U.S.P. 1,768,253; B., 1931, 170.

Grinding mill for colours, inks, enamels, and like materials. J. H. HOLMES, and G.M.S. ENGINEERING Co. Soc. ANON. (B.P. 363,456, 23.10.30).

Production of gramophone disc records. N. BODENSTEIN (B.P. 364,504, 28.10.30. Austr., 28.10.29).

Tar emulstions.—See II. Wetting etc. agents. Polymerisation products. Condensation products for varnishes etc.—See III. Pigment dyes. Azo pigments.—See IV. Priming absorbent foundations. —See IX. [Paint for] Fe or steel.—See X.

# XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Effect of selenium in rubber mixings. W. ESCH (Kautschuk, 1932, 8, 10-13; cf. B., 1931, 1108).— The beneficial effect of Se on the abrasion-resistance of vulcanised rubber is re-affirmed and further evidence quoted. Rimpel's discrepant results are possibly due to his Se being unsuitable for use with rubber.

D. F. Twiss.

Influence of grit on the tensile properties of a [rubber] mixing containing gas-black. E. P. W. KEARSLEY and C. H. PARK (Kautschuk, 1932, 8, 8–10). —A gas-black of good quality was mixed with 1—10% of the grit, collected in the refining sieves of a C-black factory, which had been powdered to pass 100-mesh. The tensile strength and modulus of vulcanisates containing 47% (on the rubber) of the gas-black were affected appreciably only when the proportion of introduced grit exceeded 3%. Low tensile strength occasionally observed in gas-black mixings is to be attributed to unsatisfactory dispersion rather than to grittiness of the material. D. F. TWISS.

Plasticity measurements in the rubber industry. J. BEHRE (Kautschuk, 1932, 8, 2—5).—Experimental results with an extrusion-plastometer of the Marzetti type are adduced in illustration of the usefulness of plasticity tests on raw and masticated rubber, as aiding factory control, and on compounded rubber, as giving evidence of the relative reinforcing effect of various fillers, the relative efficiency of different softening agents, and the relative tendency of various accelerators to lead to premature incipient vulcanisation.

D. F. Twiss.

An occurrence of "copper destruction" of vulcanised rubber and its explanation. F. KIRCH-HOF (Kautschuk, 1932, 8, 6-8).-Of two motor tubes of like composition vulcanised after being printed with a bronze pigment, one which had remained for 6 years in the dark showed bad surface-cracking in the neighbourhood of the lettering, whereas the other was free. The effect is attributed mainly to a higher degree of vulcanisation in the former tube with consequent more rapid ageing and formation of H<sub>2</sub>SO<sub>4</sub>, which converted the CuS (formed by the vulcanisation process) into CuSO4. CuS appears to be inert towards rubber, whereas the sol. CuSO<sub>4</sub> leads to rapid destruction. The main direction of the cracks appears to be decided by the calander grain of the rubber, which had been shaped by extension. D. F. Twiss.

#### Manuring of Hevea plants .- See XVI.

#### PATENTS.

Manufacture of natural rubber and artificial rubber-like masses. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 361,981, 30.8.30. Cf. B.P. 352,549; B., 1931, 896).—The condensation products of ar-tetrahydro- $\alpha$ -naphthylamine with aliphatic aldehydes (C<sub>2</sub> or over) obtained in the presence of H<sub>2</sub>Osol. alcohols and acids are applied as protective agents against ageing. The aldol product has m.p. 140—150°. D. F. Twiss.

Manufacture of products from rubber and rubberlike substances. IMPERIAL CHEM. INDUSTRIES, LTD., H. M. BUNBURY, J. S. H. DAVIES, and W. J. S. NAUNTON (B.P. 363,483, 22.11.30).—Resistance to deterioration and discoloration by ageing is imparted to rubber by incorporating a boric acid ester of a naphthol, e.g., tri- $\beta$ -naphthyl borate. An additional advantage of these agents is that, unlike the naphthols, they have no tendency to volatilise during the mixing operation and do not retard vulcanisation. D. F. TWISS.

Production of moulded articles resembling wood of compositions containing rubber. METALLGES. A.-G. (B.P. 361,941, 30.6.30. Ger., 29.6.29).-Rubber latex containing S and any other desired fillers is mixed with substances capable of producing a gradual thixotropic increase in its viscosity, e.g., alum, MgSO<sub>4</sub>, or Ca formate, and is then brought into contact with an unheated mould. After a period the still fluid portion of the mixture is removed, and the portion adherent to the wall of the mould is subjected to partial coagulation and consolidation, e.g., to the consistency of leather, by thermal or chemical means. The soft moulded article, after previously filling with suitable material such as sawdust, if desired, is then removed from the mould and vulcanised. It is advantageous to treat the moulds, prior to use, with a wetting agent, e.g., dil. aq. D. F. Twiss. alkali, MeOH, or Nekal.

Vulcanisation of rubber. R. T. VANDERBILT CO., INC., Assees. of P. I. MURRILL (B.P. 359,328, 20.1.31. U.S., 1.4.30).—Rubber is vulcanised in presence of an org. Te compound containing a  $\cdot$ CS $\cdot$ S $\cdot$  group. The Te compound is an accelerator when S is present, but in absence of S acts both as a vulcanising and an accelerating agent. Te diethyldithiocarbamate, (NEt<sub>2</sub>  $\cdot$ CS<sub>2</sub>)<sub>4</sub>Te, m.p. 123—124°, prepared by interaction of Na diethyldithiocarbamate (4 mols.), NaTeO<sub>2</sub> (1 mol.), and HCl (6 mols.), is especially suitable. C. HOLLINS.

Rubber vulcanisation accelerators. FIRESTONE TIRE & RUBBER Co., LTD., Assees. of M. H. ZIMMERMANN (B.P. 361,971, 29.8.30. U.S., 29.1.30).—Premature vulcanisation is avoided by the use of the reaction product (m.p. 130°) of mercaptobenzthiazole and CH<sub>2</sub>O as an accelerator. D. F. Twiss.

Treatment of rubber. [Anti-agers.] NAUGATUCK CHEM. Co., Assees. of W. P. TER HORST (B.P. 359,773, 29.11.30. U.S., 4.12.29).-The condensation product of a ketone, preferably aliphatic, and an arylamine, obtained, e.g., in presence of I, is applied as an anti-ager alone, or as its naphthol additive compounds, or after further condensation with an aldehyde, or as nitrosoor S-derivative. Examples are products from COMe2 (1-4 mols.) and NH2Ph, o-C7H7·NH2, 4:4'-diaminodiphenylmethane, NHPh2; from CH2O and the COMe2-NH<sub>2</sub>Ph or COMe<sub>2</sub>-NHPh<sub>2</sub> product; from aldol and the COMe<sub>2</sub>-NH<sub>2</sub>Ph product; from PrCHO and COMe<sub>2</sub>- $\mathrm{NHPh}_{2}^{2}$ ; from  $\beta$ -C<sub>10</sub>H<sub>7</sub>·OH and  $\mathrm{COMe}_{2}$ -NHPh<sub>2</sub>; from chloroacetone and 3 mols. of  $\mathrm{NH}_{2}$ Ph; from COMe<sub>2</sub> and phenyl- $\alpha$ - or - $\beta$ -naphthylamine, or NHPhEt, or NN'-diphenylethylenediamine; from COMeEt and NHPh<sub>2</sub>; from mesityl oxide and phenyl- $\alpha$ - or - $\beta$ naphthylamine ; nitrosated or sulphurised product from C. HOLLINS. COMe<sub>2</sub> and NHPh<sub>2</sub>.

Manufacture of [age-resisting] rubber. GOOD-YEAR TIRE & RUBBER CO. (B.P. 359,286, 12.12.30. U.S., 4.1.30).—A thiodiarylamine is used in conjunction with other anti-oxidants (especially non-accelerators). Examples include : thiophenyl- $\alpha$ - or - $\beta$ -naphthylamine, thiodiphenylamine,  $\alpha\alpha'$ -thio- $\beta\beta\beta'$ -dinaphthylamine ; these are used with diaminodiphenylene oxide, NN'-di- $\beta$ naphthylethylenediamine, diaminoditolylmethane, NN'-di- $\beta$ -naphthyl-p-phenylenediamine, di- $\alpha$ -naphthylformamidine, diaminocarbazole, 2 : 4-diaminophenyl

**Preservation of rubber.** GOODYEAR TIRE & RUBBER Co. (B.P. 362,172, 13.12.30. U.S., 17.2.30).—A cyclohexylnaphthylamine, obtainable by heating cyclohexylamine with  $\alpha$ - or  $\beta$ -naphthol at 280—300° under pressure, is used as an antioxidant. The  $\alpha$ - and  $\beta$ isomerides are cryst. solids which can be distilled under reduced pressure. D. F. TWISS.

Evacuating air from plastic etc. material. Vulcanisation.—See I. Polymerisation products. —See III. Rubber-coated textiles.—See VI.

#### XV.-LEATHER; GLUE.

Influence of grease on the deterioration of chestnut- and quebracho-tanned leathers by sulphuric acid. R. C. BOWKER (J. Amer. Leather Chem. Assoc., 1931, 26, 667—674; cf. B., 1931, 1021).—Deterioration was unaffected by the incorporation with 10% or 20% of a 1:1 cod oil-tallow mixture. D. WOODROFFE.

Ethanolamine soaps.—See XII.

See also A., Jan., 20, Absorption of H<sub>2</sub>O by gelatin. 61, Vegetable tannins.

#### PATENTS.

Tanning substances. J. R. GEIGY, Soc. ANON (J. R. GEIGY) (B.P. 362,797, 10.3.31. Ger., 10.3.30. Addn. to B.P. 305,013; B., 1929, 1025).—An aromatic hydroxy-carboxylic acid (*e.g.*, gallic, salicylic, *o*-cresotinic acid), or a mixture of these acids, is present in addition to urea etc. in the condensation described in the prior patent. L. A. COLES.

Salts of alkylsulphuric acids. Wetting etc. agents. Tanning agents. Inhibiting development of micro-organisms.—See III.

#### XVI.—AGRICULTURE.

Nature of the capillary rise [of water] through soils. H. A. WADSWORTH (Soil Sci., 1931, 32, 417–433). —The mechanism of the process is discussed. The min.  $H_2O$  content reached by true capillary movement approximates to the max. field capacity. The possibility that a considerable proportion of the soil- $H_2O$ is colloidally imbibed is noted. A. G. POLLARD.

Effect of replaceable sodium on soil permeability. A. E. HARRIS (Soil Sci., 1931, 32, 435–446).— The permeability to  $H_2O$  of calcareous alkali soils decreases exponentially as the replaceable Na content increases. In soils free from alkali, a high humus content is associated with greater permeability than exists in calcareous horizons. In soils containing Na the reverse is the case. Experimental data are recorded from which the effects of long periods of leaching on the replaceable Na contents and permeability of soils may be calc. A. G. POLLARD.

