# BRITISH CHEMICAL ABSTRACTS

# B.—APPLIED CHEMISTRY

JUNE 22 and 29, 1934.\*

# I.-GENERAL; PLANT; MACHINERY.

.W.O. Labuboro, geodeficial o consultation

Temperature rise in a heat-evolving medium. E. N. Fox (Phil. Trans., 1934, A, 232, 431-461).— Mathematical. The general equations of heat flow in a medium evolving heat at a const. rate varying with time are derived and solved for six important 1-dimensional cases. H. S. P.

**Refrigeration problems in chemical practice.** E. KOLBE (Chem.-Ztg., 1934, 58, 355-357).—An application of refrigeration which is capable of further development is in the separation of vapours from gases, e.g.,  $C_{10}H_8$  from coal gas. The most economical method is countercurrent washing in a packed tower with brine or the like at a low temp. Exhaust steam when available is a cheap source of refrigerating power. A combination of a refrigeration plant with the condenser of a distillation operation would be even more economical.

C. I.

Gradual separation and economy. J. P. TREUB (Rec. trav. chim., 1934, 53, 497-524).—A mathematical treatment of the separation of a binary mixture in which attention is given to the economy of the process. H. S. P.

**Removal of silicic acid from boiler feed-water.** C. SLONIM and Z. HERRMANN (Chem. Obzor, 1933, 8, 134—139; Chem. Zentr., 1933, ii, 3024).—Formation of Al(OH)<sub>3</sub> from Al solutions is hindered or prevented by Na<sub>2</sub>SiO<sub>3</sub>, a sol being formed the stability of which depends on the  $p_{\rm H}$ . The pptd. sol contains the total Al, its SiO<sub>2</sub> content depending on the  $p_{\rm H}$ . At high  $p_{\rm H}$  no SiO<sub>2</sub> is pptd. An acid and a basic modification of SiO<sub>2</sub> are postulated, only the former being absorbed by Al(OH)<sub>3</sub>. H. J. E.

Bulking values of some gums, resins, oils, and miscellaneous liquids. ANON. (Sci. Sect. Nat. Paint, Varnish & Lacquer Assoc., Inc., May, 1934, Circ. No. 462, 129-131).—Vals. for d, wt./solid gal., and bulk (gal.) per lb. are tabulated. G. H. C.

Reflectometer and colour comparator. R. S. HUNTER (Sci. Sect. Nat. Paint, Varnish & Lacquer Assoc., Inc., Apr., 1934, Circ. No. 461, 111-128).-Coeffs. of diffuse reflexion of two surfaces are compared by illuminating them normally by an electric lamp the position of which between them is adjusted till they appear equally bright when viewed at 45° by a mirror system which brings the images into contact. Filters convert the instrument into a simple spectrophotometer.

G. H. C. Coals for steam-raising. Applications of town's gas.—See II. Viscosimeters.—See V. Solid CO<sub>2</sub>.— See VII. Refrigeration. Solid  $CO_2$ .—See XIX. Homeopathic grinding.—See XX.

# PATENTS.

Gas-producer and other furnace. A. L. GALUSHA (U.S.P. 1,926,939, 12.9.33. Appl., 17.7.30).—A furnace having a rotary stepped grate is provided with stirring arms which may be removed and reversed when they show signs of incipient bending. B. M. V.

Heating of material. W. S. KING, J. P. BARLIAK, and C. E. HARRIS (U.S.P. 1,926,544, 12.9.33. Appl., 21.3.31).—A gas-fired furnace for metal sheets etc. is described; part of the gas is enriched and the whole burned with a luminous flame. B. M. V.

Drying apparatus. B. M. HESS (U.S.P. 1,926,772, 12.9.33. Appl., 17.7.29).—A tower dryer for grain etc. is arranged so that the temp. in the space where the material enters and gases leave controls the admixture of combustion gases and fresh air for normal drying and, in the event of excessive temp. rise due to, *e.g.*, spontaneous combustion, the supply of all gaseous medium is completely cut off. B. M. V.

Treatment of [large] quantities of water. P. BOUCHEROT and G. CLAUDE (U.S.P. 1,926,191, 12.9.33. Appl., 7.10.27. Fr., 15.10.26).—The degasification of  $H_2O$  for use in low-pressure power generators operating within the general atm. temp. range is effected by the following steps, in which the  $H_2O$  is (a) made to rise in a barometric tube and gases are removed, after a slight descent from the highest point, nuclei bubbles being produced by electrolysis, if desired; (b) spread in films at the low pressure; (c) removed against the pressure and ejected down another barometric column to entrain the gases from (a). B. M. V.

Purification of steam. F. GUZA (B.P. 408,413 7.1.33. Ital., 7.1.32).—An entrainment separator comprising concentric perforated tubes is described.

B. M. V.

Prevention of fire by non-toxic substances. (A, B) T. MIDGLEY, JUN., and (B) A. L. HENNE and R. R. MCNARY, Assrs. to (A, B) FRIGIDAIRE CORP. (U.S.P. 1,926,395—6, 12.9.33. Appl., 31.7.30).—(A) A study, has been made of the combustion inhibition and toxic properties of the series  $CH_3Cl-CCl_4$ ,  $CH_3F-CCl_3F$ ,  $CH_2F_2-CCl_2F_2$ , and  $CHF_3-CClF_3$  (curves given), and claim is made, in (A), for addition to the atm. of any space that may be liable to fire of any of the above F compounds in  $\lt$  the toxic quantity, and, in (B), for extinguishing fires with an unspecified quantity of F compound in which total halogen is  $\lt$  total H.

B. M. V.

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

Manufacture of thermal- and sound-insulating material. L. T. HALLETT, Assr. to PROCESS HOLDINGS Co. (U.S.P. 1,925,142, 5.9.33. Appl., 15.10.30).—A filling for placing between reinforcing coatings to form boards or slabs comprises diatomaceous earth and org. fibrous material bound by gelatinised starch or casein, the wet mixture being whipped in air with a protein frother and then dehydrated, further expansion taking place in the earlier stages of heating. B. M. V.

Calorimeters. T. C. SUTTON, and GRIFFIN & TAT-LOCK, LTD. (B.P. 408,396, 16.12.32).—A bomb with two end-caps, the internal pressure increasing the expansion of the gaskets, is described; an ignition device is claimed also. B. M. V.

Cooling system. E. CLINE (U.S.P. 1,926,970, 12.9.33. Appl., 13.2.32).—Apparatus embodying various forms of conical air- and  $H_2O$ -distributing members and suitable for cooling either fluid is described. B. M. V.

Apparatus for washing coal and other granular materials. EBBW VALE STEEL, IRON, & COAL CO., LTD., and A. H. HARVEY (B.P. 408,367, 9.11.32).—Coal is washed on stationary inclined tables, the heavy material flowing down the slope and the light being dragged up by transverse bars attached to chain conveyors. A complete scheme comprising a no. of tables and screens is described. B. M. V.

Roller grinding mills. A. E. BARRETT, and DAVEY, PAXMAN & Co. (COLCHESTER), LTD. (B.P. 408,026, 14.10.32).—In a single-roll mill a double grinding bar co-operates therewith and provision is made for the return of partly ground material from the space between the two bars back to the feed hopper. B. M. V.

Single-roll mill for paint and the like. M. LEH-MANN and E. GRÜNDLER, ASSIS. to J. M. LEHMANN (U.S.P. 1,926,907, 12.9.33. Appl., 8.7.30. Ger., 13.7.29).—The roll is journalled in the main frame while the feed hopper and grinding block are supported on separate frames which are adjustable by rocking. B. M. V.

Filter press. E. D. SEYMOUR, ASST. to VACUUM OIL Co., INC. (U.S.P. 1,926,115, 12.9.33. Appl., 7.10.32).— Every plate is grounded by a flexible cable to bus-bars which may be the supporting bars of the press.

B. M. V.

**Operation of filters.** W. L. SOUTH and C. T. ANNÉ, Assrs. to TEXAS Co. (U.S.P. 1,925,824, 5.9.33. Appl., 3.12.29).—A filter cake containing adsorbent, impurities removed from oil, and oil itself is washed in turn with a volatile solvent, a non-volatile oil, and an inert gas before opening the filter and blowing off the cake. B. M. V.

[Centrifugal] extractor. E. S. PEARCE, ASST. to RAILWAY SERVICE & SUPPLY CORP. (U.S.P. 1,926,402, 12.9.33. Appl., 27.10.30).—The wall of the basket is fluted to prevent undue packing of solids, and the extracting liquid is supplied through nozzles pointing in the direction of rotation and inclinable to cover the surface from top to bottom. B. M. V.

Discharging device for centrifugal machines. G. TER MEER, Assr. to A. T. OTTO & SONS (U.S.P. 1,925,121, 5.9.33. Appl., 12.1.32. Ger., 27.1.31).—A knife for removal of sediment, operated by hydraulic or pneumatic means, is described. B. M. V. Vacuum distillation of high-b.-p. products. G. W. RILEY, and G. SCOTT & SON, LONDON, LTD. (B.P. 408,259, 5.9.32).—The vapour space of a still is jacketed by the downflowing, outgoing vapours so as to prevent condensate from forming reflux into the still. A system of two-stage distillation in absence of steam or other v.-p. depressant is described; the condenser of the coldest still is cooled by  $H_2O$  maintained under vac. The apparatus is suitable for the treatment of oils, fatty acids, glycerin, etc. B. M. V.

Distilling apparatus. H. W. SHELDON, Assr. to VACUUM OIL CO., INC. (U.S.P. 1,926,116, 12.9.33. Appl., 28.1.31).—For used lubricating oil or other mixture of constituents having widely differing b.p., the apparatus comprises a tower having at an intermediate point a no. of steam pipes in echelon, bubbling trays above and below, and steam injection at the bottom. B. M. V.

Emulsifying machines. E. M. MULLER (B.P. 408,468, 14.6.33).—A multi-stage centrifugal pump is provided with closed impeller wheels constructed of wire gauze and preferably in wavy or zigzag formation.

B. M. V. Apparatus for indicating the level and specific gravity of liquids and the pressure of liquids or gases. A. R. ALLEN (B.P. 408,236, 29.9.32).—A pair of solenoids in line is connected by 3 wires to a (remote) pair of coils acting differentially on an armature (A) and an a.c. is applied to the outer wires. The movement of A will then follow the movement of an Fe core in the solenoids, that movement being effected by a float or pressure device. B. M. V.

[Effecting] physico-chemical operations between gases and liquids, such as absorption, evaporation, and combination. E. A. GALLARD (B.P. 408,016, 5.10.32. Fr., 6.10.31).—An apparatus comprising a rotor formed of a no. of hollow discs which irrigates its own surface and also that of an intercalating stationary housing, each part having passages for the internal flow of heating or cooling medium, is used, *e.g.*, for the separation of pure HNO<sub>3</sub> from dil. mixed acid; two other similar units are used for the concn. of the separated acids. B. M. V.

Dispensing gases in liquefied or gaseous state. LINDE AIR PRODUCTS CO., Assees. of L. I. DANA (B.P. 408,089, 3.3.33. U.S., 19.4.32).—Within the same insulating jacket as that surrounding a main storage vessel (A) there is an auxiliary vessel (B); draw-offs for both gas and liquid are provided from both A and B, a transfer conduit leads from A to B, and a filling aperture is fitted to A, thus enabling a const. supply of gas or liquid to be maintained from B while A is being filled. B. M. V.

Dehydration of gas. WHESSOE FOUNDRY & ENG. CO., LTD., and A. G. GRANT (B.P. 408,009, 11.8.33).—Heat is transferred from the gas entering a dehydrator to the same gas after dehydration by means of a pair of heat exchangers coupled by the continuous circulation of a liquid between them. B: M. V.

Jointing, packing, sealing, and like materials. V. G. WALSH (B.P. 407,979, 28.9.32).—A substitute for cork in bottle caps etc. comprises hydrated SiO<sub>2</sub> (100 pts.

p, 8 pts.), and saturated stean be pptd. in the lump fuel (resist

by wt.), fibrous material (e.g., wood pulp, 8 pts.), and coagulated latex (30 pts.). The  $SiO_2$  may be pptd. in the pores of the fibre and a wax-like or oily substance may be present. Manufacture is preferably effected in aq. solution or emulsion followed by drying and moulding by heat and pressure. B. M. V.

Friction material. R. J. NORTON, ASST. to BENDIX BRAKE Co. (U.S.P. 1,926,699, 12.9.33. Appl., 21.2.30).— SiO<sub>2</sub> gel is incorporated in an asbestos brake lining either in the form of studs or is dispersed throughout, in order to adsorb  $H_2O$  in wet weather, the adsorbent being regenerated by the heat of usage. B. M. V.

Pulverising coal.—See II. Distillation.—See III. Base-exchange silicate.—See VII. Preparing wort. —See XVIII.

# II.—FUEL; GAS; TAR; MINERAL OILS.

Nature, preparation, storage, and coking of typical coals from the Sydney area, Nova Scotia. R. E. GILMORE and R. A. STRONG (Trans. Canad. Inst. Min. Met., 1933, 36, 317-370).-These are caking, bituminous coals; analysis of a typical sample gave (on the mineral matter-free basis): H<sub>2</sub>O 2.7, volatile matter 31.7, fixed C 61.6%; calorific val. 14,910 B.Th.U./lb.; the ash averaged about 7% and the S about 3%. Some data relating to the banded constituents of the coals, their classification, the method of correction of the analyses to the mineral matter-free basis, the ash fusion temp. of mixed coals from this area, and high- and low-temp. laboratory assays of typical samples (cf. B., 1932, 583) are discussed briefly. Blends containing 30-35% of a Sydney coal, which had been washed to an ash content of approx. 3% and S 1%, 57-50% of a medium-volatile Pennsylvania coal, and the remainder Pocahontas, gave satisfactory cokes in large-scale coke-oven tests. The washed coal also behaved satisfactorily on storage. A. B. M.

Mature and immature fuels. J. ROBERTS (J. Inst. Fuel, 1934, 7, 228—230).—Some characteristics of peats, lignites, bituminous coals, and anthracites are briefly discussed with reference to particular specimens which were exhibited. The lustre, iridescence, and structure of the specimens of anthracite indicate that they have been subjected to coking temp., e.g., about 500°, during their formation. A. B. M.

Technical investigation of Northern Ontario lignite. C. TASKER (Trans. Canad. Inst. Min. Met., 1933, 36, 169-203).-The extent and position of the lignite (I) deposit at Onakawana is briefly described; it consists of two seams, analysis of average samples of which gave, for the upper (U) and lower (L) seams, respectively :  $H_2O 45.0$ , 50.0%; volatile matter 21.5, 20.8%; fixed C 25.2, 22.7%; ash 8.3, 6.5%; S 0.68, 0.79%, calorific val. 5490, 5175 B.Th.U./lb. The deposits contained three physically different types of (I), viz., woody, peaty, and earthy; the presence in Uof a large proportion (72%) of earthy (I), which disintegrates readily on drying and produces a large amount of dust, renders this seam less important economically than L [earthy (I) 24.5%]. Details are given of trials made with some 105 tons of the material at various test plants in Europe. Drying the lump (I) with

saturated steam by the Fleissner process produced a lump fuel (resistant to weathering) containing 20% H<sub>2</sub>O, having calorific val. 9000 B.Th.U./lb., and possessing good combustion properties. The (I) can be dried to 15% H<sub>2</sub>O by flue gases; the product disintegrates but should be suitable as a pulverised fuel. On low-temp, carbonisation, by the Lurgi or Kohlenveredlung process, the (I) yielded about 60% of char and  $3 \cdot 5-5\%$  of tar and oils [calc. on dry (I)]; the gas produced was used up in the process. The char must be briquetted with  $\leq 10\%$  of pitch to give a saleable product. Briquetting under high pressure without a binder was only partly successful. A. B. M.

Selection of coals for steam-raising. C. A. SEYLER (J. Inst. Fuel, 1934, 7, 181—194).—The properties of steam coals are considered in relation to Seyler's classification chart (cf. B., 1931, 1031) and an attempt is made, based on the researches of Rosin *et al.* (cf. Z. Ver. deuts. Ing., 1931, 75, 849), to develop a theory relating these properties to the rate of heat liberation, flexibility, and efficiency of combustion. A. B. M.

**Composition of coal.** W. A. BONE (Chem. & Ind., 1934, 431).—Phenols produced by treating Morwell coal with  $C_6H_6$  (I) at 285° are not derived from (I). S. C.