**Replaceable iron and aluminium in soils.** P. E. TURNER (Soil Sci, 1931, 32, 447–458).—Replaceable Al occurs only on soils of  $p_{\rm H} < 5$ , but no definite relationship exists between  $p_{\rm H}$  and replaceable Al content. The amount of replaceable Al in a soil is related to its

saturation deficit. Until the latter val. reaches a min. of 8.25 milli-equiv. (m.e.) no replaceable Al appears; from  $8 \cdot 25 - 15 \cdot 5$  m.e. the amount rises slowly and above 15.5 increases rapidly. In soils of  $p_{\rm H} > 5.1$  replacement of Al is wholly indirect, i.e., by interaction with H ions. At low  $p_{\rm H}$  ranges direct replacement occurs although the indirect type predominates. Within ranges of saturation deficit of 8.25-15.5 m.e. the ratio replaceable H: Al = 50 approx., but falls rapidly at greater deficits. The amount of replaceable Al in soils is not closely related to the degree of unsaturation. Fe does not appear in soil leachates until the saturation deficit reaches 10—12 m.e. In no soil examined was the  $p_{\rm H}$ sufficiently low for Fe to become directly exchangeable. The intensity of the thiocyanate colour produced by soils is more closely related to  $p_{\rm H}$  than to saturation A. G. POLLARD. deficit.

Determination of soil reaction with the "pehameter." K. KRUMINŠ (Z. Pflanz. Düng, 1931, 10 B, 553-556; cf. A., 1931, 699).-An improved technique for the use of the Hellige pehameter is described.

A. G. POLLARD.

Determination of  $p_{\rm H}$  values [in soils] with the antimony electrode. J. DI GLERIA (Kisérlet. Közl., 1930, 33, 193; Bied. Zentr., 1931, 1A, 481).-Pure Sb electrodes are serviceable for  $p_{\mathbf{H}}$  determinations in soils and may be used for vals. > 8.5. A. G. POLLARD.

Relation of  $p_{\rm H}$  drift to moisture content and base held in soils. A. T. PERKINS and H. H. KING (Soil Sci., 1931, 32, 409-416).—The potential drift observed in the  $p_{\rm H}$  measurement of soils by the quinhydrone electrode is not affected by the nature of the exchangeable bases present if the customary soil: H<sub>2</sub>O ratios are used, but increases as the proportion of H<sub>2</sub>O is raised. Moist soils show a larger drift than those dried prior to examination. A. G. POLLARD.

Determination of carbonates in soils by the Puri method. F. UKRADIGA and U. SAMBUROV (Nauk. Zapiski Tzuk. Prom., 1931, 12, 431-433).-A field method employing bromothymol-blue is described.

CHEMICAL ABSTRACTS. Determination of the degree of saturation of soils with lime. W. SAUERLANDT (Landw. Versuchs-Stat., 1931, 113, 69-82).-Accepted methods for determining the max. saturation capacity of soils are compared, and, in general, showed good agreement. Janert's formula (B., 1931, 454) connecting max. adsorption with heat of wetting is modified. The % of clay in soil corresponding to 1 g.-cal. of heat of wetting varied, in a number of soils, from 3.6 to 17.5. The exchange of soil cations for H<sup> $\cdot$ </sup> is complete if Kappen's ratio of soil to 0.1N-HCl is reduced to 10 g.: 200 c.c. A. G. POLLARD.

Determination of lime requirement [of soils] by direct addition of calcium carbonate. G. P. PERCIVAL (Soil Sci., 1931, 32, 459-465).-To a series of 60-g. samples of soil are added increasing amounts of CaCO3 and the mixtures, suspended in 60 c.c. of H2O, are placed in pear-shaped separating funnels through which air is bubbled to remove CO, as formed. After 20 hr. the  $p_{\rm H}$  vals. are determined by the quinhydrone electrode. By intrapolation of the titration curves thus obtained the CaO required to produce a given  $p_{\rm H}$  is calc., use being made of a factor depending on the amount of org. matter present. Results so obtained agree well with those based on hydrolytic acidity measurements.

A. G. POLLARD.

The phosphoric acid and lime question and its practical importance in soil research. H. ELLEDER (Z. Pflanz. Düng., 1931, 10B, 556-566; cf. B., 1930, 630).—The easily assimilable P content of soils varies with the amount of CaCO<sub>3</sub> present. Fixation of P in a difficultly sol. form is associated with soils having >4% CaCO<sub>3</sub>. Both the Neubauer and Lemmermann methods for determining the available P in soils are affected by  $CaCO_3$  contents of 4-5%. For such soils the Dirks and Scheffer method is preferable. Difficulties in securing the correlation of results of laboratory and field tests for P are discussed. A. G. POLLARD.

Effects of lime in plantn utrition. CLAUSEN (Forts. Landw., 1931, 6, 197-201; Bied. Zentr., 1931, 1A, 466).—The action of various dressings of CaO on yields of oats, rye, and potatoes in 9 years' cropping following its application are recorded. The response of the crops to CaO was in the order rye > oats > potatoes. In all cases the CaO effect was greater in unmanured plots and in those receiving fertilisers without N. CaO reduces the intake of K by plants, and this negative effect increases with the K deficiency of the soil. Potato scab was very marked in all limed plots and increased in the later years. Effects of CaO were more definite in moist and cool than in dry and warm seasons, and were somewhat reduced by applications of stall manure. On K-deficient soils stall manure lessened the ill-effects of CaO. A. G. POLLARD.

Effects of liming in Yestfold. P. Solberg (Tidsskr. norske Landbr., 1931, 13-19; Bied. Zentr., 1931, 1A, 467-468).-Crop increases produced by liming were greater on sandy than on heavy soils. On the latter large dressings of CaO produced greater effects than small, but on light soils results were identical. The action of CaO was more persistent on heavy than on light soils. In sown meadows timothy and clover disappeared after 2-3 years, but persisted for long periods after liming. A. G. POLLARD.

Action of calcium cyanamide in comparison with ammonium sulphate on acid sandy soils. F. NIESCHLAG (Oldbg. Landw. Blatt, 1931, 16, 253-255; Bied. Zentr., 1931, 1A, 468).-In soils having suitable numbers of bacteria capable of converting urea into NH<sub>3</sub>, the action of CaCN<sub>2</sub> is not dependent on soil reaction. An appropriate bacterial flora is maintained by light dressings of stall manure. The satisfactory use of CaCN<sub>2</sub> on normal but acid soils is demonstrated.

A. G. Pollard. Factors affecting nitrates in soils. J. W. Ames (Ohio Agric. Exp. Sta. Bimo. Bull., 1931, No. 153, 232—244).—Natural variations in the  $NO_3'$  content over small soil areas were considerable (18-58 lb. of nitrate-N per acre in  $\frac{1}{20}$  acre area). In cropped soils the NO3' content in plant rows was reduced in many cases to  $\frac{1}{4}$  of that between rows. The depletion of  $NO_3'$  in the rows was proportional to the growth of adjacent plants. Drying and rewetting of soil increased nitrification. A. G. POLLARD.

Nitrification in podsolised soils. N. V. YASHNOVA (Trans. Sci. Inst. Fertilisers, Moscow, 1930, No. 76, 50—68).—Even with a very small amount of  $NH_3$  in solution nitrification in podsols takes place. The adsorbed  $NH_3$  is available for nitrification, which is hindered by much sol. org. matter or by lack of phosphates. Increase in aëration and dissolved  $NH_3$ increases nitrification. When  $NH_3$  accumulates and nitrification is hindered *B. mycoides* multiplies greatly. Waksman's method for determining the nitrification capacity of a soil is unsuitable. With a high content of sol. org. matter denitrification is possible.

CHEMICAL ABSTRACTS.

Denitrification in the soils of the middle chernozem region. N. G. POTAPOV (Trans. Sci. Inst. Fertilisers, Moscow, 1930, No. 76, 97—111).—The number of denitrifiers, which is large, is controlled by the sol. org. matter. The disappearance of nitrates is attributed to the activity of these organisms. When denitrification is max. the soil reaction shifts towards alkalinity.

CHEMICAL ABSTRACTS. Distribution of denitrifiers in the genetic horizons. N. G. POTAPOV (Trans. Sci. Inst. Fertilisers, Moscow, 1930, No. 76, 92—96).—The number of denitrifiers decreases with the depth. Cultivation increases the number and activity of the denitrifiers (*B. stutzeri*).

CHEMICAL ABSTRACTS.

Mutual relations between Azotobacter and denitrifiers. D. M. NOVOGRUDSKI (Trans. Sci. Inst. Fertilisers, Moscow, 1930, No. 76, 69–91).—NH<sub>3</sub> is an intermediate product in the metabolism of Azotobacter chroicoccum. The NH<sub>3</sub> in the cultures favours the growth of *B. denitrofluorescens* and *B. stutzeri*.

#### CHEMICAL ABSTRACTS.

Longevity of legume bacteria on seed, as influenced by plant sap. N. PORGES (Soil Sci., 1931, 32, 481-487).—Organisms retained by seed treated, after sterilisation, with an inoculum containing sap from their sp. plants, withstood desiccation better than those from seed treated with an inoculum containing milk. A. G. POLLARD.

Effects of sorghum plants on biological activities in soil. A. D. McKINLEY (Soil Sci., 1931, 32, 469-480). -Wetting of partly or completely dried soils resulted in a marked evolution of CO2 during several weeks. Larger amounts of CO, were produced in cropped than in uncropped soils, the max. coinciding with periods of greatest vegetative growth. CO<sub>2</sub> production was characteristic for each type of plant. The micro-org. flora of soils increased during cropping, the relative proportions of filamentous fungi, bacteria, and actinomycetes varying with the nature of the crop. It is suggested that this may be due to the excretion of sol. org. matter by the plants. In fallow soils increased numbers of microorganisms were not associated with an increased production of CO<sub>2</sub>. Little, if any, increase in nitrification or N fixation in soils results from the growth of plants in them. A. G. POLLARD.

Variation in the "effect factors" of nitrogen and phosphoric acid [in plant-growth curves]. W. M. KLETSCHKOWSKY and P. A. SHELESNOW (Landw. Jahrb., 1931, 74, 353-404).—The effect of the propor-

tion of available N in soil on the "effect factor" of P, and vice versa, is discussed; it is shown to confirm the views of Rippel (B., 1926, 1024). Mathematical relationships of the parameters of Mitscherlich's yield-law formula are examined. A. G. POLLARD.

Effect of fertilisers and date of planting on the physiological development of the maize plant. R. W. GERDEL (Plant Physiol., 1931, 6, 695-714).— With adequate supplies of K, P, and N, maize plants showed marked differentiation between the vegetative and reproductive cycles, characterised by grain yields, earliness of "silking," and the period over which silking occurred among similarly treated plants. With deficient supplies of nutrients, silking was spread over a much longer period and was later. Max. leaf area, diam. of stalk, and height of plants, also earliest silking and min. period of silking, were induced by fertiliser applications considerably below those giving max. yield increases. A. G. POLLARD.

Effect of cutting and fertiliser application on grass development. C. M. HARRISON (Plant Physiol., 1931, 6, 669-684).—Close cutting of grass reduced root development. Application of fertilisers did not compensate for restricted aërial growth in the production of roots. Severe cutting killed grass by a gradual carbohydrate starvation and consequent inability of the roots to maintain their tissues. Application of N increased top growth, but did not affect the wt. of roots. A. G. POLLARD.

Sensitivity of red clover to small amounts of boron and manganese. B. E. GUILBERT and F. R. PEMBER (Plant Physiol., 1931, 6, 727-729).—Small additions of Mn and B to nutrient solutions for red clover increased both root and top growth. Red clover is much more sensitive to the absence of these elements than are the grasses. A. G. POLLARD.

Available soil calcium in relation to "dampingoff" of soya-bean seedlings. W. A. ALBRECHT and H. JENNY (Bot. Gaz., 1931, 92, 263—278).—The [H] of the nutrient is not an important factor in the "damping-off" of soya beans. The disease decreased with increasing Ca supply at all reactions in the range  $p_{\rm H}$  3.8—6.9. Ca" is more potent than K° or Mg" in preventing the disease, and free diffusible Ca" more effective than exchangeable Ca". A. G. POLLARD.

Cultivation of American soya beans in Germany. B. REWALD (Landw. Versuchs-Stat., 1931, 113, 93— 101).—American varieties of soya beans produced crops in Germany having less fat and protein, but similar phosphatide contents, in comparison with Americangrown crops. Differences are not of the same order in all varieties. A. G. POLLARD.

Ensilage of raw potatoes by embedding in potato mash. G. RUSCHMANN and G. GRAF (Landw. Jahrb., 1931, 74, 447—456).—Potatoes are steamed, mashed, and used as an embedding medium for raw potatoes for ensilage. The extent of the subsequent fermentation is largely controlled by the sugar content of the potatoes. Lactic fermentation predominates and smaller proportions of acetic and other acids occur.

A. G. POLLARD.

Nutrient requirements and histology of the cranberry, with special reference to mycorrhiza. R. M. ADDOMS and F. C. MOUNCE (Plant Physiol., 1931, 6, 653-668).—In sand cultures, cranberry plants produced greater runner growth when N was supplied as  $NH_4$  than as  $NO_3'$ . Low concess, of  $NO_3'$  promoted, and high concess. restricted, vegetative growth. Mycorrhiza occurred in all cultures, but were fewest in those free from N. If N fixation by the endophyte *Phoma* radicis occurred at all, it was quite inadequate for the growth of the plants. At no time was  $NO_3'$  observed within the tissue of the cranberry or the endophyte.

A. G. POLLARD.

Preparation of organic manure. R. G. H. WILSHAW (Malayan Agric. J., 1931, 19, 593-595).— The decomp. of composts of grass cuttings and cattle manure (judged by pentosan destruction) is rapid, and at 28° with 70-80% H<sub>2</sub>O is practically complete in 7 weeks. A. G. POLLARD.

Determination of nitrogen in fertilisers containing nitrates. T. ARND (Angew. Chem., 1932, 45, 22– 23).— $NO_3'$  is reduced by the Zn–Cu couple in MgCl<sub>2</sub> solution. The NH<sub>3</sub> formed is distilled off and determined. E. S. HEDGES.

Determination of urea-nitrogen in artificial manures. L. PINTOS (Praktika, 1929, 4, 95–99; Chem. Zentr., 1931, ii, 111).—The fertiliser (1 g.) is extracted 4 times with a little slightly warmed abs. EtOH; the filtrate is treated with  $H_2O$  (150 c.c.) and 33% NaOH (100 c.c.) and the NH<sub>3</sub> is distilled into N-H<sub>2</sub>SO<sub>4</sub>. Abs. EtOH at 8° dissolves 0.62% NH<sub>4</sub>Cl; guanidine is also sol. A. A. ELDRIDGE.

Losses of ammonia-nitrogen in storing peat or straw manure. S. I. KUZNETZOV (Trans. Sci. Inst. Fertilisers, Moscow, 1930, No. 76, 116–122).—Optimal  $p_{\rm H}$  vals. for growth are : Urobacillus duclauxii 7.4—7.7, U. macodoxii and U. pasteurii 8.2. With peat bedding the  $p_{\rm H}$  reaches 4.8 and the bacteria are incapable of producing NH<sub>3</sub> from CO(NH<sub>2</sub>)<sub>2</sub>. Hence the preservation of NH<sub>3</sub> in peat manure is not due to adsorption.

CHEMICAL ABSTRACTS. Manurial trials with raspberries. T. N. HOBLYN (J. Pomology, 1931, 9, 303—330).—Successful manuring of raspberries depends on a proper balance between N and K. N may produce more cane, but does not increase the crop except in the presence of K. K<sub>2</sub>SO<sub>4</sub> is, in general, superior to kainite for this purpose. In plot experiments, differences due to position may exceed those due to manurial treatment. A. G. POLLARD.

Canning experiments with Hevea in west Java. J. S. VOLLEMA (Arch. Rubbercultuur, 1931, 15, No. 10; Med. Proefstat. Rubber, 1931, No. 69, 481-574).— Manuring of young, slowly-grown plantations was generally successful, but with older tapping trees little benefit was obtained except in certain cases where N was applied. No appreciable difference was apparent between the effects of  $(NH_4)_2SO_4$ , NaNO<sub>3</sub>, and urea. CaCN<sub>2</sub> gave slightly poorer yields in some instances.

A. G. POLLARD.

Sarcostemma australe (caustic vine): a plant poisonous to stock. J. A. GILRUTH and D. MURNANE (J. Counc. Sci. Ind. Res., Australia, 1931, 4, 225–231).— S. australe is definitely poisonous to sheep, cattle, and horses. The active principle is sol. in EtOH, but insol. in light petroleum. It is neither a glucoside nor an alkaloid. It acts chiefly on the central nervous system. The effect is almost identical with that produced by other members of the "milkweed" family (Asclepiadacea). W. G. EGGLETON.

Poisoning of [animal] stock by fuchsia and gidgea. H. FINNEMORE (J. Counc. Sci. Ind. Res., Australia, 1931, 4, 220—224).—The fuchsia (*Eremophila maculata*), native of Queensland, is strongly cyanogenetic (one sample of leaves yielded 0.824%HCN). The glucoside-splitting enzyme, however, is not always present in sufficient quantity to cause the liberation of fatal quantities of HCN, but subsequent ingestion of leaves of certain other plants containing the enzyme, but not necessarily cyanogenetic, notably gidgea (*Acacia georgina*), may cause death.

W. G. EGGLETON. Pyrethrin I content of pyrethrum powders as an index of insecticidal power. H. H. RICHARDSON (J. Econ. Entom., 1931, 24, 1098—1106).—The toxicity of pyrethrum powders, whether fresh or deteriorated; is accurately indicated by their content of pyrethrin I.

A. G. POLLARD.

Fungicidal action of sulphur. III. Physical factors affecting the efficiency of dusts. F. WIL-COXON and S. E. A. MCCALLAN (Contr. Boyce Thompson Inst., 1931, 3, 509-528; cf. B., 1931, 821).-The toxicity of simple S dusts increases with diminishing particle size. The toxicities of dusts of different mean diam. compared on an equal-wt. basis show significant differences, but these differences disappear when comparisons are made with equal no. of particles per unit The adherence of dusts to surfaces increased area. with fineness of division. The greater toxicity of dusts containing supplementary materials (e.g., infusorial earth, bentonite) as compared with simple dusts disappears after exposure to artificial rain. Colloidal S was markedly superior to all other types of dusts after the rain test. The angle of slope of a cone of dust built up to max. height is a useful measure of its dusting quality. Differences in the toxicity of S dusts cannot be ascribed to different rates of formation of H<sub>2</sub>S, to S content, or to the acidity of aq. extracts.

# A. G. POLLARD.

Adhesives and carriers for insecticidal dusts. S. F. Ports and D. F. BARNES (J. Econ. Entom., 1931, 24, 1110—1111).—The adhesion of arsenate dusts, used without supplementary adhesives, was in the order Pb > Ca > Mg. Admixture of CaO reduced the adhesiveness of Pb arsenate. Among numerous adhesives examined, the following are noted in decreasing order of importance: commercial Fe<sub>2</sub>O<sub>3</sub>, powdered caseinglue, linseed and fish oils, finely-powdered milk. In general, best results are obtained when the adhesive forms approx. 20% of the mixture. A. G. POLLARD.

Present trend of oil sprays. E. R. DE ONG (J. Ecom. Entom., 1931, 24, 978-985).—The increasing use of oils of moderate viscosity (65-75 sec. Saybolt) and containing 85-95% of unsulphonatable matter is discussed. The repellent action of such oils to scale

insects increased with their viscosity. Pine-tar oil proved the most satisfactory preservative against bacterial decomp. for preps. emulsified with org. material (e.g., casein, blood-albumin). A. G. POLLARD.

Oil-depositing qualities of oil spray mixtures. R. H. SMITH (J. Econ. Entom., 1931, 24, 985—991).— The amount of oil deposited on sprayed trees, as judged by the amount of leaf-fall resulting, decreased with increasing proportions of blood-albumin used for emulsification. Some reduction in leaf-fall followed the use of 0.5 lb. of Ca caseinate per 100 gals. of spray, but higher proportions produced little additional effect. Insect control was of the same order as the amount of leaf-fall for oil concns. up to 1.3%, but differences appeared steadily as the oil concn. was raised to 2%. Wetting and spreading properties of oil sprays are examined and discussed. A. G. POLLARD.

Use of ammonia and ammonium salts for prevention of green mould in citrus. R. G. TOM-KINS and S. A. TROUT (J. Pomology, 1931, 9, 257–264).— Green rot in citrus produced by *Penicillium digitatum* is considerably reduced by storage in an atm. containing small concess. of  $NH_3$  such as that maintained by damp crystals of  $NH_4HCO_3$ . A. G. POLLARD.

Vacuum fumigation of pineapple planting material. H. R. HAGAN (J. Econ. Entom., 1931, 24, 1002–1012).—CS<sub>2</sub> and chloropicrin produced severe scorching of slips and crowns. HCN was non-injurious and gave efficient control of insects. A. G. POLLARD.

Fumigation problems; "protective stupefaction," its application and limitations. F. S. PRATT, A. F. SWAIN, and D. N. ELDRED (J. ECON. Entom., 1931, 24, 1041-1063).—Sub-lethal doses of HCN may cause a state of "stupefaction" in scale insects and in this condition their resistance is much increased. For successful fumigation the toxic concn. must be produced as rapidly as possible.

A. G. POLLARD.

Reproductivity of the bean weevil as affected by the vapour of ethylene oxide. G. W. HERRICK and W. R. HORSFALL (J. Econ. Entom., 1931, 24, 1084— 1086).—Exposure to non-toxic doses of ethylene oxide of adult bean weevils increased oviposition. Egg hatching was stimulated by short and decreased by long (>30 min.) exposures. A. G. POLLARD.

Red spider control in greenhouses. C. C. COMPTON (J. Econ. Entom., 1931, 24, 1094-1097).— The efficiency of rotenone preps. was markedly increased by additions of "penetrol." A. G. POLLARD.