Constitution of the humic acids of brown coal, and their chemical and physical changes on coalification. H. STACH (Braunkohlenarch., 1933, 40, 1-51; Chem. Zentr., 1933, ii, 1284).-In humic acids from various types of brown coal the C varies between 60 and 70%, and the OMe of the Me esters between 14 and 16%. The CO,H group was converted into chloride and amide, but Hofmann's reaction gave no amine, and the CO<sub>2</sub>H group could not be replaced by NH2 by means of N3H. The phenolic OH was replaceable by NH2. The acids decompose at 165-200°. HNO3 oxidation of methylated humic acids affords COMe2-sol. products of mol. wt. 843. a-Humic acids pass into difficultly peptisable β-acids, and finally into  $\gamma$ -acids, insol. in alkali. The coalification process is attributed to pressure, and the formation of CH4 to reduction processes. A. A. E.

M.p. of coal and coke ashes. H. A. J. PIETERS and G. SMEETS (Het Gas, 1933, 53, 446—448; Chem. Zentr., 1933, ii, 3076).—The Seger cone val. in a reducing atm. (I) is < that in an oxidising atm., the effect increasing with the % Fe<sub>2</sub>O<sub>3</sub>. The m.p. in (I) is raised by treating the coal with acids. H. J. E.

Specific-gravity investigation of coal samples. P. E. HALL (J. Chem. Met. Soc. S. Afr., 1934, 34, 263-269).—Finer samples can be used and more rapid operation is possible when a centrifuge is used in making the test described previously (B., 1932, 487). The sample and CCl<sub>4</sub> are placed in a test tube containing a wire-gauze basket to collect the float portion, and, after centrifuging for 20 min., the float and sink are collected, weighed, and analysed. A. R. P.

Carbonising test of Natal coal. T. R. THERON (J. South African Chem. Inst., 1934, 17, 13—14).—Details are tabulated of a 4-day test in Glover-West vertical retorts. A. G.

Carbonisation of Somerset coals. S. HOLE (Gas J., 1934, 206, 265–268).—Results obtained in horizontal retorts, with special reference to methods of control such as temp. of the condensers and calorific val. of the various gas streams, are discussed. The production of a good domestic coke is ensured by selection of coals of low const. ash content. R. N. B.

Gasification values of gas- and coking-coals. K. BUNTE (Gas- u. Wasserfach, 1934, 77, 273-277).-The cost of "carbonising" the ash content of coal is discussed. The effect of the H<sub>2</sub>O content on the heat needed for carbonisation, on the packing d of coal, and on the ease of condensation of the gas is shown graphically. The adsorption of  $O_2$  by coal increases with time of storage and has a large effect on its coking properties. Methods for costing coal based on the above data are given. Tabulated figures show the effect of inert gases on the calorific val. of the gas, and its connexion with the necessary water-gas production. The average ratios of the val. and production of coke to the coal carbonised for a no. of works are given, and the effect of the former on the relative cost of coal gas and water-gas is discussed. R. N. B.

Hydrogenation of coal. W. I. JONES (Chem. & Ind., 1934, 321-326).—The effects of varying the temp., pressure, reaction time, rate of  $H_2$  circulation, and type of coal are briefly discussed. Typical yields in the first (liquid-phase) stage of the process from 100 pts. of a British bituminous coal are : petrol (I) 31·3, middle oil (II) 37·3, heavy oil 5·0, gas 23·6, org. insol. 3·0, liquor 9·6,  $H_2$  absorbed 9·8. The (II) is then converted into (I) in the second (vapour-phase) stage of the process. All British coals, with the exception of anthracites, can give yields of light oils on hydrogenation. Under the optimum conditions up to about 200 gals. of refined (I) per ton of dry ash-free coal can be produced. A. B. M.

Industrial applications of town's gas. C. M. WALTER (J. Inst. Fuel, 1934, 7, 217-227).-The combustion efficiency of fuels is briefly discussed. When the "all-in" cost of an operation is considered it is often economically advantageous to use town's gas (I) rather than solid fuel, although the cost per B.Th.U. is 3-5 times as great. (I) is now used extensively in carburising and general heat-treatment work, forging, tube annealing, japanning and enamelling, central heating, etc. The introduction of light-wt., alloy-steel gas-storage cylinders has made practicable the use of (I), compressed to 5000 lb./sq. in., as a motor fuel. By increasing the compression ratio of the engine the power output obtainable with (I) is the same as, or even >, was previously obtained with petrol, without danger of detonation. A double-stage, diaphragmtype valve is used for reducing the pressure from that in the cylinders to slightly below atm., and a special air-gas mixer takes the place of the carburettor. An estimate of the cost of running a fleet of 12 'buses shows that a saving of about 6s. 7d. per vehicle per day could be made by using (I) at 4d. per therm in place of petrol at 1s. 07d. per gal. A. B. M.

Determination of naphthalene in gas. H. SEE-BAUM and W. OPPELT (Gas- u. Wasserfach, 1934, 77, 280—282).—The determination of  $C_{10}H_8$  (I) by picric acid (P) depends on the temp. and concn. of the latter solution. Figures are given for the solubility of P in H<sub>2</sub>O, and it is shown that as [P] decreases or the temp. of determination rises the amount of (I) recovered is < the calc. val. A new method of analysis is therefore proposed in which two wash-bottles each containing 100 c.c. of a 0.9% solution of P cooled to 4—7° are used. R. N. B.

Dry purification [of gas]. E. KREBS (Gas- u. Wasserfach, 1934, 77, 193-199).-Regeneration of fouled oxide is best carried out in situ, but the amount of O, taken up is influenced by the temp. of the purification material, which itself depends on variation of atm. temp. and conditions. 0.3-0.6 vol.-% of O2 passes through unchanged, and this amount is independent of the temp. so long as the O2 content of the inlet gas is > 1.5 vol.-%. Methods for the determination of properties such as particle size, d, packing d, and activity are described and the results obtained with natural and synthetic oxides compared. A rapid and accurate method for determining the amount of S adsorbed (apparatus described) shows that Lautamasse (I) is the most active of the oxides employed. The activity of the mass is a function of the surface/unit vol. and the velocity of reaction decreases with time in use owing to alteration of the surface of the material. With hydrated  $Fe_2O_3$  the velocity of sulphide formation is >that of conversion of sulphide into oxide; the opposite is the case with (I), whilst bog ore occupies an intermediate position. The two reaction velocities are approx. 10-2 and 10-3, and their temp. coeffs. are  $1\cdot 2$ — $1\cdot 4$  and  $1\cdot 4$ — $1\cdot 6$ , respectively. (I) continuously regenerated in boxes protected from atm. influences is considered to be the best material for dry purification of gas. R. N. B.

Physical and chemical properties of hydrogenated naphthas. J. R. STEWART (Sci. Sect. Nat. Paint, Varnish & Lacquer Assoc., Inc., Apr., 1934, Circ. No. 460, 104—110).—Evaporation rates and distillation ranges are plotted for three hydrogenated naphthas in comparison with PhMe, turpentine, and white spirit.

G. Ĥ. C.

Recovery of benzine from natural gas and [petroleum] refinery gas. N. MAYER (Chem.-Ztg., 1934, 58, 357—359).—The gas after scrubbing with  $H_2O$ and NaOH is compressed, cooled, and passed into a column fed with paraffin. The saturated paraffin passes to a fractionating column working under pressure. The distillate is freed from gases in a reflux condenser at 1 atm. and then refined and redistilled. Adaptations allow pure  $C_3H_8$  and  $C_4H_{10}$  to be made if desired. Natural gas contains much lower proportions of condensable vapours and is treated under higher pressures (25 kg. per sq. cm.). The pressure is reduced in two stages, and the distillation of the enriched absorbing oil takes place at 3—4 atm. pressure. C. I.

Miscibility of petrol and some of its components with aliphatic alcohols of various strengths. W. R. ORMANDY, T. W. M. POND, and W. R. DAVIES (J. Inst. Petroleum Tech., 1934, 20, 308–338).—The limits of miscibility in the binary combinations of MeOH,

EtOH, PraOH, PrBOH, BuaOH, BuBOH, sec.-BuOH, Bu'OH, active amyl alcohol, and a-ethylhexanol with  $C_6H_6$ , PhMe, cyclohexane, CMe<sub>2</sub>.CHMe, petrol, and n- $C_7H_{16}$  in presence of small quantities of  $H_2O$  have been determined, chiefly at 15°. The wt. of hydrocarbon, W, miscible with a fixed wt. of aq. alcohol diminishes with fall of temp. from 25° to 5°, and at const. temp. W decreases approx. in the order of the hydrocarbons named. The H<sub>2</sub>O tolerance, T, i.e., the no. of c.c. of H<sub>2</sub>O which dissolves in 1 gal. of anhyd. mixture without causing a second phase to separate, is given for each of the above mixtures and is greatest with EtOH (and aromatics only) and  $Pr^{\beta}OH$ . The increase of T by adding COMe, is < that produced by an equal quantity of the above alcohols, whilst binary mixtures of EtOH-Bu<sup> $\beta$ </sup>OH increase T to upwards of twice the calc. additive val. when mixed with aliphatic, but not aromatic, hydrocarbons. Extensive data are recorded for multi-component systems. J. G. A. G.

Miscibility of methyl alcohol with petrol and benzene. F. M. E. SHEPHERD (J. Inst. Petroleum Tech., 1934, 20, 294–307).—The data refer to the "ternary" systems petrol– $C_6H_6$ –MeOH containing 0—20 vol.-% H<sub>2</sub>O (I), petrol– $C_6H_6$ –MeOH containing COMe<sub>2</sub>, and petrol–COMe<sub>2</sub>–96 vol.-% MeOH, at 20°, 0°, and -20°. Abs. MeOH is not completely miscible with petrol, and the limits of complete miscibility in system (I) decrease with increasing [H<sub>2</sub>O] and with fall of temp. and are < the corresponding limits obtained with EtOH. The mutual solubilities are increased by adding  $C_6H_6$ , but the latter, when at high concn., crystallises at low temp. Commercial MeOH containing COMe<sub>2</sub> and H<sub>2</sub>O affords a wider range of complete miscibility with petrol and  $C_6H_6$  than does pure MeOH of equal d. J. G. A. G.

Behaviour of lubricating oils in the motor. R. OREL (Petroleum, 1934, 30, No. 15; Motorenbetr., 7, 2—7).—The decrease in  $\eta$  of the lubricating oil on use in the motor is due only to dilution with the higherboiling fractions of the petrol. The initial rapid rate of decrease is followed by a slower change until finally an approx. const.  $\eta$  is reached. Dilution, however, may still be proceeding, being counterbalanced, as far as its effect on  $\eta$  is concerned, by the increasing rate of sludge formation. The rate of dilution is less dependent on the initial  $\eta$  of the oil than on the working conditions of the motor, decreasing, *e.g.*, with increasing load. Oil consumption diminishes, and formation of coke in the oil increases, with increasing initial  $\eta$ . A. B. M.

Lubrication of bearings with lubricating greases. H. VON SCHROETER (Petroleum, 1934, 30, No. 15, 1-20; cf. Proc. World Petroleum Congr., 1933, 2, 434).—The consistency (I) of a lubricating grease (G), determined by measuring the rate of penetration thereinto of an Al rod having a conical head (wt. 65 g.; angle of cone 30°), increased with previous mechanical working, and increased also with rising temp., slowly at first and rapidly above about 60°. The method of measurement had a wider range of application than that due to Kissling. The determination of the "drop point" or "flow point" (Ubbelohde) is not sufficient to characterise the G from the viewpoint of (I). The flow of a G through a capillary tube obeys Reiner's equation

(cf. A., 1926, 678); from this vals. for the "flow-stress" and "mobility" of the G can be deduced, but the data as yet available are insufficient to correlate fully these vals. with the (I). The variation of the friction (F) with speed of rotation (R), rate of feed of G, load, and temp. has been studied on a lubricant-testing machine. Other conditions being const., F passes through a min. with increasing R; the min. val. falls with increasing rate of feed of G. There is a marked limiting val. of the load above which lubrication fails : this val. varies with the kind of G, with its (I), and, to a smaller extent, with R. The limiting load is also greatly influenced by the design of the bearing, particularly in the mode of feed of the lubricant. The results are discussed with reference to the technical application of G. A. B. M.

Refrigeration [of coal gas].—See I. Solvents.— See III.

See also A., May, 507, Pyrolysis of the lower paraffins.

## PATENTS.

**Pulverising of coal.** W. F. CAREY, E. F. BRAWN, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 407,316, 13.8.32).—Coal is pulverised to a size  $< \frac{1}{25}$  in. in a multistage roll crusher in which the individual stage-reduction ratios, with the possible exception of the ratio for the last set of rolls, lie between 2.0 and 3.6. Screens having gaps 2—3 times as wide as the gaps between the preceding rolls are installed between successive stages in order to remove flakes. A. B. M.

**Powdered fuel for internal-combustion engines.** R. PAWLIKOWSKI (U.S.P. 1,926,304, 12.9.33. Appl., 10.2.28. Ger., 14.6.27).—The fuel is ground small enough to pass through the usual injection valve and comprises a mixture of slow-burning carbonaceous material (anthracite), which alone would not burn completely in one stroke, with a smaller proportion of priming (lignite) powder rendered more combustible by fine grinding or super-drying, having low ash content etc. B. M. V.

Production of stable dispersions of coal or like carbonaceous material in oil for use as fuel or for other purposes. J. L. STREVENS and W. B. MITFORD (B.P. 407,360, 9.7. and 6.8.32) .- Coal etc. pulverised until 80-90% of it passes a 200-mesh sieve (I.M.M.) is mixed with an oil rich in aromatic, asphaltic, or naphthenic constituents, and with  $\gg 5\%$  of a non-aq. wetting or peptising agent (P), and the mixture is digested in a closed vessel under pressure at 300-370°. P may consist of an alkali-sol. OH-derivative of a hydrocarbon, a higher phenol, an oil-sol. org. acid (e.g., oleic), rubber latex, etc. The mixture may be destructively distilled (cf. B.P. 393,602; B., 1933, 738). When it is desired to use the mixture as a liquid fuel its  $\eta$ may be reduced by the addition of a light Diesel oil etc., and any solid material may be removed by filtration or centrifuging. A. B. M.

Combustion of coal. C. M. BUCK (U.S.P. 1,925,132, 5.9.33. Appl., 16.8.32).—Soft coal is heated to a temp. (120—250°) at which endothermic partial combustion takes place and renders the coal non-explosive; it is

then pulverised and injected into a combustion furnace with all or nearly all the air for complete combustion.

B. M. V.

Operating carbonising chambers or retorts. WOODALL-DUCKHAM (1920), LTD., T. C. FINLAYSON, and W. T. NEWTON (B.P. 407,381, 12.8.32).—The heating flues (H) of the retorts (R) are connected to the wastegas flue (W) by ducts controlled by dampers (D) and to a common bus-flue, which in turn is connected with W by ducts controlled by D. The arrangement permits any idle R to be maintained under heat by diverting hot waste gases from H of R in operation through those of the idle R on their way to W. A. B. M.

**Operation of coke ovens and the like.** S. P. MILLER, Assr. to BARRETT Co. (U.S.P. 1,918,709, 18.7.33. Appl., 19.12.28).—A coke-oven battery is equipped with two gas-collecting mains into which the gases evolved during the earlier and later parts, respectively, of the coking operation are passed. On cooling, these yield tars of low and high free C content, respectively. These tars, which are separately collected, are then distilled by being brought into intimate contact with another portion of the hot gases from the corresponding stage of the coking operation, whereby they yield pitches of low and high free C content, respectively. A. B. M.

(A) Production of carbon black. (B) Carbonblack producer. R. H. BROWNLEE (U.S.P. 1,925,130 -1, 5.9.33. Appl., 26.12.31).—(A) The C black is formed by dissociation during a short period of contact with hot chequerwork and the gas quickly cooled by admixture with refrigerated returned gas. (B) Dissociation is effected by partial combustion in small-bore refractory tubes. B. M. V.

Removal of carbon deposits. W. G. LOVELL and T. A. BOYD, ASSTS. to GEN. MOTORS RES. CORP. (U.S.P. 1,925,732, 5.9.33. Appl., 28.8.29).—Varnish removers, *e.g.*, a mixture of Et allyl ether, benzol, and EtOH, are applied. B. M. V.

Destructive hydrogenation of coal and allied carbonaceous materials. L. HORTON, J. G. KING, and F. A. WILLIAMS (B.P. 407,400, 21.9.32).—The pulverised material is maintained in a state of agitation in suspension in the hydrogenating gas, under suitable conditions of temp. and pressure, and the volatile products of reaction are withdrawn at various stages corresponding with successive intervals of time of treatment, the products being condensed and collected (or subjected to further treatment) separately. The process may be carried out in presence of a known hydrogenating catalyst. A. B. M.

Treatment of fuel gas [to remove naphthalene]. F. W. SPERR, JUN., ASST. to KOPPERS CO. OF DELAWARE (U.S.P. 1,924,813, 29.8.33. Appl., 21.4.31).—The cold gas is scrubbed with oil to remove  $C_6H_6$ , compressed to 4 atm., cooled, and passed through an absorbent oil to remove part of the  $C_{10}H_8$  (I) and saturate the gas with oil; it is then compressed to 18 atm. and again cooled, whereby the oil and remaining (I) are deposited.