Codling moth baits in New Mexico. J. R. EYER (J. Econ. Entom., 1931, 24, 998—1001).—The results of earlier work (Eyer and Rhodes, B., 1931, 990) are confirmed. Extracts of apple blossom and fruit were not attractive to the codling moth. Esters of malic acid were only slightly, and those of valeric acid not at all, attractive. A. G. POLLARD.

Spraying to control the codling moth in S.W. Michigan. F. SHERMAN (J. Econ. Entom., 1931, 24, 1075—1077).—Use of oil-nicotine sulphate for summer brood spraying decreased the arsenical residue on the fruit. Oil sprays alone were ineffective for summer

work and in some cases oil-nicotine sulphate preps. caused injury to fruit. A. G. POLLARD.

Effect of hydrogen-ion concentration on adsorption of mercuric chloride and its action as a seed pickle for bunt (*Tilletia tritici*). J. KRAUSS (Forts. Landw., 1930, 5, 637; Bied. Zentr., 1931, 1A, 476).—The intake of Hg from HgCl<sub>2</sub> solutions by bunt spores rises with the alkalinity of the solution, and vice versa. Similar action occurs with talcum powder and sawdust. The fungicidal action, however, increases with the acidity of the solution. Org. Hg compounds behave similarly. A. G. POLLARD.

Sulphite[-pulp] waste liquor kills certain fungi. C. W. FRITZ (Pulp & Paper Mag., 1931, 31, 1353—1354). —A number of wood-rot fungi failed to survive in media containing 20—80% of sulphite waste liquor.

A. G. POLLARD.

Fertilisers for cotton soils. J. J. SKINNER (U.S. Dept. Agric. Misc. Publ., 1931, No. 126, 9 pp.).

Rotenone, a promising insecticide, harmless to warm-blooded animals. N. SCHMITT (Wein u. Rebe, 1931, 13, 1—7).

(NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>3</sub> and mixed fertilisers.—See VII.

See also A., Jan., 37, Colorimeter for use with disc mixture. 39, Examination of soils (various). 86, Feeding of milch cows. 99, Assimilating plants and  $NH_4NO_3$ . 101, Assimilation of N by tobacco.

#### PATENTS.

Manufacture of fertilisers containing a high percentage of nitrogen. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 362,585, 8.10.30).—Conc. aq.  $NH_4NO_3$  solutions are treated with  $SO_2$  and  $NH_3$ (1:2 by vol.) to ppt.  $NH_4NO_3$ . $(NH_4)_2SO_3$ , which is removed for use as a fertiliser after, if desired, oxidation by treatment with, *e.g.*, hot air. L. A. COLES.

Insecticide. H. W. WALKER and H. S. MCQUAID (U.S.P. 1,807,078, 26.5.31. Appl., 10.5.26).—Finelypowdered coal (through 200-mesh) impregnated with 0.1-5 (1.25)% As<sub>2</sub>O<sub>3</sub> dispersed in a small quantity of an oil or grease is claimed. A. R. POWELL.

Germicide and insecticide. R. P. SOULE, Assr. to COMBUSTION UTILITIES CORP. (U.S.P. 1,809,255, 9.6.31. Appl., 11.6.24).—The material comprises a distillate of a primary tar containing at least 50% of hydroxy-acids which boil above 220°, and may be used in the form of an emulsion with soap or a neutral hydrocarbon oil, or it may be converted into alkali or alkaline-earth salts which may be used in a powder form. A. R. POWELL.

# XVII.—SUGARS; STARCHES; GUMS.

Determination of sugar losses at the diffusion battery. K. M. FRIDMAN and L. S. TVERDOKHLEBOV (Nauk. Zapiski Tzuk. Prom., 1931, 12, 375–381).— The Scheibler–Soxhlet method employing EtOH is preferred. Hot  $H_2O$  extracts pectin and gives high results. The method in which the juice is pressed from hashed pulp gives results closest to the Soxhlet method if the amount of juice in pulp is taken as 90%. Direct polarisation of diffusion  $H_2O$  and polarisation of conc.  $H_2O$  give results in accord.

CHEMICAL ABSTRACTS.

Teatini process applied to cane juice. W. C. BEDDING (Arch. Suikerind. Nederl.-Indië, 1931, 39, 1041—1049).—Raw cane juice was heated to  $60-65^{\circ}$ and limed to give a  $p_{\rm H}$  of about 12; 1 kg. of liquid SO<sub>2</sub> was added in about 45 sec., after which followed carbonatation. The ppt. resulting gave better filtration compared with ordinary procedure, making it possible to reduce the quantity of CaO by about 15%; the colour of the filtrate, however, was darker than usual, and the press-cake had a distinctly greenish colour. Further, the amount of Ca salts in the clarified juice and syrup was increased. J. P. OGILVIE.

Influence of invert sugar on the removal of amino-acids in the carbonatation of beet juices. V. KONN (Z. Zuckerind. Czechoslov., 1931, 56, 66—72).— Experiments are described demonstrating that in the carbonatation of beet juices  $\mathrm{NH}_2$ -acids are not removed by combining with invert sugar. J. P. OGILVIE.

[Sugar-juice] carbonatation studies. IV. Influence of the final alkalinity. L. DOSTÁL (Z. Zuckerind. Czechoslov., 1931, 56, 169–176; cf. B., 1931, 1151).—There exists an almost linear relationship between the total alkalinity of the unfiltered firstcarbonatation juice and its rate of filtration, which latter effect appears to be independent of the mode of defecation used. J. P. OGILVIE.

Studies on [sugar-juice] carbonatation processes in which the scums are returned. III. Comparison of ordinary carbonatation with the Pšenička process. L. Dostál (Z. Zuckerind. Czechoslov., 1931, 56, 189–193).—Semi-technical experiments showed the Pšenička process, as compared with ordinary carbonatation, to give distinctly the lighter-coloured clarified juice, though the purity, N, and ash remained about the same. Sweetening-off of the scums was also facilitated and the rate of filtration improved.

#### J. P. OGILVIE.

Use of sodium bisulphite for juice sulphitation. V. STANĚK (Z. Zuckerind. Czechoslov., 1931, 56, 142— 144).—As a means of lowering the alkalinity of the clarified juice, the addition of 0.01% of commercial Na bisulphite should be made before boiling up previous to going to the evaporator. Besides eliminating Ca salts, the colour is much decreased. The cost of this mode of sulphiting compared with that using S-oven gases depends on the % of the latter actually taken up by the juice, which varies very widely. When the gas utilisation is  $\geq 21.5\%$ , Na bisulphite addition would be the cheaper method. J. P. OGILVIE.

Composition of a deposit on the clarified juice strainer. H. W. VAN DER MAREL (Arch. Suikerind. Nederl.-Indië, 1931, 39, 1050—1051).—A slime accumulating on the clarified juice strainers was found to consist mainly of  $Ca_3(PO_4)_2$ , which had been pptd. from the clarified juice probably as the result of the presence of unslaked CaO in the filter-cake of the scum presses.

J. P. OGILVIE. Refining sugar from thick beet juice. G. Y. TARASHCHANSKI and I. I. VDOVICHENKO (Nauk. Zapiski Prom., 1931, 12, 71-84).—The juice (d 1.23), after evaporation, is filtered and then boiled in vac. pans, ultramarine and hydrosulphide being added. The  $p_{\rm H}$  of the massecuite and run-offs is about 7.5.

CHEMICAL ABSTRACTS.

Manufacture of beet sugar. Y. D. LYUBCHENKO (Sovet. Sakhar, 1931, No. 9–10, 10–13).—A procedure is described. CHEMICAL ABSTRACTS.

Influence of mixing-in other sugars on the affinability. A. DOLINEK (Z. Zuckerind. Czechoslov., 1931, 56, 93—95).—Determination of the affination no. of different mixtures of raw sugars of different affinabilities showed the unfavourable effect of the practice of mixing together raw sugars of varying physical properties. Raw sugars of good quality should be treated separately. J. P. OGILVIE.

Vac.-pan circulation.—See I. Fermentation of molasses.—See XVIII. Molisch reaction for sugars. Sugar beet as nutrient.—See XIX.

See also A., Jan., 26, 47, and 91, Hydrolysis of starch. 91, Starch-liquefying power. 93, Gum production by *Azotobacter chroococcum*. 100, Enzymic decomp. of starch.

#### PATENT.

#### Adding reagents to [sugar] solutions.—See I.

#### XVIII.—FERMENTATION INDUSTRIES.

Determination of the total nitrogen in yeast. R. S. W. THORNE (J. Inst. Brew., 1932, 38, 23-29).-The determination of N in yeast by the Kjeldahl-Gunning process, in which K2SO4 and CuSO4 are present during the digestion with  $H_2SO_4$ , gave a val. lower than the true val. as obtained by Dumas' standard method. In many cases the addition of KMnO<sub>4</sub> during digestion gave increased vals. which approximated closer to those obtained by Dumas' process, but in other cases errors amounting to 10% of the total N were present, and the determination of N by this method appeared to be influenced by the type and stage of growth of the yeast under analysis. The most suitable method was found to be a modification of that of Kjeldahl, due to Christensen and Fulmer, in which the yeast was treated with a fixed amount of H<sub>2</sub>O<sub>2</sub> and H<sub>2</sub>SO<sub>4</sub> before the usual digestion with  $H_2SO_4$ . It yielded a val. for the total N in yeast which lay within 1% of the true val. C. RANKEN.

Influence of adsorption agents on the fermentation of molasses. B. LAMPE (Z. Spiritusind., 1931, 54, 313—314).—The addition of 0.1 vol.-% of various types of "Hiagenit" (activated charcoal) activated the fermentation of both beet- and cane-sugar molasses. After a fermentation of 48 hr. without aeration, 99.3— 100% of the theoretical yield of EtOH was obtained accompanied by a decrease in the content of MeCHO, which varied slightly with the different types of C.

C. RANKEN.

British Chemical Abstracts-B.

wort and beer retain less resins with decreasing  $p_{\rm H}$ , though the decrease during fermentation becomes slightly less. The max. dissolution during boiling and min. loss during fermentation is attained with 3 hrs.' boiling. Worts of decreasing concn. dissolve more resins, but more is lost from weak worts during fermentation. A high proportion of coagulable protein in the wort removes more resins; during subsequent fermentation the difference is lessened owing to greater removal of resins from the more conc. solutions. Increased buffering favours both dissolution during boiling and retention of resin during fermentation. Though larger additions of resins increase the amounts subsequently found in both worts and beers, the proportion of the resins retained decreases with increasing additions of resins. Of the resin fractions, the  $\alpha$ -bitter acid (humulone) is retained in the greatest relative amount; its relative solubility is about 6 times that of the remaining constituents. With old hops, though the amount of  $\alpha$ -bitter acid is less, more resin dissolves, hence it appears that the alteration products of the bitter acids have a relatively higher solubility in wort and beer than the acids themselves. About 25% of the resin used is found in the wort and 20% in the beer.