A. R. P.

Disposal of gas-works' effluents and the like. HAYWARDS HEATH DISTRICT GAS Co., and A. E. WHIT-CHER (B.P. 407,374, 16.9.32).—Effluents or liquors which on evaporation leave a combustible residue are sprayed on to a heated absorbent material, *e.g.*, coke, whereby part of the liquor is evaporated and the remainder is absorbed. The liquor is preferably preheated. The coke may subsequently be used as a boiler fuel. A. B. M.

Distillation of tar at a coal-distillation plant and apparatus therefor. F. OSBORNE, ASST. to BARRETT Co. (U.S.P. 1,919,704, 25.7.33. Appl., 26.2.27).-The uptake pipes of some of the ovens of a coke-oven battery are so designed that tar can be sprayed into the ascending, hot, coal-distillation gases and so distilled, the vapours passing on with the gases to a separate collector main  $(M_1)$  and condensing system while the pitch is continuously withdrawn from an annular space surrounding the gas outlet pipe. The other ovens of the battery are connected to an ordinary collector main  $(M_2)$ . Valves and connexions are provided which permit the specially designed uptake pipes to be disconnected from  $M_1$  and connected to  $M_2$  when they are not required for tar distillation. A. B. M.

Treatment of tar used as sealing medium for the pistons of waterless-type gasholders. C. F. TOOBY and P. N. LANGFORD (B.P. 407,534, 28.3.33).— The tar which leaks from the seal and runs down the wall of the gasholder (H) is heated in contact with a stream of gas before being returned to the seal; any vapours absorbed from the gas in H are thereby removed and the  $\eta$  of the tar is maintained at the desired val. Suitable plant is described. A. B. M.

Manufacture of asphalt. BURMAH OIL Co., LTD. From H. L. ALLAN (B.P. 407,522, 25.2.33).—Suitable stock is preheated and blown with air, the process being made continuous by providing two vessels (A, B) fitted with pipe-coils and so connected that the charge being blown in A can be circulated through the coils in B, which contains a fresh charge of the material. The excess heat generated during the operation of blowing the charge in A is thus utilised for preheating the charge in B. The process is then repeated with the functions of A and B reversed. The process is particularly suitable for treating extracts obtained by the treatment of mineral oils with liquid SO<sub>2</sub>, PhNO<sub>2</sub>, PhOH, or other selective solvent. A. B. M.

(A) Asphalt emulsion. (B) Production of [asphaltwater] emulsions. P. R. SMITH, Assr. to BARBER ASPHALT Co. (U.S.P. 1,926,246-7, 12.9.33. Appl., [A] 3.7.28, [B] 11.4.30).--(A) Asphalt (I) is dispersed in an aq. solution of trialkali phosphate, a protective colloid and/or a borate being present if desired. (B) In the prep. of an emulsion as in (A), the (I) is added in molten condition. B. M. V.

Oil purification. C. M. AMBLER, JUN., ASST. to SHARPLES SPECIALTY Co. (U.S.P. 1,926,188, 12.9.33. Appl., 22.10.29).—Used railway journal oil is heated to a temp.  $(121-176^{\circ})$  at which H<sub>2</sub>O will be evolved and the foam broken, and then centrifuged while hot. B. M. V.

Liquid fuel. J. A. VANCE (U.S.P. 1,926,071, 12.9.33. Appl., 24.12.31).—An intimate 2:3 mixture of  $H_2O$  and crude petroleum is subjected to a pressure of >7000 lb. per sq. in. at 37° to produce a stable  $H_2O$ -in-oil emulsion. A. R. P. Lubricating oil testing device. R. JOHN, Assr. to PENNZOIL CO. (U.S.P. 1,925,254, 5.9.33. Appl., 11.5.31). —Used lubricating oil is absorbed in a bibulous spot on a rotatable card and compared with differently coloured standard spots. B. M. V.

Expansion valve for carrying out cracking and distillation processes. C. STILL (B.P. 409,177, 2.8.33. Ger., 5.8.32).

Heating apparatus [for mineral oils] and control of temperatures therein. W. G. LEAMON (U.S.P. 1,933,020, 31.10.33. Appl., 17.9.28).

Cracking of hydrocarbon oils. (A) J. DELATTRE-SEGUY, (B) G. EGLOFF, ASSTS. to UNIVERSAL OIL PRO-DUCTS CO. (U.S.P. 1,929,795 and 1,931,757, [A] 10.10.33, [B] 24.10.33. Appl., [A] 23.3.27, [B] 20.4.31).

Cracking process [for hydrocarbon oils]. S. P. Coleman, Assr. to Standard Oil Development Co. (U.S.P. 1,933,048, 31.10.33. Appl., 23.4.29).

Cracking [of hydrocarbon oils] in homogeneous state. E. B. PECK, ASST. to STANDARD OIL DEVELOP-MENT Co. (U.S.P. 1,933,507, 31.10.33. Appl., 21.8.29).

Conversion of hydrocarbon oils. C. W. WATSON, Assr. to TEXAS Co. (U.S.P. 1,929,529, 10.10.33. Appl., 19.7.28).

Treatment of hydrocarbon oils. W. M. STRAT-FORD, ASST. to TEXAS CO. (U.S.P. 1,930,730, 17.10.33. Appl., 25.7.29). F. W. HALL and J. H. GRAHAME, ASSTS. to TEXAS CO. (U.S.P. 1,930,974, 17.10.33. Appl., 7.1.30). E. F. NELSON, ASST. to UNIVERSAL OIL PRO-DUCTS CO. (U.S.P. 1,930,211, 10.10.33. Appl., 22.10.30).

Apparatus for treating liquids [hydrocarbon oils]. E. C. D'YARMETT, ASST. to FRACTIONATOR Co. (U.S.P. 1,932,514, 31.10.33. Appl., 28.8.29).

Conversion of hydrocarbons. J. G. Alther, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,930,221, 10.10.33. Appl., 26.5.28).

Rectification of hydrocarbons. C. D. GARD, Assr. to UNION OIL CO. OF CALIFORNIA (U.S.P. 1,930,166, 10.10.33. Appl., 14.7.30).

Apparatus for treating hydrocarbon vapours. W. F. Sims and V. U. Cloer, Assrs. to Panhandle Re-FINING Co. (U.S.P. 1,930,372, 10.10.33. Appl., 23.11.29).

Cracking of petroleum oil. G. Egloff, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,930,231, 10.10.33. Appl., 17.10.23. Renewed 17.3.32).

Continuously treating [cracked] petroleum distillates. J. B. WEAVER (U.S.P. 1,929,861, 10.10.33. Appl., 9.10.30).

Apparatus for refining liquids [petroleum oils]. R. C. OSTERSTROM and R. T. TUCKER, ASSTS. to PURE OIL CO. (U.S.P. 1,930,597, 17.10.33. Appl., 12.9.27).

Mixer for petroleum oil refining. C. L. ERICKSON, Assr. to STANDARD OIL Co. (U.S.P. 1,932,655, 31.10.33. Appl., 18.9.30).

Manufacture of gasoline. J. W. BROWN, Assr. to GRAY PROCESSES CORP. (U.S.P. 1,930,453, 10.10.33. Appl., 1.4.29).

Production of gasoline from petroleum hydrocarbons. A. D. SMITH and N. H. RANSTEAD, ASSIS, to JENKINS PETROLEUM PROCESS Co. (U.S.P. 1,928,985, 3.10.33. Appl., 19.6.29).

Gasoline purifier. H. THOMAS, ASST. to SUN OIL Co. (U.S.P. 1,929,907, 10.10.33. Appl., 10.4.30).

Resolution of oil-water suspensions and loose emulsions. H. F. FISHER, Assr. to PETROLEUM RECTIFYING CO. OF CALIFORNIA (U.S.P. 1,932,093, 24.10.33. Appl., 3.7.29).

Gas producer. Washing coal. [Oil] filters. Distilling apparatus.—See I. NO<sub>2</sub>-compounds.— See III. Saturating fabric [with asphalt etc.].— See VI. Al<sub>2</sub>O<sub>3</sub> from coal ash.—See VII. Road material.—See IX. Corrosion-inhibiting compound.—See X. Polymerised coumarone.—See XIII.

# III.—ORGANIC INTERMEDIATES.

Manufacture of solvents. L. PIATTI (Chem.-Ztg., 1934, 58, 353-355).—Short descriptions are given of: (1)  $C_6H_6$  recovery by activated C, (2) hydrogenation of  $C_{10}H_8$ , PhOH, etc., (3) synthesis of MeOH, (4) prep. of  $C_2H_2$  derivatives, (5) prep. of solvents from petroleum fractions and by fermentation methods. C. I.

 $[C_{10}H_8$  from coal gas by] refrigeration.—See I. Determining  $C_{10}H_8$  in gas. Miscibility of petrol with alcohols,  $C_6H_6$ , etc.—See II. Vulcanisation accelerators.—See XIV.

See also A., May, 496, Electro-org. oxidations of PhCHO, CH<sub>2</sub>Ph·OH, etc. 508, Prep. of chloropicrin from CH<sub>4</sub>. 516, Electrolytic prep. of semicarbazide hydrochloride. 517, Electrolysis of Et<sub>2</sub>O solutions of the Grignard compound. 520, Prep. of 3 : 3-diamino- and -dihydroxy-azobenzene and analogous compounds. 521, Prep. of di- $\beta$ -naphthol. 523, Prep. of aminosalicylic acid. 527, Prep. of 1 : 4-di-*p*-toluidinoanthraquinone. 530, Bases of the type of Fast-violet B. 533, 4-*p*-Dialkylaminophenylpyridines.

#### PATENTS.

Hydration of olefines. DISTILLERS Co., LTD., W. P. JOSHUA, and H. M. STANLEY (B.P. 408,982, 18.10.32).—  $C_3H_6$  and  $\Delta^{\alpha_-}$ ,  $\Delta^{\beta_-}$ , and *iso*- $C_4H_8$  are converted into the corresponding alcohols by passage with H<sub>2</sub>O over mixed orthophosphates (and borates) of Mn, Cu, Fe, Co, or U containing excess of free H<sub>3</sub>PO<sub>4</sub> at 100—300°/> 100 atm. The preferred catalyst is 1 Mn oxide: 2.6 H<sub>3</sub>PO<sub>4</sub>: 0.5 B<sub>2</sub>O<sub>3</sub>. H. A. P.

Treatment of [paraffin] hydrocarbons. C. D. LOOKER, E. B. KESTER, H. B. HASS, and W. Z. FRIEND, ASSTS. to BALTIMORE GAS ENG. CORP. (U.S.P. 1,923,630, 22.8.33. Appl., 27.2.30).—The hydrocarbons are treated with a N oxyhalide at 20—100°, and the product is hydrolysed (HCl).  $C_4H_{10}$  with NOCl gives, after hydrolysis, COMEEt, AcOH, EtCO<sub>2</sub>H, PrCO<sub>2</sub>H, chlorobutanes, NH<sub>2</sub>OH,HCl, and NH<sub>4</sub>Cl. H. A. P.

Oxidation of gaseous hydrocarbons. J. E. BLUDWORTH, ASST. to HANLON-BUCHANAN, INC. (U.S.P. 1,923,610, 22.8.33. Appl., 24.12.30).—A saturated gaseous hydrocarbon and an oxidising gas (air) are introduced into a long, narrow tube (T) at an initial temp. of about 200° and heated during passage through T to 230—480° with further injection(s) of air in order to bring about a stepwise oxidation. MeOH, COMe<sub>2</sub>, and MeCHO are formed. Apparatus is claimed.

## H. A. P.

Distillation [of products of chlorination of pentane]. E. E. AYRES, JUN., Assr. to FIDELITY PHILA-DELPHIA TRUST Co. (U.S.P. 1,926,189, 12.9.33. Appl., 4.2.27. Renewed 27.1.33).-After the formation of amyl alcohol (I), amyl chloride (II), and other chlorides by the chlorination of C<sub>5</sub>H<sub>12</sub> followed by hydrolysis, a const.-boiling mixture (C.B.M.) of (I) and (II) is distilled off in a rectifying still (A) and the condensate returned to the next stage of hydrolysis; subsequently a quantity of H<sub>2</sub>O is maintained in A until the higherboiling chlorides have been removed with it as C.B.M., the (I) which distils over therewith being separated in the condensate by gravity and returned to the next distillation. Finally the still is operated to yield finished (I). B. M. V.

Manufacture of high-molecular oxygen and thioethers. HENKEL & Co. G.M.B.H. (B.P. 409,030, 26.10.32. Ger., 29.12.31).—Aliphatic (>  $C_8$ ) or cycloaliphatic mono-OH- or -SH-compounds free from CO<sub>2</sub>H are heated with glycide (I), epichlorohydrin (II), or thioglycide (III) (and an etherification catalyst). Examples are cyclohexyl, b.p. 128—130°/12 mm., and dodecyl  $\gamma$ -chloro- $\beta$ -hydroxy-*n*-propyl ether, b.p. 193—195°/13.5 mm., dodecylthiol + (II), naphthenylthiols + (II), and dodecyl alcohol + (III), (I), or (II) (equal wts.).

H. A. P.

Production of vinyl compounds. BRIT. CELANESE, LTD. (B.P. 409,132, 18.4.33. U.S., 16.4.32).—A vinyl halide (chloride) is condensed with a phenol in presence of AlCl<sub>3</sub>, FeCl<sub>3</sub>, etc. The products are polymerised (by ultra-violet light). H. A. P.

Manufacture of esters. (A) O. SPRING, (B) S. R. MERLEY and O. SPRING, and (c) S. R. MERLEY, ASSIS. to DOHERTY RES. CO. (U.S.P. 1,924,575 and 1,924,615—6, 29.8.33. Appl., [A] 4.8.30, [B] 11.6.28, [C] 18.9.30).— Alkyl sulphates are treated with a salt of an org. carboxylic acid [Ca(OAc)<sub>2</sub>] in (A) CCl<sub>4</sub> at  $\leq 90^{\circ}$  under pressure, (B) saturated liquid hydrocarbons at approx. 90°, and (c) a stable ester (hexyl acetate) at  $\leq 80^{\circ}$ , the appropriate alcohol is added to combine with free acid formed, any excess of H<sub>2</sub>SO<sub>4</sub> is neutralised [Ca(OH)<sub>2</sub>], and the ester separated by fractional distillation. Apparatus is claimed. H. A. P.

[Preparation and uses of] octyl alcohol esters. L. P. KYRIDES, ASST. to MONSANTO CHEM. Co. (U.S.P. 1,923,938, 22.8.33. Appl., 23.9.29).—Esters of  $\beta$ -ethyl*n*-hexanol (phthalate, acetate) (preps. described) are used as plasticisers for nitrocellulose and synthetic resins.

H. A. P.

Manufacture of high-molecular organic esters. HENKEL & Co. G.M.B.H. (B.P. 408,754 and 408,749, [A] 22.12.32, [B] 20.12.32. Ger., [A, B] 4.1.32. [A] Addn. to B.P. 403,883; B., 1934, 355).—Esters of nonaromatic halogenated acids containing  $\ll C_2$  and aliphatic or cycloaliphatic OH-compounds containing  $\ll C_6$  are (A) combined with tert.-amines and (B) condensed with org. OH-compounds or their alkali derivatives. Examples are: (A) pyridinium salts from dodecyl, octadecyl (I), and cyclohexyl chloroacetates, mixed dodecyl  $\gamma$ -chloro-butyrate and -valerate, and the chloroacetates of the alcohols corresponding to the fatty acids of coconut oil, and the NHEt<sub>3</sub> salt from (I), and (B) dodecyl phenoxyacetate, m.p. 33—34°, b.p. 240— 245°/17 mm., and phenylethyl ethoxyacetate, b.p. 148—152°/12 mm. H. A. P.

Preparation of wax-like esters. D. K. TRESSLER, Assr. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,925,063, 29.8.33. Appl., 4.12.29).—(Alkali or alkalineearth) salts of higher fatty acids are condensed with chlorohydrins of unsaturated fatty acids, esters (glycerides), or ketones. Examples are Na stearate and "lactonic ester" of oleic acid chlorohydrin at 160°, chlorohydrin of lard oil at 177°, Et oleate chlorohydrin at 160°, and oleone chlorohydrin at 187°. H. A. P.

Cleaning agents. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 408,708, 28.10.32).—The cleansing action of synthetic detergents of sulphonic acid or sulphuric ester type containing  $\ll C_8$  is improved by admixture with a H<sub>2</sub>O-sol. metaphosphate. H. A. P.