F. E. DAY.

Manioc for brewing purposes. STAIGER (Z. Spiritusind., 1931, 54, 322) .- Manioc and its modifications tapioca and sago are prepared from cassava root, the approx. composition of which is H<sub>2</sub>O 11.3, protein 1.3, fat 0.3, starch 73.6, sugar 0.6, fibre 2.0, and ash 1.8%. To ensure efficient saccharification of the manioc and good yields of EtOH, it is necessary to gelatinise the manioc at 100° for at least 2 hr. prior to its hydrolysis. A max. yield of 45.6 c.c. of EtOH per 100 g. is obtained by steaming the manioc for 1-2 hr. at 2-3 atm. pressure followed by saccharification with 10% of malt at 55°, rising later to 62°. It is considered that it is not permissible to use manioc in Germany for the manufacture of EtOH. C. RANKEN.

"Kvass": its origin and composition. W. SIEFERT (Oesterr. Chem.-Ztg., 1931, 35, 2-4).--"Kvass," a mildly alcoholic, slightly acid, beer-like beverage prepared by Austrian peasants by fermentation of bread or various cereal grains, with or without addition of sugar and press-yeast, had its origin in Russia. The acidity is caused by subsidiary fermentation processes, whereby AcOH, lactic acid, and mannitol are produced, the last-named being further decomposed by a rod-like bacterium into  $HCO_2H$  and more AcOH and lactic acid. Fermentation with a pure wine yeast gives a liquor containing more EtOH and less acid.

W. G. EGGLETON. Determination of sulphurous acid in beer by the official method. F. MENDLIK (Chem. Weekblad, 1931, 28, 714).—The sample required (100 c.c.) is too small to permit an accurate determination, and in addition the other volatile compounds present interfere. The errors of the titrimetric method are demonstrated.

H. F. GILLBE. Clarification of wines by potassium ferrocyanide. D. COSTA (Annali Chim. Appl., 1931, 21, 526-534).— The efficacy and wholesomeness of this process for wines containing excess of Fe salts is confirmed. A

summary of the regulations in force at present in Germany, Austria, and Hungary in connexion with this practice is given. O. F. LUBATTI.

Acid substances entering into the composition of wines. II. Volatile acids. L. SEMICHON and M. FLANZY (Ann. Falsif., 1931, 24, 516-534; cf. B., 1930, 389).-In determining the volatile acidity of a wine, 50 c.c. of the sample are saturated with Ca(OH)2 and conc. to approx. 25 c.c. to drive off EtOH. The cooled liquid is acidified with large excess of conc. aq. tartaric acid and filtered after 1 hr., the filter being washed 4 times with 5 c.c. of boiled distilled H<sub>2</sub>O. Filtrate and washings are made up to 50 c.c. and 10 c.c. of this solution are heated on the water-bath to drive off CO2 and then steam-distilled by the method of Blarez ("Vins et spiriteux," edn. 1908, 131) until 50 c.c. of distillate show no acidity to litmus. The distillate is titrated with lime-water in presence of litmus to a distinct blue, or the authors' standard method (B., 1930, 389) is used. If the volatile acids present as esters are to be determined along with the free volatile acids and those present as salts, the sample, after saturation with Ca(OH), is heated under reflux for 1 hr. If necessary, corrections are made for the presence of SO<sub>2</sub>, salicylic acid, and lactic acid. Conditions affecting the completeness of removal of the volatile fatty acids and contamination of the distillate with lactic acid are discussed. A method of determining HCO<sub>2</sub>H, AcOH, propionic and butyric acids, respectively, in presence of each other is based on selective oxidation of these by K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> under different conditions. W. J. BOYD.

Coloured standard solution for the Barbet test. B. LAMPE (Z. Spiritusind., 1931, 54, 319–320).— Reducing impurities in spirit are determined by the Barbet test in which 50 c.c. of the spirit are mixed at 18° with 2 c.c. of aq.  $KMnO_4$  (0.2 g. per litre) and the time is determined for the colour of the liquid to change from red-violet to pale salmon. The difficulty of an unstandardised coloured end-point has now been removed by the provision for comparison of a stable, standard salmon-coloured solution which is prepared from a mixture of inorg. salts. With its use, the time readings by independent observers vary by < 1 min. for the same spirit. C. RANKEN.

See also A., Jan., 45, Formation of dextran by Leuconostoc mesenterioides. 47 and 91, Diastatic hydrolysis of starch. 90, Enzymes (peroxidase) and light. Determination of starch-liquefying power. Malt amylases. Prep. of invertase. 92, Proteolytic enzymes. Histaminase. Enzymic production of esters. Glucosulphatase. Pure soya-urease. 93, Production of itaconic acid and mannitol. Aspergillus oryzæ enzymes. Gum production by Azotobacter chroöcoccum. 100, Enzymic decomp. of starch.

#### PATENT.

Therapeutic extracts.—See XX.

# XIX.—FOODS.

Molisch reaction for sugars. Detection of sucrose in milk. B. ROMANI (Annali Chim. Appl., 1931, 21, 535-538).-Neumann's modification of the Molisch

reaction gives a strong coloration with sucrose. A drop of milk is treated with one drop of 20% alcoholic solution of  $\alpha$ -naphthol followed by 3 c.c. of conc. HCl and boiled for 3—4 min. After 5 min. the mixture is shaken with 2 vols. of CHCl<sub>3</sub>. In presence of 3—5% of sucrose the milk shows a decided violet colour and the CHCl<sub>3</sub> extract becomes pink. Alternatively, 1.5 c.c. of serum, 2 drops of 20% alcoholic  $\beta$ -naphthol, and 4 c.c. of conc. HCl are treated as above and shaken with 4 vols. of CHCl<sub>3</sub>. The serum containing sucrose gives a deep green colour, the CHCl<sub>3</sub> extract becomes green.

O. F. LUBATTI. Relation of "feathering" and heat-stability of cream to fat clumping produced by homogenisation. F. J. DOAN (J. Dairy Sci., 1931, 14, 527-539). -Mechanical destruction of fat clumps increases the heat-stability of homogenised cream. It is suggested that fat clumps accelerate the separation of casein by acting as centres of coagulation. Double homogenisation is a better method for stabilising cream than the addition of salts. Addition of milk solids (e.g., condensed skim-milk) improves stability in some cases, but excessive amounts have the reverse effect. Cream should not be homogenised at temp. below that of preheating unless the latter is  $> 76.5^{\circ}$  for 30 min. Salt stabilisers (e.g., Na citrate) should be added prior to homogenisation, since they act both by correcting the salt balance and by preventing the formation of fat clumps. The tendency of cream to "feather" in coffee is paralleled by the degree of fat clumping. A. G. POLLARD.

Effect of single and double homogenisation of cream on coagulation by heat and by rennet and on separation of the fat. B. H. WEBB (J. Dairy Sci., 1931, 14, 508—526).—Homogenisation of cream lowered its stability to coagulation by heat or by rennin, according to the temp. and pressure used. A second homogenisation with pressures up to 500 lb. increased the stability of creams containing > 15% of butter fat, but with > 2000 lb. pressure stability was reduced. Homogenisation facilitated the separation of fat from sterilised sweet cream in proportion to the pressure used. Re-homogenisation enhanced this effect.

A. G. POLLARD.

(A) Distribution of solid matter in thick and thin egg-white. (B) Measurement of deterioration in the stored hen's egg. W. F. HOLST and H. J. ALMQUIST (Hilgardia, 1931, 6, No. 3, 45-48, 49-60).---(A) The gradual liquefaction of the white of eggs during storage is not accompanied by changes in the % of solids or in refractive index. Measurements of the latter val. offer a rapid means of determining the total solids in egg-white. (B) Separation of "thick" and "thin" white of egg is effected by pouring the white over a sieve having 9 meshes to the in. The vol.-% of "thick" white is a better index of freshness than is the "yolk index" of Sharp and Powell (B., 1930, 1089). A. G. POLLARD.

Tchakva (Batoum) tea. L. GOBERT and (MME.) S. GOBERT (Ann. Falsif., 1931, 24, 542-545).--Methods and results of analysis of tea from Tchakva are given. W. J. BOYD.

Determination of caffeine in coffee and coffee extracts. A. BONN and C. DESGREZ (Ann. Falsif.,

1931, 24, 546-547).-The caffeine solution (75-100 c.c.) is evaporated to dryness on the water-bath and the residue mixed with 15 g. of freshly calcined MgO. After keeping for 1 hr. at room temp., the mixture is placed in a dry flask with 125 c.c. of dry CHCl<sub>3</sub> and heated under reflux for 45 min. Any CHCl<sub>3</sub> lost is exactly replaced and the cooled liquid is filtered, the vol. of liquid collected being noted. The CHCl<sub>3</sub> is evaporated off and the dry residue redissolved in a few c.c. of CHCl<sub>3</sub> and transferred to a beaker. The solvent is driven off and the residue taken up in 10 c.c. of 5% HCl. Sufficient 5% SiO2,12WO3 is added to ppt. the caffeine (25-30 drops for 0.01 g. of hydrated caffeine), and the mixture is warmed until the ppt. dissolves and left for 24 hr. The ppt. is then filtered off, washed with 5% HCl, dried, ignited, and weighed (1 g. of  $SiO_2, 12WO_3 \equiv 0.2236$  g. of hydrated caffeine). The method can be used for the determination of caffeine in W. J. BOYD. urine.

Cooking. I. Chemical changes of carbohydrates in the sweet potato according to various methods of cooking. O. SINODA, S. KODERA, and C. OYA (Biochem. J., 1931, 25, 1973—1976).—There is a loss of  $H_2O$  after cooking, as well as a decrease in sol. sugars and dextrins. The starch fraction always increases in cooking. There is an increase in digestibility after boiling and steaming. Owing to the action of heat and  $H_2O$  the starch is partly hydrolysed, whilst sol. sugars and dextrins polymerise to some extent. S. S. ZILVA.

Effect of leaching on the nutritive value of forage plants. H. R. GUILBERT, S. W. MEAD, and H. C. JACKSON (Hilgardia, 1931, 6, No. 1, 13—26).—Leaching losses from a number of forage plants and straw consisted of SiO<sub>2</sub>-free ash constituents (principally Cl with some P and Ca), N-free extractives (chiefly sugars), and small proportions of crude protein.

A. G. POLLARD.

Soya-bean investigations [Germany], 1930. W. RIEDE and B. REWALD (Landw. Versuchs-Stat., 1931, 113, 83—92).—Numerous analytical data are presented in an examination of the soya bean as a green fodder, hay, silage, and oil-seed crop.

#### A. G. POLLARD.

Starch equivalent of maize silage. G. FINGER-LING, K. SCHMIDT, and B. HEINTZSCH [with P. EISEN-KOLBE, M. JUST, and F. KRETZSCHMANN] (Landw. Versuchs-Stat., 1931, 113, 103-120).—The starch equiv. of silage from maize grown in a wet year was lower than for that grown in a dry year. (Cf. B., 1931, 1120.) A. G. POLLARD.