(A, B, E-H) Preparation of [aliphatic] carboxylic acids. Preparation of (c) aliphatic, (d) [aliphatic], carboxylic acids from olefinic hydrocarbons, carbon monoxide, and steam. (A) J. C. WOODHOUSE, (B, E-H) G. B. CARPENTER, (C) W. E. VAIL, and (D) A. T. LARSON and W. E. VAIL, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,924,762-9, 29.8.33. Appl., [A] 11.11.32, [B] 28.6.32, [C] 31.12.31, [D] 25.11.31, [E-H] 24.8.31).-(E) An olefine, CO, and steam are caused to interact at 100-500° (325°)/25-900 (700) atm. in presence of a non-volatile inorg. acid catalyst (H<sub>3</sub>PO<sub>4</sub>-C). Examples cover 2-5 pts. of  $C_2H_4$  + 70—90 pts. of CO + 8—25 pts. of  $H_2O \rightarrow EtCO_2H$ . Other catalysts, preferably used on active C, are: (A) volatile inorg. acids formed in situ (HCl from chlorinated hydrocarbons) or Cl<sub>2</sub>, (B) volatile halides (H halides) + a solid acidic catalyst (H<sub>3</sub>PO<sub>4</sub>), (c) B phosphate, (D) NH4 halides, (F) H halides + halides of alkali or alkalineearth metals, Cu, Mg, Fe, Co, Ni, Bi, Mn, Pb, Te, Zn, Cd (HCl +  $ZnCl_2$  or CaCl<sub>2</sub>, or HBr + NaBr), (G) combined acidic oxides from groups III-VI (silico-tungstic or -molybdic or phosphotungstic acids, Cr vanadate), and (H) the halides of (F) alone. H. A. P.

Manufacture of hydroxydicarboxylic acids. STANDARD BRANDS, INC. (B.P. 408,834, 22.6.33. U.S., 18.7.32).—An unsaturated dicarboxylic (fumaric) acid (I) is oxidised (to tartaric acid) by a chlorate (II) and  $OsO_4$  at 40—55° in > 10% (20—25%) solution, (I) and (II) being added together in equiv. proportions as reaction proceeds. Os present in the gases evolved is recovered by passage through saturated aq. (I).

H. A. P.

Manufacture of salts of polyhydroxy[mono]carboxylic acids derived from polyaldoses. CHEM. WORKS, FORMERLY SANDOZ (B.P. 408,987, 18.10.33. Switz., 22.10.32).—The salts of metals of groups VII and VIII (I) (Mn, Fe, Ni, Co) with polyhydroxymonocarboxylic acids (lactobionic) (II) are prepared by metathesis from (II) or their Pb, Ca, Sr, or Ba salts and salts or oxides etc. of (I), and are isolated by evaporation or pptn. with an org. solvent (MeOH, EtOH). H. A. P.

[Manufacture of] soluble fatty acid condensation products. I. C. SOMERVILLE, ASST. to RÖHM & HAAS Co. (U.S.P. 1,924,052, 22.8.33. Appl., 9.5.30).—A H<sub>2</sub>O-sol. phenol-aldehyde resin is condensed at  $> 200^{\circ}$  with a long-chain unsaturated acid, *e.g.*, oleic or ricinoleic, and the product is sulphonated with the usual reagents to give compounds which are useful as tanning agents, mordants for basic dyes, wetting agents, etc. S. M.

Recovery of furfural[dehyde] from filter-cakes containing it. A. O. JAEGER, ASST. to SELDEN CO. (U.S.P. 1,923,672, 22.8.33. Appl., 28.5.30).—Furfuraldehyde (I) is recovered from filter-cakes from purification of hydrocarbons (anthracene) by distillation in steam, with addition of a H<sub>2</sub>O-insol. solvent for (I) if desired.

H. A. P.

Production of aminotrimethylbenzenes. A. ALT, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,923,697, 22.8.33. Appl., 13.11.29).—N-Methylxylidines are heated with halides of group II (ZnCl<sub>2</sub>) at 230—300° (250—290°). H. A. P.

Manufacture of [hydr]oxyethylated [A] By-di-[hydr]oxypropylaminobenzenes and [B]  $\beta$ -[hydr]oxy-y-alkoxypropylaminobenzenes. I. G. FARBEN-IND. A.-G. (B.P. 409,237 and Addn. B.P. 409,238, [A] 21.11.33, [B] 22.11.33. Ger., [A] 24.12.32, [B] 15.2.33).---(A) The corresponding aryl-βγ-dihydroxypropylamines and (B) their  $\gamma$ -alkyl ethers are heated with  $(CH_2)_2O$  (at 150—160°) under pressure. Examples are the  $N-\beta$ -hydroxyethyl derivatives of (A)  $\beta\gamma$ -dihydroxypropylaniline, b.p. 244-245°/12 mm., -m-toluidine, b.p. 247-248°/12 mm., and -3-amino-p-tolyl Me ether, b.p. 237-242°/12 mm., and (B) β-hydroxy-γ-methoxypropylaniline, b.p. 212-214°/11 mm., β-hydroxy-γ-ethoxypropyl-m-toluidine, b.p. 218°/11 mm., and 3-β-hydroxyγ-methoxypropylamino-p-tolyl Me ether, b.p. 213-214°/11 mm. H. A. P.

**Preparation of trichlorobenzene [and p-dichlorobenzene].** E. C. BRITTON, Assr. to Dow CHEM. Co. (U.S.P. 1,923,419, 22.8.33. Appl., 16.3.32).—The crude eutectic mixture of o- and p-C<sub>6</sub>H<sub>4</sub>Cl<sub>2</sub> is chlorinated at 30—50° (40—50°) until half-converted into C<sub>6</sub>H<sub>3</sub>Cl<sub>3</sub> (I) (the o-C<sub>6</sub>H<sub>4</sub>Cl<sub>2</sub> is preferentially chlorinated), the (I) and unchlorinated C<sub>6</sub>H<sub>4</sub>Cl<sub>2</sub> are separated by distillation, and p-C<sub>6</sub>H<sub>4</sub>Cl<sub>2</sub> is separated from the latter by crystallisation. Apparatus is claimed. H. A. P.

Manufacture of nitro-compounds. J. F. O'BRIEN (U.S.P. 1,922,709, 15.8.33. Appl., 20.12.27).—The coaltar fraction of b.p. 95—115°,  $d \ 0.862$  is heated under reflux to remove fractions of lower b.p. until its d is 0.904. The product is nitrated first at 20°, and then at  $30-70^{\circ}$ , with HNO<sub>3</sub> ( $d \ 1.44$ ) and conc. H<sub>2</sub>SO<sub>4</sub>, and yields an explosive ingredient of the approx. composition:  $o-C_6H_4Me\cdotNO_2$  39,  $p-C_6H_4Me\cdotNO_2$  58, and hydrocarbons 3%. H. A. P.

Manufacture of symmetrically-substituted azobenzene compounds. I. G. FARBENIND. A.-G. (B.P. 408,676, 14.10.32. Ger., 17.10.31).—A 2-halogeno-5nitro-benzenesulphonic or -benzoic acid is reduced to the azo compound (or to hydrazo, which is oxidised to the azo compound) and the Cl substituted by NH<sub>2</sub> or OH. Examples are: 4:4'-dichloro-  $\rightarrow 4:4'$ - dihydroxy-azobenzene-3 : 3'-dicarboxylic acid, and 2 : 5 :  $1-C_6H_3Cl(NO_2)\cdot SO_3H \rightarrow hydrazo \rightarrow azo compound \rightarrow 4 : 4'-diaminoazobenzene-3 : 3'-disulphonic acid.$ 

H. A. P.

Carrying out the Friedel-Crafts reaction. BRIT. CELANESE, LTD. (B.P. 409,131, 18.4.33. U.S., 16.4.32).— Hydrated FeCl<sub>3</sub> is used as catalyst. Examples are PhOH (1 mol.), Bu<sup> $\gamma$ </sup>Cl (1 mol.) or *tert*.-amyl chloride, and FeCl<sub>3</sub>,6H<sub>2</sub>O (0 · 2 mol.) at 80—90°. H. A. P.

Preparation of [C-]alkylated phenols. Röhm & HAAS Co. (B.P. 409,111, 24.2.33. U.S., 23.3.32).— Phenols are treated at  $< 70^{\circ}$  (0—25°) with di- or triisobutylene, diisoamylene, or higher homologues of these and < 1 equiv. of an acidic catalyst (0.075—0.3 mol. of conc. H<sub>2</sub>SO<sub>4</sub>). Examples are ααγγ-tetramethylbutylphenol, m.p. 84—85° [-phenoxyacetic acid (CH<sub>2</sub>Cl·CO<sub>2</sub>H), m.p. 108—109°], -o-, m.p. 49—50°, and -p-cresol, m.p. 46—47°, and -β-naphthol, m.p. 128—129°. H. A. P.

Manufacture of phenates [phenoxides]. J. J. GREBE and W. H. Dow, Assrs. to Dow CHEM. Co. (U.S.P. 1,924,313, 29.8.33. Appl., 29.6.28).—PhCl and aq. NaOH are mixed at < the reaction temp. (320°) and heated by superheated steam to > 320°. Plant is described. H. A. P.

Preparation of amylcresols [bactericides]. W. G. CHRISTIANSEN, G. RIDGE, and W. A. LOTT, Assrs. to E. R. SQUIBB & SONS (U.S.P. 1,922,153, 15.8.33. Appl., 15.1.32).—A cresol (o-) is condensed with amylene in presence of  $H_2SO_4$  at  $< 10^{\circ}$  (-15 to  $-5^{\circ}$  in Et<sub>2</sub>O). H. A. P.

Production of arylglycines. J. E. JEWETT, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,923,730, 22.8.33. Appl., 22.10.29).—An arylamine, alkali cyanide, and  $CH_2O$  are allowed to interact in presence of a  $H_2O$ sol, salt of the arylglycine (e.g., from a previous batch). H. A. P.

Production of aromatic thioacylamides. J. D. KENDALL, and ILFORD, LTD. (B.P. 408,638, 6.10.32).— The corresponding acylamide is converted into its iminohalide by a P<sup>v</sup> halide (PCl<sub>5</sub>) in an inert solvent capable of dissolving POCl<sub>3</sub> (C<sub>6</sub>H<sub>6</sub>), and a sol. sulphide (H<sub>2</sub>S) added with cooling (to  $< 0^{\circ}$ ). Examples include the prep. of: thioacet-*p*-phenetidide, m.p. 118—119°, -*p*-anisidide, m.p. 119°, -*o*-toluidide, m.p. 61—64°, -*m*-xylidide, 98—99°, - $\beta$ -naphthalide, m.p. 151°; 5thioacetamido-acenaphthene, m.p. 145°, and -pyrogallol Me<sub>3</sub> ether (in solution); 4-thioacetamido-1-methylnaphthalene, and - $\alpha$ -naphthyl Me, m.p. 135°, and Et ether, m.p. 117°; 1-thioacetamido- $\beta$ -naphthylEt ether; 2-thioacetamido-diphenylene oxide and -fluorene, m.p. 165°; thioacetamidoquinol Me<sub>2</sub> ether, m.p. 86—88°; *m*-nitrothioacetanilide; bisthioacet-*m*- and -*p*-phenylenediamide and -benzidide; and thiopropionanilide, m.p. 68—69°. H. A. P.

(A) 5-Amino-2-carboxy-, (B) 5-nitro-2-carboxy-, and (c) 5-nitro-2-cyano-diphenyl. W. S. JONES and W. BRAKER, ASSTS. to E. R. SQUIBB & SONS (U.S.P. 1,922,205-7, 15.8.33, [A] 26.1.32, [B, C] 15.2.32).—
(c) 5-Nitro-2-cyanodiphenyl, m.p. 132—134°, is prepared by the Sandmeyer reaction from the 2-NH<sub>2</sub>-compound, (B) hydrolysed by aq. H<sub>2</sub>SO<sub>4</sub> in AcOH to 5-nitrodiphenyl-

2-carboxylic acid, which (A) is reduced by Sn and HCl to the 5- $NH_2$ -compound. H. A. P.

Manufacture of phenylphenols [hydroxydiphenyls]. W. J. HALE, Assr. to Dow CHEM. Co. (U.S.P. 1,922,695, 15.8.33. Appl., 18.12.30).—PhCl or PhBr (1 equiv.) is heated with (added to) aq. alkali phenoxide (2 equivs.) and free phenol (1 equiv.) at 350—420° under pressure. Yields of 28% from PhCl and 40% from PhBr are obtained. H. A. P.

[Manufacture of] 2-naphthalenemonosulphonic acid. C. L. MASTERS, ASST. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,922,813, 15.8.33. Appl., 15.9.32).—  $1-C_{10}H_7 \cdot SO_3H$  formed during sulphonation of  $C_{10}H_8$  is preferentially hydrolysed by diluting the sulphonation mixture so that its b.p. is  $145-155^{\circ}$  ( $149-151^{\circ}$ ) and distilling away the  $C_{10}H_8$  formed at this temp. with steam at such a rate that the requisite concn. of acid is maintained. Apparatus is claimed. H. A. P.

Production of aryl[am]ides of 2:3-hydroxynaphthoic acid. T. S. KERE, ASST. to NAT. ANDINE & CHEM. Co. (U.S.P. 1,917,890, 11.7.33. Appl., 14.7.31).— 2:3-Hydroxynaphthoic acid is converted by heating with a slight excess of PCl<sub>3</sub>, POCl<sub>3</sub>, or SOCl<sub>2</sub> (at  $110^{\circ}$  the b.p.) in an inert medium (PhCl) into its chloride, which is then condensed with the appropriate amine.

H. A. P.

Production of nitrosulphonic acids of polyhydroxyanthraquinones. D. G. Rogers, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,924,166, Appl., 20.3.30).-Heteronuclear (aa'-)poly-29.8.33. hydroxyanthraquinones are sulphonated (22% oleum) at 80-115° (80-85°) in presence of an alkali sulphate, and, after seeding with the appropriate alkali salt of one of the products, nitrated. Thus technical 1:5-(containing 1:8-)dihydroxyanthraquinone is heated at 80-85° with 22% oleum and Na2SO4, seeded with Na2 1:5-dihydroxyanthraquinonedisulphonate, and nitrated  $(HNO_3-H_2SO_4 \text{ at } 25-35^\circ)$ . The product on reduction gives a blue acid dye. The NO<sub>2</sub>-compound may be separated from the 1:8-(OH)2-isomeride by pptn. by dilution and washing with H<sub>2</sub>O. H. A. P.

Preparation of amino - derivatives of anthraquinone series. P. H. GROGGINS, Dedicated to GOVT. AND PEOPLE OF U.S.A. (U.S.P. 1,923,618, 22.8.33. Appl., 30.7.32).—Hålogenoanthraquinones are heated under pressure with NH<sub>3</sub>, an inorg. oxyhalogen compound (salt), and other catalysts (Cu salts) if desired. Thus 2-chloroanthraquinone, with aq. NH<sub>3</sub> and small amounts of KIO<sub>3</sub> and Cu(NO<sub>3</sub>)<sub>2</sub>, at 195° gives 95% of theory of the 2-NH<sub>2</sub>-compound. H. A. P.

Manufacture of organo-lead compounds of phenols. E. I. DU PONT DE NEMOURS & Co. (B.P. 408,967, 18.10.32. U.S., 20.10.31).—Pb tetra-alkyls (PbEt<sub>4</sub>) are heated with phenols at 75—175° (140—160°), in a solvent in presence of SiO<sub>2</sub> gel, if desired. Compounds of the type ArO·PbEt<sub>3</sub> are formed, and are for use in varnishes etc. or as bactericides, fungicides, etc. Examples are : phenoxy-, decomp. 205°, otolyloxy-, decomp. 185°, resorcyloxybis-, decomp. 235°, and pyrocatechyloxybis-lead triethyl, decomp. 245°. H. A. P. Apparatus for production of gaseous ozonides. L. H. ROGERS, ASST. to KNOX TERPEZONE CO. of AMERICA (U.S.P. 1,924,805, 29.8.33. Appl., 23.10.30).—Ozonised air is passed through a tube (I) containing a spiral around which is wrapped a wick (II) kept moist with pinene from a container below (I) into which dips the lower end of (II). A. R. P.

C<sub>10</sub>H<sub>8</sub> from fuel gas.—See II. Dry-cleaning fluids. —See VI. HCl [from chlorinations].—See VII. Abs. EtOH.—See XVIII.

## IV.—DYESTUFFS.

## Colour lakes .- See XIII.

See also A., May, 487, Colour lakes. S dyes. 503, Abs. colorimeter. 533, 4-p-Dialkylamino-phenylpyridines. 535, Dyes from acenaphthene-quinone.

# PATENTS.

Manufacture of azo dyes [ice colours]. I. G. FARBENIND. A.-G. (B.P. 409,014, 22.10.32. Ger., 26.10.31).-Diazotised o-nitro-amines of the carbazole, fluorene, fluorenone, or diphenylene sulphide, -sulphone, or oxide series are coupled with 2 : 3-hydroxynaphthoic arylamides. Examples are: 2-nitro-3-aminodiphenylene oxide  $\rightarrow$  2:3-hydroxynaphthoic 5-chloro-2:4dimethoxyanilide (bordeaux) or β-naphthalide (I) (bluishred); 2-nitro-3-aminocarbazole  $\rightarrow$  2:3-hydroxynaphthoic 4-methoxy-o-toluidide or  $\rightarrow$  4-chloro-o-toluidide (II) (corinth), or  $\rightarrow \beta$ -naphthalide (bluish-violet); 3-nitro-2-aminofluorenone  $\rightarrow 2:$  3-hydroxynaphthoic-panisidide (brownish-red) or  $\rightarrow m$ -nitroanilide (brownishred); 3-nitro-2-aminofluorene  $\rightarrow$  2:3-hydroxynaphthoic 3-chloro-4: 6-dimethoxyanilide; 3-nitro-2-aminodiphenylene sulphide  $\rightarrow$  (II) or  $\rightarrow$  (I) (all red-bordeaux); and 3-nitro-2-aminodiphenylenesulphone  $\rightarrow$  (II) (red-H. A. P. orange).

Manufacture of [chromiferous] azo dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 408,590, 29.9.32) .- O-Ethers of sulphonated o-hydroxyazo dyes containing ·SO<sub>2</sub>·NH<sub>2</sub> or substituted ·SO<sub>2</sub>·NH<sub>2</sub> in the same nucleus as the OH-group are heated in H2O with Cr compounds (salts) at 100-150°, the O-alkyl (aryl etc.) group being eliminated. Examples are Cr compounds from :  $2:1:4-NH_2 \cdot C_6H_3(OMe) \cdot SO_2 \cdot NEt_2$  (I) (or  $\cdot$ NHEt,  $\cdot$ NHMe,  $\cdot$ NMe<sub>2</sub>,  $\cdot$ NHPh, or  $\cdot$ NPh<sub>2</sub>)  $\rightarrow$ 2:6:8-C10H5(OH)(SO3H)2 (II) (bordeaux); 5-Cl-derivative of  $(I) \rightarrow 1$ -m-sulphophenyl-3-methyl-5-pyrazolone (III) (orange);  $2:4:1:5-NH_2 C_6H_2Cl(OMe) SO_2 NH_2$  $\begin{array}{l} (\mathrm{IV}) \rightarrow (\mathrm{III}) \ (\mathrm{red}) \ ; \ 2:1:4\text{-}\mathrm{NH}_2 \cdot \tilde{\mathbb{C}}_6 \mathrm{H}_3 (\tilde{\mathbb{O}}\mathrm{Me}) \cdot \mathrm{SO}_2 \cdot \mathrm{NH}_2 \ (\mathbb{V}) \\ \rightarrow \ 2:4\text{-}\mathrm{dihydroxyquinoline-6-sulphonic} \ \mathrm{acid} \ (\mathrm{red}) \ ; \end{array}$ 5-OMe-derivative (VI) of (V)  $\rightarrow$  2:3:6-C<sub>10</sub>H<sub>5</sub>(OH)(SO<sub>3</sub>H)<sub>2</sub> (VII) (violet); (IV)  $\rightarrow$  (II) (violet) or  $\rightarrow$  (VII) (blueviolet); 2-aminoquinol Me<sub>2</sub> ether 5-sulphonamide  $\rightarrow$ (II) or 5-sulphondiethylamide  $\rightarrow$  (VII) (blue); (IV)  $\rightarrow$ 2:4-C10H6(OH)·SO3H (blue); (VI)  $\rightarrow$ 2:8:6-C10H5(OH)2·SO3H (blue-green); (I)  $\rightarrow$  8-hydroxyquinoline-5-sulphonic acid (yellow-brown); 1:2:6- $\begin{array}{l} \rm NH_2 \cdot C_{10}H_5(OMe) \cdot SO_2 \cdot NEt_2 \rightarrow 1:4:8 \cdot C_{10}H_5(OH)(SO_3H)_2 \\ (blue) \ or \ \rightarrow \ 1:8:3 \cdot C_{10}H_5(OH)(SO_2 \cdot NH_2) \cdot SO_3H \ or \ \rightarrow \end{array}$ 1:4:8-C<sub>10</sub>H<sub>5</sub>(OH)(SO<sub>2</sub>·NEt<sub>2</sub>)·SO<sub>3</sub>H (greenish-blue); and 2-aminophenyl Me ether 4-sulphon-cyclohexyl-, -benzyl-, or -diphenyl-amide  $\rightarrow$  (II). [Stat. ref.] H. A. P.

Manufacture of [tris]azo dyes. I. G. FARBENIND. A.-G. (B.P. 408,677, 14.10.32. Ger., 17.10.31).—Green direct cotton dyes are obtained by diazotising a disazo dye from tetrazotised benzidine or its derivatives coupled with a salicylic acid and a 1-amino- $\beta$ -naphthyl ether or its 6- or 7-SO<sub>3</sub>H and coupling the product with a 1-amino-8-naphtholsulphonic acid in presence of a *tert*.-amine (aq. C<sub>5</sub>H<sub>5</sub>N). Examples are : o-C<sub>6</sub>H<sub>4</sub>(OH)·CO<sub>2</sub>H (I)  $\leftarrow$ benzidine  $\Rightarrow$  1 : 2 : 6-NH<sub>2</sub>·C<sub>10</sub>H<sub>5</sub>(OH)·SO<sub>3</sub>Na  $\Rightarrow$  1 : 8 : 3 : 6-NHAc·C<sub>10</sub>H<sub>4</sub>(OH)(SO<sub>3</sub>H)<sub>2</sub> (II) or the corresponding Bz derivative ; (I) or 2 : 3-C<sub>6</sub>H<sub>3</sub>Me(OH)·CO<sub>2</sub>H  $\leftarrow$ o-tolidine  $\Rightarrow$  1 : 2-NH<sub>2</sub>·C<sub>10</sub>H<sub>6</sub>·O·C<sub>2</sub>H<sub>4</sub>·O·SO<sub>3</sub>H (III)  $\Rightarrow$ (II) ; and (I)  $\leftarrow$  3 : 3'-dichlorobenzidine  $\Rightarrow$  (III)  $\rightarrow$  (II). H. A. P.

[Manufacture of blue] printing colours of indanthrone series. E. I. DU PONT DE NEMOURS & Co. (B.P. 409,029, 26.10.32. U.S., 26.10.31).—Halogenated indanthrones (I) in hydroazine (or azine) form are treated with a mixture of sulphonating and dehydrating (Ac<sub>2</sub>O,  $P_2O_5$ , SO<sub>2</sub>Cl<sub>2</sub>) agents. Alternatively, (I) are treated with ClSO<sub>3</sub>H and added to (I) in H<sub>2</sub>SO<sub>4</sub>. H. A. P.

Manufacture of [cyanine] dyes. KODAK, LTD. From EASTMAN KODAK Co. (B.P. 408,569, 30.6.32) .--- A 2-halogeno- (iodo-) quinoline alkylo-salt is condensed with a 2-methyl-oxazole, -thiazole, or -selenazole alkylo-salt in presence of a base (NEt<sub>a</sub>). Examples are : 1:1':4trimethyl- (from 2:4-dimethyloxazole and 2-iodoquinoline methiodide), 4-phenyl-1-methyl-1'-ethyl-, 1:4dimethyl-1'-ethyl-, and 4-phenyl-1: 1'-diethyl-oxazoloψ-cyanine iodide; 1-methyl-1'-ethyl-, 1:4-dimethyl-1'ethyl-, 4-phenyl-1: 1'-dimethyl-, 4-phenyl-1: 1'-diethyl-, 4-phenyl-1: 6'-dimethyl-1'-ethyl-, 4:7'-dimethyl-1: 1'diethyl-, and 4-phenyl-1: 3-dimethyl-1'-ethyl-thiazolo- $\psi$ -cyanine iodide; 1:4:1'-trimethyl-, 4-methyl-1:1'-diethyl-, and 4-phenyl-1-methyl-1-ethyl-selenazoloψ-cyanine iodide. H. A. P.

Manufacture of cyanine dyes. KODAK, LTD. From EASTMAN KODAK Co. (B.P. 408,571, 30.6.32) .--The prep. of cyanine dyes (except 11-halogeno- or 11-nitro-dicarbocyanines) by methods involving the elimination of acids is carried out in presence of an equiv. amount of a strong org. base or its salt with a weak acid (cf. B.P. 408,569; preceding abstract). Typical examples are: 1:1'-diethyl- $\psi$ -cyanine iodide from quinaldine ethiodide, 2-iodoquinoline ethiodide, and NEt<sub>3</sub>; 2:1'-diethylthioisocyanine iodide from 2-methylbenzthiazole ethiodide (I), quinoline ethiodide, C5H5N, and NEt<sub>3</sub>; 9-bromo-2: 2'-diethylthiodicarbocyanine iodide from (I) (2 equivs.), NHPh•CH:CBr•CH:NPh, HBr, and piperidine (II); and 1:1'-diethyl-2:2'tricarbocyanine iodide from quinaldine etho-p-toluenesulphonate (2 equivs.), NHPh·[CH]<sub>5</sub>:NPh, HCl, and (II) H. A. P. (2 equivs.).

Sulphonated hydroxyanthraquinones.-See III.

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

Use of micro-drawings, photographs, and films in textile chemistry. H. REUMUTH (Chem.-Ztg., 1934, 58, 345-347).—Examples are described, with illustrations, of the use of photomicrographs and kinematomicrographs in textile chemistry. A. G. Circulation in sulphite digesters. O. BRUNE (Papier-Fabr., 1934, 32, 206-208).—Circulation with direct cooks depends on the shape of the digester (I), quantity of steam (II), and method of charging (III). The importance of keeping a correct (I) in relation to (II) and (III) is explained. D. A. C.

Effect of chip length in sulphite pulping. J. N. MCGOVERN and G. H. CHIDESTER (Paper Trade J., 1934, 98; T.A.P.P.I. Sect., 227-232).-Small-scale experiments to determine the effect of the length (L) of western hemlock (Tsuga heterophylla) heartwood chips, cooked by the sulphite process, on degree of penetration (P), and on fibre length (F), physical-strength characteristics (S), yield, and bleachability of the resulting pulp have been carried out. P increases parabolically with increasing L, but the rate of pulping is not significantly affected. S increases up to  $L \downarrow - \frac{1}{2}$  in., but not beyond. Disregarding ray cells there is a gradual decrease in the no. of shorter fibres and an increase in the no. of longer ones up to  $L \downarrow$  in., clearly indicating the reduction in F with decrease in L. Assuming that no reduction in F occurs during pulping, the chipping operation reduces F by 28% in the case of  $\frac{3}{4}$ -in. chips, and 67% with  $\frac{1}{4}$ -in. chips. Except for  $\frac{1}{4}$ - and  $\frac{1}{4}$ -in. chips F appears to have no direct bearing on S. H. A. H.

Comparison of the German and Swedish methods for determining the degree of cooking of sulphite and sulphate pulps. V. HOTTENROTH (Papier-Fabr., 1934, 32, 221-222).—A reply to criticisms of the German method of classification of the hardness of pulps (cf. Johansson, B., 1933, 1002; 1934, 446). D. A. C.

Snyder-Maclaren de-inking process [for paper]. F. H. SNYDER and S. F. M. MACLAREN (Paper Trade J., 1934, 98; T.A.P.P.I. Sect., 216—217).—Greatly improved results in the removal of printer's ink from groundwood papers are claimed by the use of Na silicate (of high  $SiO_2$  content) and palmitic or oleic acid. The advantages of such a mixture over Na oleate alone is attributed to the low surface-tension properties of the colloidal solution. The process is also applicable to chemical wood papers. Simplicity of operation, low cost, and retention of the original fibre characteristics are claimed as the chief advantages. H. A. H.

Viscosity-testing procedure for [paper-]coating colour, and standardisation of laboratory viscosimeters. P. S. DUNN and C. G. LANDES (Paper Trade J., 1934, 98; T.A.P.P.I. Sect., 213-216).—A no. of testing variables are examined; frothing is stated not to affect results. Glycerin solutions are satisfactory for standardisation. H. A. H.

Sources of error in measuring opacity of paper by the contrast-ratio method. D. B. JUDD (Bur. Stand. J. Res., 1934, 12, 345—351).—The contrast ratio is the ratio of the brightness of the illuminated sample when backed by a black-lined cavity to its brightness when backed by MgO. The errors obtained when other materials are substituted for MgO are evaluated.

E. S. H.

See also A., May, 485, Capillary penetration of fibrous materials. 539, Sericin fractions of silk.

## ......PATENTS. ... mi mitolandi)

Separation of bast fibres from shives. C. C. HERITAGE, E. R. SCHAFER, and L. A. CARPENTER, Dedicated to U.S.A. (U.S.P. 1,922,366, 15.8.33. Appl., 17.11.31).—Seed flax straw is separated into bast fibres and shives by shaking to remove chaff etc., cutting, and extracting with org. solvents (e.g., EtOH- $C_6H_6$ ). The solvent is removed and the material heated with dil. alkali, washed, dried, and shredded. It is then screened (a) through a 12-cut, (b) through an 8-cut plate screen, and (c) through a 24-mesh wire. The material retained at (c), which contains 85% of bast fibre, is then passed over a Wilfley table. D. A. C.

Manufacture of artificial leather. R. B. HnL, Assr. to Brown Co. (U.S.P. 1,923,622, 22.8.33. Appl., 16.1.29).—A porous cellulose web (paper or felt) is impregnated with latex and a vegetable tanning agent containing conc. waste sulphite liquor and then dried. F. R. E.

Manufacture of shoe stiffeners. S. P. LOVELL, Assr. to ARDEN BOX TOE CO. (U.S.P. 1,923,631, 22.8.33. Appl., 9.10.31).—A bibulous fibrous foundation, e.g., felt, is impregnated with a solution of nitrocellulose, preferably in a volatile,  $H_2O$ -sol. solvent, e.g., EtOAc, dried, steamed, and cut to shape. F. R. E.

Preparation of cellulose derivatives. G. B. ELLIS. From Soc. DES USINES CHIM. RHÔNE-POULENC (B.P. 408,730, 22.11.32).—Cellulosic material (sulphite pulp, bleached rags, cotton, etc.), after impregnation with an org. or inorg. salt [NaCl, KCl, MgClO<sub>4</sub>, Ba(CNS)<sub>2</sub>, KOAc, benzylpyridinium chloride], is treated with liquid or gaseous (CH<sub>2</sub>)<sub>2</sub>O; the reaction product may be esterified or etherified with or without previous removal of the salts. F. R. E.

Improvement of cellulose derivatives. I. G. FARBENIND. A.-G. (B.P. 408,806, 11.4.33. Ger., 11.4.32). —Diglycollates of ethers of polyhydric alcohols containing only 1 free OH (e.g., monoalkyl ethers of ethylene,  $\alpha\beta$ -propylene, and  $\alpha\gamma$ -butylene glycols) are used as softening agents and plasticisers for cellulose nitrate or acetate, or ethylcellulose, etc. Fastness to light is claimed. H. A. P.

Production of lustreless artificial silks. A. T. MAXIMOFF, ASST. to CHÂTILLON SOC. ANON. ITAL. PER LA SETA ARTIF. (U.S.P. 1,923,495, 22.8.33. Appl., 21.5.29. Ital., 8.1.29).—2—10% of milk is added to the cellulose solution, e.g., viscose, before spinning. F. R. E.

Manufacture of (A) dull-lustre artificial silk, (B) filaments, threads, etc. (A) M. WADEWITZ and R. ELSSNER, (B) R. ELSSNER and H. L. JOHNSON, ASSTS. to AMER. GLANZSTOFF CORP. (U.S.P. 1,922,903 and 1,922,910, 15.8.33. Appl., [A] 30.9.32, [B] 1.4.33).— A delustrant, (A) Ph<sub>2</sub> and pine oil dissolved in  $C_6H_6$ , or (B) Ph<sub>2</sub> and  $C_{10}H_8$  in powder form or dissolved in  $C_6H_6$ , is added to the cellulose solution [(A, B) viscose, or (B) cuprammonium] before spinning. F. R. E.

Manufacture of artificial sponges [from viscose]. I. G. FARBENIND. A.-G. (B.P. 408,617, 12.10.33. Ger., 12.10.32).—The sponge mass, consisting of viscose, fibrous material, and salts, is pressed continuously through a tube which is heated near its exit to cause coagulation, either complete or sufficient to enable the mass to preserve its shape; coagulation may be assisted by introduction of steam into the tube or by use of a tapered tube, and, if necessary, may be completed subsequently in a hot salt solution. F. R. E.