Nutrient value of sugar beet. G. FINGERLING (Landw. Versuchs-Stat., 1931, 113, 1--68).-In a comparison of dried sugar beet with ordinary dried slices from the sugar factories, the higher nutrient val. of the former in respect of digestible org. matter and N-free extractives is chiefly attributable to its sugar content. Starch vals. of the two materials are similar. The digestibility of dried beet for pigs was notably high. Beet, whether dried or fresh, had a higher milkproducing val. than the slices, increased yields being accompanied by increased proportions of dry matter

(chiefly lactose and minerals), but somewhat lower fat content. A. G. POLLARD.

Examination of sugar-beet leaves [as a feedingstuff]. T. REMY [with A. DHEIN, F. MEYERS, and E. OHLY] (Landw. Jahrb., 1931, 74, 405-446).--Washing of beet leaves for feeding purposes is unnecessary unless badly fouled or rotting. Best silage is obtained from chopped fresh leaves containing 80-85% $H_2O$ . Washed leaves should be pressed to remove excessive moisture prior to filling into the silo. Various methods of preparing material for ensilage are discussed.

A. G. POLLARD.

Manipulation of distillation methods for determination of water content. O. SCHIMON (Chem.-Ztg., 1931, 55, 982–983).—Attention is directed to the advantages of  $C_2Cl_4$  as the distilling liquid in preference to  $C_2H_2Cl_4$ . The results of comparative trials on rye meal and tobacco are tabulated.

E. S. HEDGES.

See also A., Jan., 71, Prep. of glutenin and of gluten proteins. Analysis of proteins. 84, Dietary properties of soya-bean "milk" and cow's milk. Vegetable proteins. 97, Vitamin-C in potatoes.

# PATENTS.

Table jellies [in divided blocks]. M. COHEN, and ROWNTREE & CO., LTD. (B.P. 364,250 and 364,753, 13.11.30).

Drying [of fish etc.].—See I. Vitamin-rich foodstuffs.—See XX.

#### XX.-MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Deterioration of certain medicaments under the influence of light. H. V. ARNY, A. TAUB, and A. STEINBERG (J. Amer. Pharm. Assoc., 1931, 20, 1014— 1023, 1153—1158).—The degree and rate of deterioration of 35 medicaments and chemicals, stored under varying conditions of exposure to light, and in 8 types of commercial glass containers (flint, green, blue, and amber), have been measured. Spectrophotometric transmission curves of these glasses and also of 5 Corning glass filters, samples of Coeburgh green, red flash, and window-pane glasses are given. Direct sunlight is 3—12 times as destructive to light-sensitive chemicals as is diffused light, which is only slightly more active than datkness. Protective action of various stabilising catalysts has been examined. E. H. SHARPLES.

Simplified Fromme method for determination of alkaloids in Solanaceæ drugs and Coca leaves, and determination of alkaloids in Ephedras. W. PEYER and F. GSTIRNER (Pharm. Ztg., 1931, 76, 1440—1442).—10 g. of the drug are macerated for  $\frac{1}{2}$  hr., with frequent shaking, with 100 g. of Et<sub>2</sub>O and 7 g. of 10% aq. NH<sub>3</sub> and then filtered. The residue is washed with a few c.c. of H<sub>2</sub>O, a further 5 c.c. are added to the filtrate, and the whole is shaken vigorously. After keeping, 60 g. of the clear Et<sub>2</sub>O solution are evaporated and the residue, after weighing, is dissolved in 10 c.c. of Et<sub>2</sub>O, 25 c.c. of HCl (0.25%) are added, the mixture is shaken, and the Et<sub>2</sub>O distilled off. After cooling, the wt. of the liquid is brought to 30 g. by addition of 1% HCl and, after shaking with 0.5 g.

of talc, is filtered. 25 g. of the filtrate mixed with 8 drops of 10% aq. NH<sub>3</sub> are extracted with 20, 15, and 10 c.c. of CHCl<sub>3</sub> (2 min. each extraction), the combined extracts are filtered, and the filtrate is evaporated. The residue is dissolved in 10 c.c. EtOH and 25 c.c. H<sub>2</sub>O and titrated with 0.1N-HCl to methyl-orange; then no. of c.c. of 0.1N-NCl  $\times 0.578 = \%$  hyoscyamine. This method is recommended for Solanaceæ drugs, and modifications of it for the determination of alkaloids in Folia Coca, F. Jaborandi, and Rhizoma Gelsemii are described. Emulsion formation is minimised and the time thereby shortened. A modification of the Krishna and Ghose method (J.S.C.I., 1929, **48**, 67 T) for the determination of ephedrine in ephedras, in which the extraction is conducted with Et<sub>2</sub>O alone, is described.

E. H. SHARPLES.

Factors influencing stability of fluid extract of ergot. M. I. SMITH and E. F. STOHLMAN (J. Pharm. Exp. Ther., 1931, 43, 621–635).—The rate of deterioration of extracts of ergot is increased when the temp. is raised and is very slow at 0°. At 50° it is increased when oxidising and decreased when reducing agents are added. The stability does not vary between  $p_{\rm H} 2 \cdot 2$  and 5 \cdot 2. W. O. KERMACK.

Detection of cocaine in presence of novocaine by means of cobalt thiocyanate. J. L. Young (Amer. J. Pharm., 1931, 103, 709-710). 4-5 drops of a 2%aq. solution of Co(CNS)<sub>2</sub> are added to a small quantity of the sample; if cocaine only is present a Prussianblue-coloured ppt. is formed which remains undissolved on addition of 4-5 drops of a (freshly prepared) 10% solution of SnCl<sub>2</sub> in HCl (1:1). If only novocaine is present the entire solution turns blue and on the addition of SnCl<sub>2</sub> the ppt. dissolves, forming a pink solution. Lactose, NHPhAc, morphine, and ecgonine salts do not interfere, but codeine and diacetylmorphine salts must be absent. E. H. SHARPLES.

Tinctures of cinchona. III. Acetone extract from cinchona bark and its relation to the stability of tinctures of cinchona. A. LICHTIN (Amer. J. Pharm., 1931, 103, 702-708; cf. B., 1931, 742).— The COMe<sub>2</sub> extract of the bark has been separated into three components the reactions of which are described. A tincture prepared from COMe<sub>2</sub>-extracted bark and kept in an alkali-free bottle showed no discoloration or pptn. after 1 year. COMe<sub>2</sub> extracts a large proportion of the hydrolysable tannins, which are largely concerned in the deterioration, and probably the content of hydrolysable material in the tincture bears a relationship to its stability. E. H. SHARPLES.

Removal of nicotine from tobacco smoke while smoking. R. KISSLING (Chem.-Ztg., 1932, 56, 31).—A commercial prep. consisting of 0.6% of FeSO<sub>4</sub>,(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>,6H<sub>2</sub>O with a little tartaric and citric acid in 96.6% of H<sub>2</sub>O and 2.73% of EtOH is shown to be useless when injected into cigars. E. S. HEDGES.

Formosan eucalyptus oil. A. R. PENFOLD (Perf. Ess. Oil Rec., 1931, 22, 402).—This oil remains unchanged when 2 c.c. are treated with 5 c.c. of a 2% solution (by vol.) of Br in AcOH. Genuine oil distilled from a eucalypt produces a purple colour if crude and a pale green to brown if rectified. Two samples of the

Formosan oil examined had  $d_{15}^{15}$  0.9220—0.9224,  $\alpha + 3.7^{\circ}$  to  $+4.7^{\circ}$ ,  $n_{D}^{\circ}$  1.4606—1.4617, congealing point —8° to —10.5°, and contained 72—85% of cincole. One sample contained an appreciable amount of safrole. E. H. SHARPLES.

Indian ephedras. S. KRISHNA and T. P. GHOSE (Indian Forest Records, 1931, 16, Pt. II, 1-32).—See J.S.C.I., 1929, 48, 67—70 т.

Caffeine in coffee.  $H_2O$  content [of tobacco].— See XIX.

See also A., Jan., 52, o-Alkoxybenzhydrylamines. 54, Syntheses of adrenaline and related compounds. 55, Amidines of pharmacological interest. 60, Flavouring substances of horseradish and black radish. 66, Hg compounds of 5-phenyl-5-ethylbarbituric acid. 68, Lobinine. 69, Au derivatives of 2-thiolbenziminazole-5-arsinic acid. 70, Arylthioarsinites. 72, Determination of alkaloids. 88, Physostigmine analogues. Digitalis preps. Detection and determination of lobeline. 94, Stable diphtheria toxin. 95, Prep. of vaccine virus. Conc. anti-vaccinia serum. Extraction of insulin. Acetylation of cryst. insulin. 96, Insulin preps. Sexual hormones. 97, Hormone preps. Vitamins (various). 99, Properties of alkaloids and climatic conditions. Alkaloidal content of Hyoscyamus niger. Pyrethrum flowers. 101, Alkaloids of Lunasia amara and of Chinese gelsemium.

#### PATENTS.

Preparing a [soluble] calcium-carbohydrate compound. E. STERN (U.S.P. 1,807,608, 2.6.31. Appl., 2,10.26. Ger., 12.10.25).—A solution or suspension of carbohydrates, preferably containing a sugar, is gradually treated with  $Ca(OH)_2$  at moderate temp. and the product is evaporated *in vacuo*. If necessary, it is neutralised by adding an acid salt.

E. H. SHARPLES.

Production of extracts serving for therapeutic purposes and for increasing the vitamin content of foodstuffs. MATRO GES.M.B.H. (B.P. 362,023, 11.9.30. Ger., 12.9.29).—Rootlets obtained in malting (cf. B.P. 328,942; B., 1930, 686) are extracted with EtOH or a fat solvent, the extract is saponified, and, after removal of soap by pptn., the liquid is conc. and again extracted to give an extract from which the vitamin-rich substances are removed by crystallisation. All operations following the removal of soap are conducted in an O<sub>2</sub>-free atm. E. H. SHARPLES.

Production of alkyl ethers of alkaloids. Boor's PURE DRUG Co., LTD., F. L. PYMAN, and H. H. L. LEVENE (B.P. 359,180, 4.10.30).—Harmol or harmalol is alkylated with an alkyl salt above  $C_1$  in presence of caustic alkali, preferably in H<sub>2</sub>O or EtOH. The Pr<sup>a</sup> (m.p. 204 · 5°; B,HCl, m.p. 259—261°), Pr<sup>g</sup> (m.p. 180 · 5—181°; B,HCl, m.p. 277 · 5°), Bu<sup>a</sup> (m.p. 220°; B,HCl, m.p. 232—234°; Rideal-Walker coeff. 350), *n*-heptyl (m.p. 131—132°; B,HCl, m.p. 228°), *n*-dodecyl (m.p. 119—120°; B,HCl, m.p. 208—208 · 5°), β-diethylaminoethyl (m.p. 295°; B,HCl, m.p. 167—168°), CH<sub>2</sub>Ph (m.p. 213°; B,HCl, m.p. 257°), *n*-nonyl (m.p. 114°; B,HCl, m.p. 205—207°), and *n*-amyl (m.p. 206—207°; B,HCl, m.p. 192—194°) ethers of harmol, and the Bu<sup>a</sup> (m.p. 173°; B,HCl, m.p. 213 $\cdot$ 5°) and Pr<sup>a</sup> (m.p. 196—197°; B,HCl, m.p. 232—234°) ethers of harmalol are described. C. HOLLINS.