Manufacture of chemical pulp. J. H. Ross, JUN. (U.S.P. 1,922,262, 15.8.33. Appl., 31.10.30).—The optimum concn. of cooking liquor is maintained in the digester (D) throughout the cooking process by injecting under pressure fresh liquor into D through the external circulating system, the corresponding quantity of liquor being withdrawn from D through a relief valve.

D. A. C. Sulphite-cellulose digestion and removal of turpentine and other volatile matters present in the chips. G. HAGLUND, Assr. to PATENTAKTIEE. GRÖNDAL-RAMÉN (U.S.P. 1,922,160, 15.8.33. Appl., 25.3.32. Swed., 15.4.31).—The chips are first heated at 110—150° with aq. alkali sulphite to vaporise the turpentine, oils, etc., which are led off with the steam, condensed, and separated. The residue is then subjected to the usual sulphite digestion. It is claimed that the production of sticky resins is thereby inhibited.

D. A. C.

Purification [decolorisation] of cellulose material. H. P. BASSETT (U.S.P. 1,923,292, 22.8.33. Appl., 31.8.31).—The material, e.g., kraft pulp, is pretreated by agitating or heating with aq.  $HNO_3$  (0.05—5%), separated, washed, treated with hot alkali (8—10% NaOH), again washed, and bleached. F. R. E.

Removal of mechanical impurities from cellulose and other similar fibre pulps. A. LAMPÉN (U.S.P. 1,926,546, 12.9.33. Appl., 2.4.32. Finl., 9.4.31).— The pulp is rapidly projected along a foraminous surface by a sort of beater, with the object of allowing liquid and impurities to pass through the screen while retaining a moving layer of pulp thereon, which is skimmed off at a no. of points sufficiently close to prevent formation of an impervious layer of pulp. B. M. V.

Lithographic re-transfer paper. K. NAUMANN and E. NESTLER (R. NAUMANN UMDRUCKPAPIERFABR.) (B.P. 408,872, 18.4.33. Ger., 5.4.33).—The re-transfer paper is lightly marked on the rear side either by painting or water-marking, to indicate the direction of orientation of the fibres. D. A. C.

Coloured paper.-See VI.

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

Dyeing machinery and dye-works equipment. F. C. CRAWSHAW (J. Soc. Dyers & Col., 1934, 50, 149).— A summary of a lecture dealing mainly with constructional materials.

See also A., May, 503, Abs. colorimeter.

## PATENTS.

Coloration of [cellulose ester or ether] materials. BRIT. CELANESE, LTD., H. C. OLPIN, and G. H. ELLIS (B.P. 408,574, 1.10.32).—o-Nitro-amines containing  $\leq$ 1 acylamido-group in the same or a different nucleus are claimed. Examples are 1:2:5-NH<sub>2</sub>·C<sub>6</sub>H<sub>3</sub>(NO<sub>2</sub>)·NHBz (greenish-yellow), 1:2:4-NH<sub>2</sub>·C<sub>6</sub>H<sub>3</sub>(NO<sub>2</sub>)·NHAc, and

Fixation of dyes on coloured paper. C. Z. DRAVES, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,926,614, 12.9.33. Appl., 24.7.31).—The paper is dyed with an acid dye which is fixed by dipping the paper in a 0.5—25% solution of a di- or tri-arylguanidine, *e.g.*, the Ph<sub>2</sub> compound, which ppts. the dye. A. R. P.

Saturating of fabric. J. ZAVERTNIK and A. A. MACCUBBIN, Assrs. to BARRETT Co. (U.S.P. 1,920,541, 1.8.33. Appl., 11.9.29).—Paper, felt, and especially roofing paper is first sprayed with the hot tar, asphalt, or other impregnating liquor (L), and is then led on a supporting perforated surface (e.g., a rotating drum) through a bath of L, suction being applied from the supported side in order to secure penetration of L.

A. J. H.

Treatment of textile fabrics. BRIT. CELANESE, LTD., H. DREYFUS, and W. A. DICKIE (B.P. 408,654, 8.7.32).—Fabrics (yarn) of cellulose acetate or other org. derivative are treated with solvents so as to produce a reduction in length of thread of 5—30% (10—15%). Suitable solvents are 35—55% aq. COMe<sub>2</sub> and 38—43% aq. dioxan. A springy fabric with a dull lustre and soft handle is produced. H. A. P.

Impregnation of fibres with rubber. U. PESTA-LOZZA, and SOC. ITAL. PIRELLI (B.P. 408,213, 30.9.32).— Thorough penetration of rubber latex into yarn is ensured by pre-wetting the yarn with a hot solution of an alkali or  $NH_4$  soap, using a yarn which is only slightly twisted, and treating it in an untensioned condition.

A. J. H.

Waterproofing of wool. MERKEL & KIENLIN G.M.B.H. (B.P. 408,985, 20.10.32. Ger., 21.10.31).— The matting and loss of air-permeability which occur when wool is waterproofed by means of an Al soap are avoided by pretreating the wool with an oxidising agent, e.g.,  $Cl_2$ , HOCl,  $KMnO_4 + H_2SO_4$ ,  $H_2O_2$ . A. J. H.

**Dry-cleaning processes and apparatus therefor.** DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 405,662, 9.8.32. Ger., 4.3.32).—Rapid drying of textile material containing residual dry-cleaning solvent (CCl<sub>4</sub>, C<sub>3</sub>HCl<sub>3</sub>) and contained in the perforated drum (*D*) usually employed is effected by means of a current of hot air admitted through one or both ends of *D* instead of through the central shaft as is usual. A. J. H.

Clarification of used dry-cleaning fluids. R. A. MORGEN (U.S.P. 1,926,813, 12.9.33. Appl., 11.7.31).— The solvent is treated with activated siliceous material, *e.g.*, Creede clay, to adsorb acidic constituents, and simultaneously with alkali to neutralise these, all being removed together by filtration or settling. B. M. V.

Cleaning agents. Fatty acid condensation products. Alkylated phenols.—See III. Adhesive solution.—See XV.

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

Determination of sodium in pure and commercial potassium salts by the uranyl acetate method. F. ALTEN and H. WEILAND (Mitt. Kali-Forsch.-Anst., 1933, 75, 11—16; Chem. Zentr., 1933, ii, 2860).— Details are given for determining Na<sub>2</sub>O in pure and commercial K salts, in K salts containing phosphate, and in mixed fertilisers. L. S. T.

Manufacture and uses of "dry ice" [solid carbon dioxide]. W. POHLMANN (Chem.-Ztg., 1934, 58, 336—338).—Various German machines for production of solid  $CO_2$  (I) are described. Whereas in America 90% of the output of (I) is used in the prep. of icecream, in England 40% is sold for use as  $CO_2$  and not as a refrigerant. Reasons for the slight development of the industry in Germany are discussed. C. I.

Oxygenating cyanide solutions.—See X. Determining K in mixed fertilisers.—See XVI. Solid  $CO_2$ .—See XIX. Ca glycerophosphate.—See XX. Determining CO in air.—See XXIII.

See also A., May, 498, Prep. of  $N_2H_4$ ,  $H_2SO_4$ ,  $SCl_2$ , and  $AsCl_3$ .

## PATENTS.

Manufacture of hydrochloric acid. J. P. BAXTER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 408,578, 7.10.32).—Gaseous HCl derived from org. chlorinations is passed with live steam up an absorption tower (T)in countercurrent to preheated H<sub>2</sub>O at 100—110°, the effluent vapours serving to preheat the H<sub>2</sub>O. A 20% solution of HCl is collected from the base of T.

W. J. W.

Baking powder. E. H. WIGHT, ASST. to OBERPHOS Co. (U.S.P. 1,926,572, 12.9.33. Appl., 8.8.31).— $H_3PO_4$ and sufficient CaO to form CaHPO<sub>4</sub> are digested in an autoclave under a pressure (P) sufficiently high to maintain the mixture in a slurry, P is then released to evaporate most of the  $H_2O$ , and other ingredients, e.g., NaHCO<sub>3</sub>, are added and the mixture is dried in vac. with agitation. A. R. P.

(A, B) Extracting the phosphorus content from phosphorus-containing materials, (B) and the potassium content from potassium-containing materials. (A, B) R. E. VIVIAN and (B) C. G. FINK, Assrs. to INTERNAT. AGRICULTURAL CORP. (U.S.P. 1,926,072-3, 12.9.33. Appl., 3.6.31).--(A) An intimate mixture of phosphate rock and C is heated at 700° in a current of Cl<sub>2</sub> to produce CaCl<sub>2</sub> and volatilise PCl<sub>3</sub> and POCl<sub>3</sub>. (B) Orthoclase (I) is added to the above mixture and the resulting gases are passed over a hot mixture of (I) and C to produce AlCl<sub>3</sub>, FeCl<sub>3</sub>, PCl<sub>3</sub>, and KCl. A. R. P.

Preparation of [a base-]exchange silicate. H. M. GRAY, ASST. to H. W. MARKS (U.S.P. 1,926,618, 12.9.33. Appl., 3.3.30).—Aq. Na<sub>2</sub>SiO<sub>3</sub> is run slowly into aq. Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> containing HNO<sub>3</sub> so as to obtain a gel containing only half the possible Na content; washing and drying are carried out as usual. On running hard H<sub>2</sub>O through the product a neutral effluent is obtained. A. R. P.

Manufacture of double salts of calcium nitrate. E. LÜSCHER, ASST. to LONZA ELEKTRIZITÄTSWERKE U. CHEM. FABR. A.-G. (U.S.P. 1,926,492, 12.9.33. Appl., 11.5.31. Switz., 13.5.30).—Double salts of  $Ca(NO_3)_2$  and  $KNO_3$  or  $NH_4NO_3$ , e.g.,  $5Ca(NO_3)_2$ ,  $NH_4NO_3$ ,  $10H_2O$ , are crystallised from hot solutions and the hot crystals while still moist with mother-liquor are dusted with

CaCO<sub>3</sub> dust to produce a scatterable cryst. powder. A. R. P.

Preparation of calcium chloride [sticks]. H. RYNALSKI, ASST. to SOLVAY PROCESS CO. (U.S.P. 1,926,653, 12.9.33. Appl., 30.10.30).—The fused salt is cast into moulds containing an Fe wire or rod which acts as a reinforcement. A. R. P.

[Froth] flotation [of fluorspar]. O. W. GREEMAN and H. A. LILLY, Assrs. to ALUMINUM CO. OF AMERICA (U.S.P. 1,926,045, 12.9.33. Appl., 7.8.31).—Prior to flotation of CaF<sub>2</sub>, an oxidising agent (e.g., KMnO<sub>4</sub>,  $K_2Cr_2O_7$ , or Cl<sub>2</sub>) is added to the treatment H<sub>2</sub>O together with a modifier comprising a cresol or xylenol. The frothing agent may be oleic acid. B. M. V.

Extraction of alumina [from coal ash]. R. F. JAMES, ASST. to DETROIT EDISON CO. (U.S.P. 1,926,744, 12.9.33. Appl., 4.11.29).—Ash from furnaces fired with pulverised coal is treated on an electromagnet to separate Fe material, then mixed with  $1 \cdot 25$ —2 mols. of Na<sub>2</sub>CO<sub>3</sub> per mol. of Al<sub>2</sub>O<sub>3</sub> and 1—2 mols. of CaCO<sub>3</sub> per mol. of SiO<sub>2</sub>, and the mixture is sintered at 1200°, ground, and leached with H<sub>2</sub>O to recover NaAlO<sub>2</sub> and leave a CaO–SiO<sub>2</sub> residue from which cement clinker can be made.

A. R. P.

Activation of clay. F. W. HUBER (U.S.P. 1,926,148, 12.9.33. Appl., 27.8.32).—Bentonite or montmorillonite is heated with 7—35% of HCl or  $H_2SO_4$  in a 2:1 pulp ratio (R) with violent agitation. The acid is then removed by settling and decantation, R being kept const. A. R. P.

Manufacture of sexavalent chromium compounds from chromium alloys. M. J. UDV, ASST. to ELECTRO METALLURG. Co. (U.S.P. 1,926,758, 12.9.33. Appl., 23.7.28).—Finely-divided ferrochromium is mixed with CaO and about 1% of Na<sub>2</sub>CO<sub>3</sub> and the mixture is roasted to produce Fe<sub>2</sub>O<sub>3</sub> and CaCrO<sub>4</sub>. The product is leached with dil. H<sub>2</sub>SO<sub>4</sub> and a slight excess of CaCO<sub>3</sub> is added to ppt. dissolved Fe<sup>11</sup> and leave a solution of CrO<sub>3</sub>.

A. R. P.

Manufacture of zinc [dihydrogen] phosphate. V. M. DARSEY, ASST. to METAL FINISHING RES. CORP. (U.S.P. 1,926,266, 12.9.33. Appl., 7.2.31).—Zn is dissolved in aq.  $H_3PO_4$  to give a solution containing 15% of Zn and 50—55\% of  $H_3PO_4$ , which yields on slow cooling orthorhombic crystals of Zn(HPO<sub>4</sub>)<sub>2</sub>,2H<sub>2</sub>O.

A. R. P.

Apparatus for liquefying [melting] carbon dioxide. L. L. MOHLER, Assr. to CO-2 APPLIANCE Co. (U.S.P. 1,925,492, 5.9.33. Appl., 2.9.31).—A pressure vessel with thermostatically controlled heating element for converting solid CO<sub>2</sub> into liquid is described.

B. M. V.

Manufacture of carbon dioxide ice. F. R. ZUMBRO, Assr. to FRICK Co. (U.S.P. 1,925,619, 5.9.33. Appl., 28.4.30).—A mixture of CO<sub>2</sub> liquid and snow is

pressed to  $\ll 60.4$  lb./sq. in., preferably by a plunger, in a vessel surrounded by refrigerant (*R*) below the f.p. of CO<sub>2</sub> at that pressure. *R* may be CO<sub>2</sub> snow under atm. pressure and produced by expanding CO<sub>2</sub> liquid *in situ*. B. M. V.

**Production of hydrogen.** F. HANSGIRG, Assr. to AMER. MAGNESIUM METALS CORP. (U.S.P. 1,926,587, 12.9.33. Appl., 19.2.31. Austr., 18.6.29).—An intimate mixture of wood charcoal and MgO, MgCO<sub>3</sub>, or Mg(OH)<sub>2</sub> is allowed to fall down a shaft furnace, heated at 500°, up which a current of steam is passed, whereby a mixture of H<sub>2</sub> and CO<sub>2</sub> free from CO is formed. The gas is scrubbed with H<sub>2</sub>O under pressure to remove CO<sub>2</sub>, and the MgO residue is mixed with more C for re-use. (Cf. U.S.P. 1,836,919; B., 1932, 886.) A. R. P.

Absorption etc. between gases and liquids.— See I. Acid-resisting enamel.—See VIII. Waste-H<sub>2</sub>O from S mining.—See XXIII.

## VIII.--GLASS; CERAMICS.

Attack of majolica enamel by water in relation to its chemical composition. W. KERSTAN (Sprechsaal Keram., 1933, 66, 526, 537; Chem. Zentr., 1933, ii, 3027).—Enamels prepared with cryolite are readily attacked by  $H_2O$ , and those with little alkali and a moderate  $H_3BO_3$  content are less attacked. Replacement of PbO by  $\geq 9\%$  BaO or  $\geq 18\%$  CaO improved the resistance to  $H_2O$ . ZnO, MgO, Na<sub>2</sub>O, and SiO<sub>2</sub> had a small effect. With  $B_2O_3$  the optimum resistance was obtained by replacement of 2% of the PbO.

H. J. E. Cause of adherence of base enamels free from adhering oxides on sheet iron. A. DIETZEL and K. MEURES (Sprechsaal Keram., 1933, 66, 647—652; Chem. Zentr., 1933, ii, 3027).—Adherence of the enamel occurs at a stage during the baking at which it contains magnetite crystals and another type of crystal which was not identified. Adherence also occurs when the magnetite is oxidised to hæmatite. H. J. E.

Applications of town's gas.-See II.

See also A., May, 494, Measurement of reaction rates [of quartz]. 504, Prep. of sintered Pyrex glass filters, and of microscopic glass spheres. 505, SiO<sub>2</sub>-glass from Libyan desert. 507, Separation of clay constituents.

## PATENTS.

Glass furnaces. W. E. EVANS. From O'NEILL MACHINE Co. (B.P. 408,265, 1.10.32).—A fore-hearth is separated from a melting tank by a curtain wall and is made shallow at the gathering point (P) with a bottom sloping steeply to direct any chilled glass sheared from the moulds back into the tank. Both cooling and heating devices are provided to regulate exactly the temp. at P. B. M. V.

Manufacture of glass. P. E. HARTH, Assr. to NAT. PIGMENTS & CHEM. Co. (U.S.P. 1,925,381, 5.9.33. Appl., 7.2.31).—Ba aluminate or some other compound of Ba, Al, and O is added to bottle-glass batch before fusing the mixture. B. M. V.

Manufacture of [acid-resisting] vitreous enamel composition. C. J. KINZIE and J. A. PLUNKETT, Assrs. to TITANIUM ALLOY MANUFG. Co. (U.S.P. 1,925,560, 5.9.33. Appl., 11.3.30).—TiO<sub>2</sub>, SiO<sub>2</sub>, and Na<sub>2</sub>CO<sub>3</sub> are fused together at  $800-1200^{\circ}$  to form frits of the following alternative compositions : Na<sub>2</sub>O 30.69 or 16.23, TiO<sub>2</sub> 39.60 or 20.94, SiO<sub>2</sub> 29.71 or 62.83%. B. M. V.

# IX.—BUILDING MATERIALS.

Apparatus for automatic recording of setting time of cement. N. DAVEY (J. Sci. Instr., 1934, 10, 148-150).—The penetrating needle is cleaned automatically after each contact with the cement.

C. W. G.

Bending of marble. (LORD) RAYLEIGH (Proc. Roy. Soc., 1934, A, 144, 266—279).—The rigidity of marble which has been baked at 100° is diminished; the effect becomes more marked as the baking temp. (T)is raised up to 350°. The progressive change of mechanical properties is traced as T is raised. The results are interpreted as due to the unequal expansion of calcite by heat, the expansion being a max. along the optic axis and negative in the perpendicular direction.

L. L. B.

S. C.

Effect of fatigue in insolation [of granite]. D. T. GRIGGS (Science, 1934, 79, 319).—Coarse-grained granite subjected to temp. changes corresponding with > 100 years' exposure to insolation in an arid climate showed a slight development of cracks along cleavages in the felspar. L. S. T.

Determination of methoxyl in wood. W. ENDER (Angew. Chem., 1934, 47, 257–258).—Wood is treated with 72% H<sub>2</sub>SO<sub>4</sub> and the diluted mixture distilled to remove MeOH, which is then determined in the distillate as MeO·NO (cf. A., 1934, 630).

## PATENTS.

Production of a water-repellant [building material]. P. L. MOREAU (B.P. 408,846, 25.7.33).— A semi-liquid product formed by hydrolysing mutton tallow or wool fat with excess of a 6% solution of aq. NH<sub>3</sub> in presence of an alkaline silicate is added to a mortar or cement mixture. C. A. K.

Plastic [plaster] composition. J. KEETH (U.S.P. 1,926,543, 12.9.33. Appl., 18.7.31).—A non-cracking plaster is composed of clay 6, paper pulp 2 pts., and sawdust 1 pt., manufactured wet and dried for shipment. B. M. V.

Manufacture and laying of a bituminous road material. W. J. TENNANT. From WIGANKOW & Co., NEUZEITLICHER STRASSENBAU G.M.B.H. (B.P. 408,897, 6.11.33).—Stone material coated with hard bitumen (I) is cooled to 25—30° below the m.p. of the bitumen (I) and treated with a separating substance, e.g., flue dust. At the time of laying, the material is treated with a mixture of (I) and a solvent, e.g., C<sub>6</sub>H<sub>6</sub>, to restore the binding power. C. A. K.

Production of road coverings. H. WIEDEMANN (B.P. 407,460, 16.11.32. Ger., 17.11.31).—The road surface is formed of plates of asphalt (I) or other bituminous material into the upper layers of which pieces of coke have been pressed. The coke is preferably saturated with tar under pressure before admixture with the (I). A. B. M. Asphalt-coated [sea]shell [for road surfaces]. W. R. PARKER and W. L. HOLBROOK (U.S.P. 1,926,630, 12.9.33. Appl., 14.6.32).—The shell material is freed from mud by washing and the surfaces of the clean shells are roughened, sprayed with bituminous material at about 82°, and cooled quickly in air. B. M. V.

Sound-insulation material.—See I. Al<sub>2</sub>O<sub>3</sub> from coal ash.—See VII.

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

Behaviour of spathic iron ore on roasting. W. LUYKEN and L. KRAEBER (Stahl u. Eisen, 1934, 54, 361-364).-The ore used consisted of FeCO<sub>3</sub> 75.9,  $MnCO_3$  15.7,  $SiO_2$  1,  $MgCO_3 + CaCO_3$  6%. On heating at 700-800° in absence of air and removing the gases as formed, the residue consisted of (Fe,Mn)O solid solution and (Fe,Mn)O,Fe2O3 spinel (I), the mol. ratio (R) of  $\text{Fe}_2\text{O}_3$ : FeO being 1:3; the gases evolved contained 15% CO. When the gases were kept in contact with the oxides Rfell from 1:1.7 at 600° to 1:3.5 at 900° and the  $CO: CO_2$  ratio from 1:4.9 to 1:11, *i.e.*, the O content of the residue decreased rapidly with rise in temp. In presence of regulated amounts of O2 the CO is oxidised and hence R is increased. With 1 mol. of O2 to 6 mols. of FeCO3 (i.e., just sufficient O to form Fe<sub>3</sub>O<sub>4</sub>) complete conversion of the ore into (I) was obtained at 700-800°. With larger proportions of O<sub>2</sub> (I) was slowly converted into cubic, ferromagnetic Fe<sub>2</sub>O<sub>3</sub>, which in the intermediate stages formed a solid solution with (I). After complete oxidation practically no hexagonal Fe<sub>2</sub>O<sub>3</sub> (II) could be detected by X-rays. On direct roasting of the ore with free access of air, however, much nonmagnetic (II) was produced, the proportion increasing with the time and temp. of roasting; under no practical conditions was it found possible to obtain a completely non-magnetic residue. A. R. P.

Chemical and physical properties of Thomas pig iron and their effect on steelworks practice. K. EICHEL (Stahl u. Eisen, 1934, 54, 229-236).— The effect of varying composition of the pig Fe on the course of the refining process in the Thomas converter (I) is shown by data (tables and graphs) collected over 14 months' operation of the process in a Saar steelworks. The total loss of Fe in (I) is almost a linear function of the Si content of the Fe and is especially high with high Si at relatively low temp. Addition of scrap to the charge reduces the conversion losses by diluting the Si content, but the quantity, nature, and time at which the scrap is added are dependent entirely on the character of the pig Fe.

A. R. P.

Slagging of manganese and iron in the Thomas converter. O. SCHEIBLICH (Stahl u. Eisen, 1934, 54, 337-344, 365-370).—The basicity of the slag (S) in the Thomas converter is of paramount importance in determining the Mn losses in S, the partition coeff., K = (MnO)[Fe]/[Mn](FeO), being 6 times as great when S is more acid than  $CaSi_2O_5$  as when S

541

is more basic than  $Ca_2SiO_4$ . The efficiency of the Mn in the charge depends directly on the basicity of Sand inversely on the Mn content of the metal (I). When (I) is high the loss of Mn in S is increased, the O content of the Fe at the end of the refining is higher, and the Fe losses in conversion are increased. An analysis of the costs of converting pig Fe with  $2 \cdot 5\%$  and  $1 \cdot 5\%$  Mn is given, which shows that no advantage is to be gained technically or economically from the use of a high-Mn pig Fe. A. R. P.

Determination of small amounts of zinc in steels and irons. H. A. BRIGHT (Bur. Stand. J. Res., 1934, 12, 383-389).—Pptn. of Fe<sup>...</sup> with aq. NH<sub>3</sub> is sufficiently accurate when the Zn content is 0.005-0.30%. The method involving extraction of Fe<sup>...</sup> by Et<sub>2</sub>O is the most satisfactory when about 0.001% of Zn is present. Pptn. of Zn as ZnS by H<sub>2</sub>S in presence of a citrate buffer is satisfactory when the Zn content is > 0.003%. E. S. H.

Oxidation of liquid copper melts with small contents of nickel, arsenic, tin, and antimony. P. SIEBE and C. BUSSE (Metall u. Erz, 1933, 30, 273-280; Chem. Zentr., 1933, ii, 3036).—The oxidation of Cu at 1100° by N<sub>2</sub> containing  $4 \cdot 8\%$  O<sub>2</sub>, and with additions of  $\gg 1\%$  Sn, Ni, Sb, and As, has been measured. The order of efficiency in arresting oxidation is As > Sb > Ni > Sn. The technical difficulty of removing Sn, Sb, and Ni from Cu is due to solubility of their oxides in molten Cu. Ni is largely present as free metal. As should be removed by vaporising from the melt. H. J. E.

Silver-copper alloys containing phosphorus. H. MOSER, E. RAUB, K. W. FRÖHLICH (Metallwirts., 1933, 12, 497-501; Chem. Zentr., 1933, ii, 3038-3039).—P is a very active deoxidising agent for Ag-Cu alloys. The phase diagram of the system Cu-Ag-P is discussed (cf. A., 1932, 1196). The effect of P on the mechanical properties of alloys with 83.5% and 50% Ag has been measured. Alloys containing 1% P are almost unattacked by hot AcOH. H. J. E.

Corrosion of brass in water subjected to  $p_{\rm H}$ correction. E. W. MOORE (J. New England Water Works' Assoc., 1934, 48, 47—58).—Two sets of 60:40 brass pipe were immersed in H<sub>2</sub>O, the  $p_{\rm H}$  range of which was adjusted to 6.0—11.0, for periods of 60 and 120 days respectively. The containers were closed but were shaken daily to re-aërate the H<sub>2</sub>O. Analysis of the H<sub>2</sub>O at the end of the periods showed that the dezincification of the brass appeared to decline from  $p_{\rm H}$  6.0 to a min. at  $p_{\rm H}$  10.0 and then to rise slightly to  $p_{\rm H}$  11.0. The dissolution of Cu depended on that of Zn, min. Zn dissolved coinciding with max. Cu. Protective films retarded dezincification only at  $p_{\rm H}$  7.5—9.5. C. J.

Thermal expansion of bearing bronzes. P. HIDNERT (Bur. Stand. J. Res., 1934, 12, 391-400).— The linear thermal expansion of cast bearing bronzes (Cu-Sn and Cu-Sn-Pb) has been determined between 20° and 200°. Addition of Sn to Cu or of Pb to Cu-Sn increases the expansion coeff. Equations expressing these effects have been derived. E. S. H. Sedimentometric analysis of slimes. N. LUB-MAN (Tzvet. Met., 1933, No. 1, 44-61).—Dispersion analysis (Wiegner and Odén) can be applied to highly dispersed phases in ore flotation. To obtain a true picture of the state of dispersion as a result of grinding it is necessary to stabilise the suspension. With complete stabilisation the distribution of slime particles is independent of  $\eta$  and other mol. properties of the liquid medium. The most suitable stabilisers are alizarin-red in aq. alkaline, and oleic acid in hydrocarbon, medium. CH. ABS.

Increase in the plasticity of metals by plastic rotation. V. D. KUZNETZOV, D. V. KONVISAROV, and V. I. STROKOPITOV (Compt. rend. Acad. Sci. U.R.S.S., 1934, 1, 399–402).—The plastic deformation of Al, Cu, and Fe wire has been studied. Plastic deformation is increased by rotation and by raising the temp.

Passivity of magnesium in chromic acid, and the chemical cleaning thereof after corrosion. M. CHAUSSAIN and H. FOURMIER (Compt. rend., 1934, 198, 1035—1037; cf. B., 1930, 462; 1934, 150).— Immersion of Mg in boiling aq.  $CrO_3$  produces imperfect passivity if 0.1—0.15% H<sub>2</sub>SO<sub>4</sub> is present. Accordingly, in cleaning corroded Mg with aq.  $CrO_3$ , if SO<sub>4</sub>" is suspected therein or in the corroded metal sufficient BaCrO<sub>4</sub> should be added to ppt. the SO<sub>4</sub>"; and similarly AgCrO<sub>4</sub> if Cl' is present. C. A. S.

Oxygenating [cyanide] solutions [for gold extraction]. T. K. PRENTICE (J. Chem. Met. Soc. S. Afr., 1934, 34, 244—256).—A higher extraction of Au combined with a low consumption of NaCN is obtained by increasing the O content of the liquor to 6 mg. per litre. This may be effected at very little cost by passing the pulp through a closed cylinder, where it is subjected to the action of compressed air for 1—2 min., on its way to the leaching tanks. Probably most of the beneficial action of the aëration is due to the oxidation of cyanicides and reducing agents which interfere with the Au extraction. A. R. P.

Recovery and purification of platinum metals, especially from residues. W. SAVELSBERG (Metallbörse, 1933, 23, 541-542, 573-574, 605-606, 637-638; Chem. Zentr., 1933, ii, 2886).—In working up residues, especially from Ni electrolyses, Ni and Cu must be completely removed. This is done by melting the Cu-Ni-Fe matte and roasting and lixiviating with dil.  $H_2SO_4$ . The residue from lixiviation is treated with Pb in a Pb bath or in a graphite crucible with a reducing litharge melt. Cupellation of the Pb with addition of Ag, if necessary, gives a Ag or Pt button with 40-45% Pb. L. S. T.

Platinum plating. A. E. W. SMITH (Metal Ind., 1933, 43, 201-202).—Pt plate is appreciably harder than Ni plate. Results of exposure of Pt plated on Ag-coated brass are recorded;  $H_2S$  produced greatest corrosion. In no case was the Pt attacked, corrosion being due to the presence of pinholes. Ni-, Pt-, and Cr-plated steels were tested with 2% EtOH-I; Ni was dissolved in 1 day, whereas Pt showed signs of pinholing and Cr signs of cracking at the edges in 2 days. Ptreflects 65%, Ag 92%, of visible light rays. CH. ABS.

W. R. A.

Influence of heavy metals on aluminium alloys. P. RÖNTGEN and W. KOCH (Z. Metallk., 1934, 26, 118).— Corrections to a previous paper (cf. B., 1934, 408).

Analytical control of cadmium-plating solutions. E. E. HALLS (Metallurgia, 1934, 9, 183—184).—Total Cd is determined by electrolysis at  $4 \cdot 5$ —5 volts, using a rotating, Cu-plated, Pt-gauze cathode, or by evaporation of the solution with conc. H<sub>2</sub>SO<sub>4</sub> followed by titration with aq. K<sub>4</sub>Fe(CN)<sub>6</sub>, using UO<sub>2</sub>(OAc)<sub>2</sub> as external indicator. Free KCN is determined by titration with  $0 \cdot 1N$ -AgNO<sub>3</sub> and alkalinity (NaOH +  $\frac{1}{2}$ Na<sub>2</sub>CO<sub>3</sub>) by titration with H<sub>2</sub>SO<sub>4</sub> (phenolphthalein) after pptn. of the total CN' with AgNO<sub>3</sub>. CO<sub>3</sub>" is determined by pptn. with BaCl<sub>2</sub> and acidimetric titration of the BaCO<sub>3</sub>. A. R. P.

Applications of town's gas.—See II. Enamelled sheet Fe.—See VIII. Newton  $H_2O$  and corrosion. —See XXIII.

See also A., May, 482, Systems Al-Ba, Cu-Si, Mn-Si, Ni-Zn, Ge-Cu. Alloys of Si with Cr, Mn, Co, and Ni. 483, Pr-Mg alloys. Recrystallisation of Fe alloys. Austenite-martensite transformation. System Fe-Fe<sub>3</sub>C-ZrC-Fe<sub>3</sub>Zr<sub>2</sub>. X-Rays and physical metallurgy. 486, Passivity of Fe and steel in acids. Protective films on Al in acids. 487, Colloidal Au. 496, Nb-plate, and separation of Nb from Ta. 499, Electrolytic prep. of Zn.

#### PATENTS.

(A) Apparatus for (B) method of, sintering. B. W. GONSER, ASST. to AMER. SMELTING & REFINING CO. (U.S.P. 1,926,043-4, 12.9.33. Appl., [A] 24.1.30, [B] 23.8.32. Renewed [A] 2.12.32).—In a sintering machine of the continuous conveyor type, the main charge is deposited with a backwardly inclined slope on which additional fuel is deposited at an adjustable point, usually near the toe. The process is especially suitable for ores containing Zn. B. M. V.

**Open-hearth furnace.** S. NAISMITH (U.S.P. 1,925,494, 5.9.33. Appl., 7.3.30).—In an open-hearth reversing furnace (F) operated with blast-furnace gas, the heated gas from the regenerator (R) enters F through a port smaller than the air port, but the passage from F to the gas R is larger than that to the air R, the adjustment being effected preferably by opening and closing supplemental passages. B. M. V.