Smoking tobacco. D. H. WIGGINS (U.S.P. 1,808,707, 2.6.31. Appl., 7.1.29).—SiO<sub>2</sub> gel is either mixed with the tobacco or arranged as a capsule in the path of the smoke, to absorb nicotine. E. H. SHARPLES.

Separating the active substances of the posterior lobe of the hypophysis. I. G. FARBENIND. A.-G. (B.P. 362,860, 8.9.30. Ger., 7.9.29).—Solutions containing such active substances are subjected to a limited or fractional adsorbtion, preferably with  $SiO_2$  or a silicate and in a feebly acid solution. The substance that increases the blood pressure is removed from the adsorbent by means of a dil. alkaline solution; that substance acting on the uterus remains in solution.

#### E. H. SHARPLES.

Purification of extracts of sexual hormone-likeacting substances. SCHERING-KAHLBAUM A.-G. (B.P. 362,019, 11.9.30. Ger., 27.9.29. Addn. to B.P. 343,779; B., 1931, 515).—The MeOH is replaced wholly or partly by other  $H_2O$ -sol. org. liquids in which the hormones are sol., and the light petroleum by other non-miscible org. liquids in which the hormones are insol.

E. H. SHARPLES.

Manufacture of arsinic acids. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 359,610, 11.8.30).— 4-Nitropyrocatechol carbonate is condensed with a suitable amine so as to give a 5-nitro-2-hydroxyphenyl carbamate, *e.g.*, with NHEt<sub>2</sub> (m.p. 162°), piperidine, hydrazine, etc., which is reduced to amine, converted by the diazo reaction into the arsinic acid, and finally hydrolysed with HCl to pyrocatechol-4-arsinic acid.

C. HOLLINS.

X-Ray photographs.—See XXI. Disinfection etc. —See XXIII.

# XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Variation of photographic sensitivity with different light sources. R. DAVIS and G. K. NEELAND (Bur. Stand. J. Res., 1931, 7, 843—850).—The ratio of the sensitivity of panchromatic plates to that of the ordinary plates is greater with incandescence lighting than with sunlight, and the panchromatic plates are less sensitive to such lighting than to sunlight. Approx. factors for adapting the exposure to the type of illuminant are given. E. S. HEDGES.

See also A., Jan., 29, Micellar theory of the latent image. Photographic unit of intensity. Sensitivity. Combined photographic effects of cathode rays, X-rays, and other radiations.

#### PATENTS.

Production of X-ray photographs. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 363,346, 9.7., 6.8., and 15.8.30).—X-Ray photographs of hollow organs of warm-blooded organisms are made by filling these organs with a solution of Na iodo- or di-iodo-methanesulphonate (cf. B.P. 353,477; B., 1931, 1132). Aliphatic residues may be substituted in the mol.

J. LEWKOWITSCH.

(A) [Taking and projecting colour-record photographs in] colour photography. (B, c) Taking colour-record photographs on sensitised layers provided with lenticular elements. C. NORDMANN (B.P. 363,387, 363,409, and 363,447, [A] 17.9.30, [B] 18.9.30, [c] 14.10.30. Fr., [A] 17.9.29, [B] 19.9.29, [c] 15.10.29. [c] Addn. to [A]).

# XXII.—EXPLOSIVES; MATCHES.

Determination of the constituents of methane used in testing galleries [for explosives]. K. NIEDERER (Z. ges. Schiess- u. Sprengstoffw., 1931, 26, 257-261).—The bulk of the  $CH_4$  is first separated from  $H_2$ ,  $N_2$ ,  $O_2$ , and CO by condensation with liquid air, the remaining constituents, viz.,  $C_2H_2$ ,  $C_2H_4$ ,  $C_2H_6$ ,  $C_3H_6$ ,  $C_3H_8$ , and  $C_4H_{10}$ , being separated by fractional distillation. The apparatus and method are described, and details of the determination of the various separated components are given. W. J. WRIGHT.

Determination of moisture in blasting and propellent explosives and similar materials. W. FERMAZIN (Z. ges. Schiess- u. Sprengstoffw., 1931, 26, 370-372).-Notevarp's method (A., 1930, 560) gives high results with explosives containing residual solvent, as ethoxides are formed. Tests with BaCl<sub>2</sub> (14.76%)  $H_2O$ ) by Dupré's method gave 10.63-12.07% when a layer of ignited sand was placed between the  $BaCl_2$ and the  $CaC_2$ , 12.56-13.08% when fine wire mesh was used instead of sand, and 13.96-14.72% when the H<sub>2</sub>O level in the bath was so adjusted that only the lowest part of the CaC<sub>2</sub> layer was below this level, the exothermic formation of C<sub>2</sub>H<sub>2</sub> being thereby unaffected. Disadvantages of Dupré's test are the difficulty of maintaining the H<sub>2</sub>O level, and decomp. of the Ca(OH)<sub>2</sub> formed. In a modified test, the substance is heated in a current of dried N<sub>2</sub> and the vapours evolved are conducted into CaC<sub>2</sub>, the C<sub>2</sub>H<sub>2</sub> produced being passed into ammoniacal Cu<sub>2</sub>Cl<sub>2</sub> solution. The pptd. Cu<sub>2</sub>C<sub>2</sub> is dissolved in 24% HCl, the solution evaporated, and the residue, after treatment with  $H_2O$  and dil.  $H_2SO_4$ and subsequent addition of excess of H<sub>2</sub>SO<sub>3</sub>, pptd. with cuprammonium thiocyanate. Results obtained with BaCl<sub>2</sub>, EtOH (75%), Et<sub>2</sub>O, and nitrocellulose powder W. J. WRIGHT. are reported.

Improved Taliani stability test for nitrocellulose. J. GOUJON (Z. ges. Schiess- u. Sprengstoffw., 1931, 26, 217-221, 261-264, 289-293, 330-333, 361-365, 400-403; cf. B., 1931, 995).-Incomplete expulsion of H<sub>2</sub>O from the nitrocellulose in the Taliani test causes irregularities; decarbonated CP<sub>2</sub>, on account of its greater hygroscopicity, decomposes more rapidly than CP<sub>1</sub>, and in carbonated samples the chalk, in presence of moisture, has a stabilising effect. The nitrocellulose need not be dried, either at 40° or 100°, in a current of air. It may be dried at  $100^{\circ} \pm 5^{\circ}$  for 2 hr. in the tube of the apparatus without risk of H<sub>2</sub>O absorption if, after drying, the tube is at once attached to the apparatus and the side tap kept closed till the bath temp. is  $135^{\circ}$ ; no CaCl<sub>2</sub> tube is required. Dry CP<sub>1</sub> decomposes more rapidly than dry CP2. A description of the apparatus for the modified test is given ; aq. glycerin is used in the bath. The side tap is kept open for 30 min. to establish

equilibrium, and the duration of the test is timed from the introduction of the sample. It is unnecessary to prolong the decomp. to a pressure of 300 mm., since at 100 mm. there is sufficient differentiation of nitrocelluloses. Experiments with wet nitrocellulose showed that in presence of chalk Vieille's test (110°) is without significance. On the other hand, in the modified Taliani test the chalk does not act as a stabiliser, and a true measure of the stability is obtained. Although dried, carbonated nitrocellulose appears to show a slightly reduced stability as compared with the decarbonated material, which may be explained by the formation of  $Ca(NO_2)_2$ , further experiments showed that it is preferable not to remove the chalk, as that involves a slight purification of the nitrocellulose. Curves denoting the decomp. of  $\rm CP_1$  and  $\rm CP_2$  at 110–135° showed uniform increase in pressure. An investigation was made of the effect on the stability of nitrocellulose of the degree of beating, the number of washings, alkali washing, and boiling, and a scheme of purification was evolved. It is shown that it is unnecessary to renew the H<sub>2</sub>O after each boiling, that the nitrocellulose should be boiled with acid H<sub>2</sub>O until the acidity reaches a min. and then boiled with alkali, and that the time of boiling should be governed by the fineness of the material. It is possible to stabilise highly nitrated cellulose in the form of flocks. W. J. WRIGHT.

Decomposition of explosives on detonation. A. HAID and A. SCHMIDT (Z. ges. Schiess- u. Sprengstoffw., 1931, 26, 253-257, 293-298).-The nature of the reaction products and the degree of their dissociation are considered for three classes of explosives : (1) those with an O balance, ensuring complete combustion of C and  $H_2$ ; (2) those containing only sufficient O for conversion of the C into CO and  $CO_2$ ; and (3) those with a deficiency of O, resulting in the formation of CH<sub>4</sub> and other hydrocarbons or, if the explosive contains N, of CH4, C2N2, HCN, and NH3. Figures are given for the increase in the CO<sub>2</sub>: CO ratio with rising pressures of 50-50,000 atm., and for the decrease with temp. rising from 2000° to 4000°. The experimental methods are described, and the composition of the explosion gases, heat of explosion, explosion temp., sp. gas vol., and CO<sub>2</sub>: CO ratio are tabulated for trinitrotoluene, picric acid, tetryl, guncotton, nitropentaerythritol, and blasting gelatine, initiated with various compositions.

W. J. WRIGHT.

Detonation of explosives. HAID, GOETZE, SELLE, KOENEN, SCHMIDT, and BECKER (Jahresber. Chem.tech. Reichsanst., 1930, 8, 124—129; Chem. Zentr., 1931, ii, 178).—In detonation with heavy tamping in a Pb block, the work done by the gases results in rapid cooling; the composition of the gases is not markedly altered. The fall in detonation velocity resulting from long storage is due not to chemical changes, but to physico-mechanical changes in the explosive mixture. A. A. ELDRIDGE.

Determination of detonation velocity. W. FRIEDERICH (Z. ges. Schiess- u. Sprengstoffw., 1931, 26, 221-222).—To determine the velocity of the detonating fuse, a short length and an 8-m. length are attached at one end to a detonator, their other ends being inserted in narrow borings in a steel block fixed over a

revolving drum so that the open ends of the borings are close to the drum surface. After detonation, the distance between the two marks on the drum is measured, from which and from the known speed of the drum the velocity of the fuse is calc. Results with pentaerythritol tetranitrate fuse are tabulated.

W. J. WRIGHT.

[Detonation] velocity measurement by means of the optical chronograph. HAID, GOETZE, SELLE, KOENEN, SCHMIDT, and BECKER (Jahresber. Chem.-tech. Reichsanst., 1930, 8, 121–124; Chem. Zentr., 1931, ii, 178).—The detonation velocity of Hg fulminate and Pb azide in detonators is about 4700 m. per sec.

A. A. ELDRIDGE.

Vaseline employed in smokeless powders. R. Poggi (Annali Chim. Appl., 1931, **21**, 500–506).—The products of reaction of nitrous vapours with vaseline containing unsaturated compounds are unstable. Vaseline of this nature appears to be unsuitable for the stabilisation of smokeless powders. The analytical data obtained from two vaselines of different nature are given, the tests applied being : Rosenmund I val., the Düneberg KMnO<sub>4</sub> test, the Dumas N determination, and the author's colorimetric test. O. F. LUBATTI.

Volatility and persistence of chemical-warfare substances. J. B. NIELSEN (Z. ges. Schiess- u. Sprengstoffw., 1931, 26, 420-423).--Leitner's formula for the persistence of a volatile substance in terms of its v.p. and mol. wt. and of those of H<sub>2</sub>O may be taken to represent the volatility, if the stability of the substance and the effect of the atm. and alkalis in soil are left out of account. An expression has now been derived correlating the heat of volatilisation, the abs. temp. in equilibrium, the mol. wt. and v.p. of the liquid, and the abs. temp. of the surrounding atm. and the relative volatilities of chloropicrin, phosgene, diphosgene, and "mustard gas" (solid and liquid) are tabulated over the range  $-10^{\circ}$  to  $30^{\circ}$ . The use of Leitner's formula in practice entails a comparison of the volatility of the substance with that of H<sub>2</sub>O, vaporised in dry air. As the persistence is influenced by the chemical properties, especially with slow-volatilising substances, such as mustard gas, an empirical stability correction factor should be devised. In comparing different warfare materials, it is preferable to refer their persistence to their vol. rather than to their wt., since they are usually confined in projectiles of similar capacities.

W. J. WRIGHT.

See also A., Jan., 11, Pb azide and other explosives. 52, Tetranitrosopyrocatechol and its derivatives.

# XXIII.—SANITATION; WATER PURIFICATION.

Sludge digestion. H. BACH (Sewage Works' J., 1931, 3, 561-569).—The  $CH_4$  obtained by the anaërobic fermentation of sewage solids may be produced in three ways: the bacterial combination of  $H_2$  and  $CO_2$ , the splitting action of bacteria on fatty acids and salts, and the combining-splitting action on amines and  $H_2$ , e.g.,  $NH_2Me + H_2 = CH_4 + NH_3$ . Agitation of the digesting sludge is recommended as it maintains even conditions throughout the tank and, by breaking up the coarser particles, renders them more amenable to the process. The digestion of 50% of the original org. matter is considered to be the practical limit of the method. C. JEPSON.

Sewage investigations at the University of Illinois. H. E. BABBITT (Sewage Works' J., 1931, 3, 580—587).—As a result of tests carried out in the Engineering Experiment Station the following conclusions have been drawn. The draining qualities of sludge are improved by freezing, the entrained water separating very quickly after thawing. Heavy rain falling on open drying beds may accelerate the final drying of sludge rather than retard it. The application of artificial heat to Imhoff tanks in order to facilitate sludge digestion is impractical. It is advantageous to conduct sludge digestion as a two-stage process as grease inhibits digestion and its retention in a primary compartment aids digestion in the succeeding one. C. JEPSON.

Operation of the Collingswood, N.J., activatedsludge plant. I. O. LACY (Sewage Works' J., 1931, 3, 636-646).—This plant, designed to treat  $1\frac{1}{3}$  million gals./day, is treating approx.  $\frac{1}{2}$  million gals. and is operated by Link-Belt mechanical agitators. Owing to the proximity of inhabited houses the sewage is chlorinated before entering the preliminary settlement tank and also as it is finally discharged from the plant. The surplus sludge is digested in separate tanks and dried on covered beds. The total power consumption is approx. 100 h.p. per million gals. C. JEPSON.

Operation of the pre-aëration plant at Decatur, III. W. D. HATFIELD (Sewage Works' J., 1931, 3, 621-635).-The results obtained from over 3 years' operation of the pre-aëration plant at Decatur (cf. B., 1928, 693), where the sewage is abnormal owing to starch wastes, indicate that a 30-40% reduction in the biochemical O demand is obtained either in the presence or absence of returned sludge. This reduction must be due to the easily oxidisable matter which is removed by chemical oxidation, biological decomp. which takes place very rapidly, or a mechanical sweeping out of solution of volatile matter. If the reaction is biological, sufficient inoculation must exist on the tank walls or be introduced along with the influent from the return sludge which is mixed with the raw sewage before entering the Imhoff tanks. C. JEPSON.

Digestion of activated and primary sludge at Salinas, Calif. T. R. HASELTINE (Sewage Works' J., 1931, 3, 599-614).-Descriptions of the plant used and of the first year's experience in the collection, digestion, and disposal of fresh and activated sludge solids are given in detail. A digestion tank of 34,100 cu. ft. capacity is provided for a population of 10,000 with an average daily flow of 620,000 gals. of sewage. Without temp. control an average of 0.5-0.8 cu. ft. of gas per head per day was obtained when activated sludge was not being digested and 0.8-1.3 cu. ft. when this was the case. The high gas production may be due to the high grease content of the sewage (340 p.p.m.). The final sludge is run on to open drying beds and is ready for removal in 10-20 days. The addition of surplus activated sludge to the incoming sewage is recommended as an effective means of reducing the total vol. of sludge to be passed to the digestion tank. C. JEPSON.

Multi-stage sludge digestion at Los Angeles County Sanitation District's plant. A. M. RAWN (Sewage Works' J., 1931, 3, 570-579).-As a result of successful small-scale trials, a novel sludge digestion plant was placed in operation at Los Angeles in July, 1931. It consists of two batteries of four tanks, each of which is divided into three bays. The mixed sludge and "seeding" is pumped into the two outer bays of the first tank and passes by gravity from the centre bay of this tank to the outer bays of the succeeding one and so on until all four tanks have been traversed. The bays in each tank are interconnected by a 12-in. opening at the foot of the partition wall and the drawoff is from near the bottom of the centre bay of one tank by an inclined pipe to the surface of the outer bays of the next. In this way advantage is taken of any vertical separation which occurs and the passage of the heavier non-volatile solids through the tanks is expedited. Tanks II and III are heated by hot-water coils and along with tank I are fitted with stirring apparatus. The last tank in the series acts as a sedimentation basin in which the supernatant water is separated before the sludge is passed to the sludgedrying beds. C. JEPSON.

Excreta disposal plant at Kyoto, Japan. S. NISHIHARA (Sewage Works' J., 1931, 3, 656-661).--Excreta from the unsewered portion of the district is delivered at the sewage-disposal plant and after dilution with 38 times its vol. of water is treated in biolysis tanks of the Imhoff type and in spiral-flow activatedsludge tanks. The sludge produced is pumped into a two-storey digestion tank, the upper portion of which is used mainly for the partial dewatering of surplus activated sludge; the lower compartment comprises the digestion tank proper. The tank contents are circulated once daily, and displaced supernatant water is returned to the aëration tanks for further treatment. C. JEPSON.

Gas collection from the digestion of activated sludge at Rockville Centre, N.Y. W. GAVETT (Sewage Works' J., 1931 3, 615-620).-The sewage-disposal plant consists of a diffused-air activated-sludge tank dealing with settled sewage, followed by percolating filters and sludge-digestion tanks with glass-covered drying beds. It was designed for a population of 20,000 people (of whom 13,000 are connected), and is dealing with an average flow of 1,250,000 gals. per day. The sludge-digestion tanks have a capacity of 63,600 cu. ft. and receive a daily addition of 330 cu. ft. of sludge from the preliminary settlement tanks (97% H<sub>2</sub>O) and 1600 cu. ft. (98.8% H<sub>2</sub>O) from the activated-sludge tank. The gas produced  $(1 \cdot 0 \text{ cu.ft. per head})$  is used for power and for heating the sludge-digestion tanks, with satisfactory results. C. JEPSON.

Elimination of dust in mine air. W. J. WALKER and A. O. G. LE ROUX (J. Chem. Met. Soc. S. Africa, 1931, 32, 64—75).—The results of a preliminary investigation dealing with the use of cellulose solutions in spray form for the purpose are given. C. B. MARSON.

Chemotropic responses of the housefly, the greenbottle fly, and the black blowfly. E. W. LAKE, D. C. PARMAN, F. C. BISHOPP, and R. C. ROARK (U.S. Dept.

Agric. Tech. Bull., 1931, No. 270, 10 pp.).—Among numerous materials examined, pine-tar oils have high repellent vals. for all species. A. G. POLLARD.

Colour removal in Pacific coast water supplies. P. F. BOVARD and K. SHIBLEY (J. Amer. Water Works' Assoc., 1931, 23, 2129—2137).—The high average colour in the supply of Marshfield, Oregon, is greatly reduced by clarification with 1.73 grains/gal. of  $Al_2(SO_4)_3$  and 0.06 grain/gal. of  $Ca(OH)_2$  at  $p_H$  5.2. As a corrosion-preventive the  $p_H$  is raised to 7.5 after filtration and prior to aëration. C. JEPSON.

Boiler-feed water.—See I. Corrosion in waterpipes.—See X. Concn. control.—See XI.

See also A., Jan., 38, Radioactivity of natural waters.

#### PATENTS.

Apparatus for separate removal of floating and non-floating substances from sewage or other waste waters. W. RADERMACHER and C. DELKESKAMP (B.P. 362,567, 27.9.30).—In the operation of doublestorey tanks, an unsightly layer of floating substances may be prevented by first passing the sewage through a submerged pipe with a longitudinal opening in its upper part. The floating substances pass through the slit and non-floating matter passes into the sedimentation tank proper. The deposition in the pipe of materials from outside is prevented by means of a sloping baffle.

C. JEPSON.

Absorbent for gas filters particularly for breathing purposes. G. K. E. H. STAMPE (B.P. 362,552, 22.9.30).—A filter especially for use with gases containing  $H_2S$  and  $NH_3$  consists of a mixture of  $Cu_2Cl_2$ with absorptive material, *e.g.*, activated C or SiO<sub>2</sub> gel, impregnated with Pb(NO<sub>3</sub>)<sub>2</sub> solution.

L. A. Coles.

Disinfection and destruction of insect pests. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 358,508, 24.6.30. Addn. to B.P. 349,004; B., 1931, 908).—Di(hydroxyaryl)-amines, -alkylamines, or oxides are diluted with water or other solid or liquid diluents for disinfecting and sterilising preps., with talcum etc. for toilet powders, or with petroleum jelly, lanoline, etc. for cosmetic ointments. Suitable compounds are 5:5'-dibromo(or -chloro)-2:2'-dihydroxydiphenyl oxide, 3:3':5:5'-tetrabromo-2:2'-dihydroxydiphenyl oxide, 4:4'-dihydroxydiphenylamine, 2:2'-dihydroxydiphenyl oxide, tetrachloro-4:4'-dihydroxydiphenyl oxide, dichloro-4:4'-dihydroxydiphenylamine, 3:5-dibromo-4:4'-dihydroxydiphenylamine. C. HOLLINS.

Purification of boiler water. R. O. HENSZEY (B.P. 362,626, 12.11.30).—Excessive concn. of solids in boiler water is prevented by withdrawing a portion thereof in a predetermined ratio to the vol. of make-up water and replacing it with a clean supply. Heat exchangers are arranged so that the water withdrawn under pressure delivers up its heat, first to the comparatively large vol. of make-up water and condensate returns, and then to the more conc. replacing water, before being finally discharged at approx. atm. b.p.

C. JEPSON. Double distillation.—See I.