[Open-hearth] furnaces. K. M. SIMPSON, Assr. to INTERNAT. CHROMIUM PROCESS CORP. (U.S.P. 1,925,941— 2, 5.9.33. Appl., 29.6.31).—(A) Besides the normal end-ports, the furnace is provided with burners projecting downwardly through the roof, the air for which is preheated in a regenerator adjacent the normal ones, all the combustion gases leaving through the endports. (B) In a roof burner, the fuel nozzle is arranged to serve also as a valve to regulate the air supply.

## B. M. V.

Cupola. W. P. BREWER and J. T. JONES, ASSIS. to TENNESSEE PRODUCTS CORP. (U.S.P. 1,925,519, 5.9.33. Appl., 29.6.32).—The cupola is provided with a  $H_2O$ jacket and, in the neighbourhood of the tuyères, with heat-conducting blocks extending right through the refractory lining. B. M. V. Purification of ferrous metals. C. T. HENNIG (U.S.P. 1,925,247, 5.9.33. Appl., 12.8.31).—A composition (mainly  $Na_2CO_3$ ), as described in U.S.P. 1,826,881 (B., 1932, 682), is added to molten pig Fe prior to treatment in the converter. B. M. V.

Differential graphitisation of cast[-iron] articles. H. L. SPENCE and H. A. SCHWARTZ, ASSTS. to NAT. MALLEABLE & STEEL CASTINGS Co. (U.S.P. 1,925,116, 5.9.33. Appl., 15.5.29).—Articles, e.g., valve tappets, of white cast Fe, which it is desired to render partly hard and partly soft, are, after casting, allowed to cool below the A1 point, and inserted to the necessary degree through a refractory, heat-insulating plate; the parts to be softened are heated to well above A1, quenched, annealed at slightly above A1, and cooled slowly. The latter treatment may be repeated at rather below A1 if desired. B. M. V.

Manufacture of surface-hardened cast-iron articles. M. L. A. BABINET, ASST. to NITRICASTIRON CORP. (U.S.P. 1,924,344, 29.8.33. Appl., 13.6.31).—Claim is made for grey cast Fe containing C 2—4 (3.2), Al  $\gg$ 1585 (0.6), Si 2—4.5 (4.1), Cr 1.5—3.5 (2.1), and Ti and/or V  $\gg$  0.5% and case-hardened in NH<sub>3</sub>.

A. R. P.

Manufacture of chrome and similar steels. K. M. SIMPSON (U.S.P. 1,925,940, 5.9.33. Appl., 16.4.30).— The steel is made in an open-hearth furnace, heated top and bottom, the latter by combustion in independently controlled flues under the hearth ; the top of the steel is heated to 1600° and the bottom 55—82° lower.

B. M. V.

Manufacture of stainless steel. A. F. PLOCK (U.S.P. 1,924,804, 29.8.33. Appl., 29.12.31).—Fe is refined in an electric furnace under a CaO-Fe<sub>2</sub>O<sub>3</sub> slag until the C is reduced to < 0.1%, the slag is removed and replaced by a 4 : 1 mixture of CaO and CaF<sub>2</sub>, and a 350 : 60 : 500 : 150 mixture of CaO, CaF<sub>2</sub>, chromite (50% Cr<sub>2</sub>O<sub>3</sub>), and 56 : 27 : 17 Si-Cr-Fe alloy is added slowly to the slag so that the Cr is gradually reduced into the Fe.

A. R. P.

[Alloy for making] heat-treating apparatus [for nitrogenisation of steel]. J. W. HARSCH, ASST. to LEEDS & NORTHRUP CO. (U.S.P. 1,926,234, 12.9.33. Appl., 7.9.32).—The parts subjected to the action of nascent N are made of an alloy containing 20—90% Ni and 10—40% Cr, *e.g.*, an alloy of Ni 60, Cr 33, and Fe 6% free from Mn. A. R. P.

Coating metal [steel] with zinc phosphate. V. M. DARSEY, ASST. to METAL FINISHING RES. CORP. (U.S.P. 1,926,265, 12.9.33. Appl., 8.12.30. Renewed 10.7.33).— The steel is dipped in aq.  $Zn(HPO_4)_2$  containing 3% of  $Zn(CN)_2$  at 75°. A. R. P.

Corrosion-inhibiting compound. A. A. OEDING (U.S.P. 1,925,672, 5.9.33. Appl., 25.9.31).—A composition for addition to the cooling  $H_2O$  of automobiles etc. comprises yellow Na<sub>2</sub>CrO<sub>4</sub> 20, paraffin oil 15, sulphonated red oil 50, liquid soap 2, liquid bark extract 5,  $H_2O$  to 100 wt.-%. B. M. V.

Treatment of metalliferous material. [Production of coated zinc oxide agglomerates.] E. H. BUNCE and C. J. LENTZ, Assrs. to NEW JERSEY ZINC Co. (U.S.P. 1,926,032, 12.9.33. Appl., 29.4.31).—Briquettes

of coal and ZnO (roasted ZnS) are fed on to a chainconveyor belt (I) passing through a horizontal coking furnace (II), (I) being covered with fines and then with a bed of coarser material from a previous coking operation. Coking is effected by means of hot non-oxidising gases passed through a series of flues in the top of (II) in such a way that the temp. of the gases progressively rises from 300° at the feed end to 900° at the discharge end. A. R. P.

Welding composition [for bearing metals]. F. L. THOMPSON (U.S.P. 1,926,412, 12.9.33. Appl., 12.7.32).— A welding rod of Zn 90, Cu 5, and Sb 5% is claimed. A. R. P.

Welding-rod manufacture. R. E. FRICKEY and A. W. MCCLARY, Assrs. to WELDING SERVICE, INC. (U.S.P. 1,926,090, 12.9.33. Appl., 13.8.29).—For the deposition of, e.g., Ni steel by the electric arc, the rod is coated with a mixture of Fe–Cr, Mn–Ti, CaMoO<sub>4</sub>, fireclay, and aq. gum arabic, the proportions being : Cr 3.37, Mn 1.52, Ti 0.49, Mo 0.12% of the total rod. The above coating after application is allowed to dry and then protected with a mixture of Na silicate, SiO<sub>2</sub>, talc, and H<sub>2</sub>O. B. M. V.

Lead cable sheath containing lithium. E. KOCH, Assr. to MAYWOOD CHEM. WORKS (U.S.P. 1,926,545, 12.9.33. Appl., 19.4.33. Ger., 1.11.28).—The Pb contains Li 0.002-0.010% and, if desired, Cd 0.15-0.18and/or Sb  $\gg 0.5\%$ . B. M. V.

[Nickel-gold alloy] resistance wire. J. PICCARD, Assr. to HERCULES POWDER Co. (U.S.P. 1,926,213, 12.9.33. Appl., 17.1.30).—Claim is made for an alloy of Au 58.4 and Ni 41.6% drawn to wire having a resistance of 360 ohms per yard, suitable as bridge wire for blasting caps. A. R. P.

Casting method and apparatus [for tungsten carbide]. D. WILLCOX, ASST. to AJAX ELECTROTHERMIC CORP. (U.S.P. 1,926,573, 12.9.33. Appl., 24.12.31).— The apparatus comprises a heat-insulated, high-frequency induction furnace containing a melting crucible (C) above which is a graphite mould (M); both C and M can be heated by separate, H<sub>2</sub>O-cooled induction coils and both are packed in C powder. Means are provided for inverting the whole apparatus. A. R. P.

[Aluminium] solder. (A-c) C. C. CALLIS, (A, B) R. B. DERR, and (c) E. J. KRATZ, Assrs. to ALUMINUM Co. of AMERICA (U.S.P. 1,926,853-5, 12.9.33. Appl., 30.7.32).—An alloy for soldering Al and its alloys comprises (A) Sn 66—69, Zn 27.5—28.5, and Al 2.5—6.5%, (B) Sn 47.5—49, Zn 47.5—49, and Al 2.5—6.5%, or (c) Sn 37—45 (44), Pb 37—45 (44), Zn 9—21 (10), and Al 1—5 (2) %. A. R. P.

Working of aluminium-magnesium alloy. J. A. NOCK and E. H. DIX, JUN., Assrs. to ALUMINUM CO. OF AMERICA (U.S.P. 1,926,057, 12.9.33. Appl., 14.1.31).— Cast Al alloys with 5—15% Mg are soaked at  $> 290^{\circ}$ (430°) for a prolonged period (*e.g.*, 20 hr.), then allowed to cool to  $< 310^{\circ}$ , and worked by hammering or rolling at 260—310°. A. R. P.

Heating material [metal sheets].—See I. Compounds from Cr alloys.—See VII. Ta-Ni alloy and vac.-tube electrodes.—See XI. Metal-pickling baths.—See XX.

# XI.—ELECTROTECHNICS.

Measurement of adsorbed moisture films on non-conductors. W. M. THORNTON (J. Inst. Electr. Eng., 1934, 74, 448-452).-New electrical methods of determining the presence of adsorbed films of moisture on insulators are described. An ellipsoid (E) hygrometer is described, consisting of a quartz E suspended between two plates, to which an alternating field is applied, in a closed vessel. The E moves into the direction of the field under the action of two forces, viz., (a) that due to the true dielectric const. of the quartz, and (b) that due to the conducting film. If (a) is known, (b) can be calc. and the extent of the adsorption obtained. The resistivity of a film of moisture on glazed porcelain was approx. 10<sup>9</sup> ohms per sq. cm. An ionic wind hygrometer is also described. A. J. M.

Recovery etc. of Pt metals. Pt- and Cd-plate.— See X. Measuring  $p_{\rm H}$ .—See XV. Determining replaceable bases in soils. Electrodialysis and soil phosphates.—See XVI. Sugar-boiling control.— See XVII.

See also A., May, 496, Nb-plate. Electro-org. oxidations of PhCHO,  $CH_2Ph\cdot OH$ , etc. 499, Electrolytic prep. of Zn. 506, Separation of clay constituents. 516, Prep. of semicarbazide hydrochloride. Electrolysis of  $Et_2O$  solutions of the Grignard compound. 520, Prep. of 3:3'-diamino- and -dihydroxy-azobenzene and analogous compounds.

## PATENTS.

Induction furnace. T. H. LONG, ASST. to WESTING-HOUSE ELECTRIC & MANUFG. Co. (U.S.P. 1,926,811, 12.9.33. Appl., 2.4.31).—A system of induction coils and condensers is described, whereby a multiphase source of power may energise a single-phase heater coil. B. M. V.

Manufacture of electrical rectifiers. D. G. ACKERLY, ASST. to UNION SWITCH & SIGNAL CO. (U.S.P. 1,926,132, 12.9.33. Appl., 11.9.29).—Cu sheet is heated at 1000° in  $O_2$  until it is coated with  $Cu_2O$  and an outer film of CuO. The latter is then reduced to Cu by cathodic reduction in a fused salt bath containing CaCl<sub>2</sub> 66, KCl 6, and NaCl 28% (m.p. 504°), using an Fe anode. A. R. P.

[Tantalum-nickel alloy] electrode for electrondischarge devices. E. A. GIARD, ASST. to CUTLER-HAMMER, INC. (U.S.P. 1,926,846, 12.9.33. Appl., 15.8.31).—A grid for radio valves comprises an alloy of Ni with  $\geq 30$  (20)% Ta which is oxidised superficially by heating in air and then heated in vac. to reduce or volatilise the NiO, leaving a firmly-adherent coating of Ta<sub>2</sub>O<sub>5</sub>. A. R. P.

(A) Electrode elements of vacuum tubes. (B) Cleaning and regenerating compound for electronic tubes. H. J. MILLER (U.S.P. 1,925,075-6, 29.8.33. Appl., [A] 9.6.30, [B] 4.8.30).--(A) The anode consists of an alloy of Ni with 0.5--7% Si the surface of which has been coated with NiSiO<sub>3</sub> by oxidation in the air. (B) A getter for radio valves comprises an alloy of Ba 3-12 (4·3), Mg 6-20 (7·4), and Sn 91-78%.

A. R. P.

Electrode [for neon and mercury-vapour tubes] and its manufacture. F. L. HUNTER, JUN., Assr. to FANSTEEL PRODUCTS CO., INC. (U.S.P. 1,926,336, 12.9.33. Appl., 13.9.30).—The electrode comprises a compressed cylinder of an intimate mixture of TaC (88) and CsCl, RbCl, and LiCl (12%). A. R. P.

Indicating the level and sp. gr. of liquids etc.— See I. Pb cable sheath. Ni-Au wire. Casting WC. —See X.

# XII.—FATS; OILS; WAXES.

Saponification and saponification value. T. RUEMELE (Seifensieder-Ztg., 1933, 60, 700-701; Chem. Zentr., 1933, ii, 2914).—With EtOH-KOH, alcoholysis or hydrolysis precedes saponification. Grossfeld and Mechlinski's method for determining sap. val. is recommended. A. A. E.

Fat, oil, or fatty acid determination by melting out with wax or ceresine. C. A. ROJAHN and J. HAUGG (Apoth. Ztg., 1933, 48, 1117—1120; Chem. Zentr., 1933, ii, 3021).—Fatty acids in soap are liberated with mineral acids and extracted by melting with 3—4 g. of wax (I), ceresine (II), or a mixture of (I) and (II). The fatty acids are determined by weighing the cake which separates on cooling. The method is also applied to emulsions, liniments, ointments, etc. H. J. E.

Tung oil fruits from Paraguay. B. REICHERT (Pharm. Zentr., 1934, 57, 282).—Physical characteristics of the fruit and chemical consts. of the oil are given.

G. H. C. Gelation of Aleurites oil (China-wood oil) by antimony halides. (MLLE.) M. T. FRANÇOIS (Compt. rend., 1934, 198, 1046—1048).—Tung and Abrasin oils can be detected by their gelation (G) in a 10% solution of SbCl<sub>3</sub> in CHCl<sub>3</sub> (not SbBr<sub>3</sub> or SbI<sub>3</sub>) and can be determined within 10%, if adulterated, by the time taken for G to take place under standard conditions. R. S. C.

Bulking vals. of oils.—See I. Tung oil groves.— See XVI. Butter fat.—See XIX.

See also A., May, 510, Hydrogenation of ricinoleic acid. 539, Silk sericin. 568, Vitamins in fish-liver oils.

#### PATENTS.

Treatment of tung oil. W. B. STODDARD, T. H. GEIGER, and L. M. BURGESS, ASSTS. to TUNG OIL PRODUCTS, INC. (U.S.P. 1,924,524, 29.8.33. Appl., 10.3.31).— Tung oil is bodied at about 125° in presence of, e.g., 0.3% of ZnCl<sub>2</sub> (dissolved in COMe<sub>2</sub>); addition of small amounts (e.g., 5%) of unsaturated (linseed oil) fatty acids and salicylic acid (e.g., 1%) is advantageous. Polymerisation with ZnCl<sub>2</sub> and COMe<sub>2</sub> peroxide is claimed. E. L.

Packaging of edible [cod-liver] oils. W. G. CHRISTIANSEN and F. R. CHAPPELL, ASSIS. to E. R. SQUIBB & SONS (U.S.P. 1,924,708, 29.8.33. Appl., 17.1.30).—The oil is packed in opaque containers (or containers coated with a film containing an opaque substance, e.g., Congo-red), of which the interior is coated with an oil-insol. film containing an antioxidant e.g., quinol,  $p-\mathrm{NH}_2\cdot\mathrm{C}_6\mathrm{H}_4\cdot\mathrm{OH}$ . E. L.

Vac. distillation.—See I. Fatty acid condensation products. Wax-like esters.—See III. Fish-liver oil.—See XX.

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

Making paint exposures in south-eastern Florida. C. H. DAVIS and B. F. DAVIS (Sci. Sect. Nat. Paint, Varnish & Lacquer Assoc., Inc., Apr., 1934, Circ. No. 457, 84—96).—Refinements in technique are described. Racks to turn away from the sun during examination facilitate detection of failures. Marine exposures must be made well away from harbours on account of grease and sewage affecting the vegetation. The methods of preparing, labelling, and exposing panels in the particularly destructive conditions of Florida are given.

G. H. C.

[Riveted] panels for antifouling paint tests. H. A. GARDNER (Sci. Sect. Nat. Paint, Varnish & Lacquer Assoc., Inc., Apr., 1934, Circ. No. 454, 64—65).

G. H. C.

Attack of linseed oil paints by moulds. I, II. K. WEISE (Farben-Ztg., 1934, 39, 412-413, 444-445). --I. A linseed oil film provides a good medium for the growth of *Aspergillus*, *Penicillium*, etc. The presence of mucilage, by providing N, P, S, and metals, assists the development of moulds; the same result may be obtained artificially by adding suitable salts. Stand oil films are somewhat more resistant to moulds.

II. Most pigments (including Pb pigments, and inert Zn pigments such as lithopone) have little effect on the growth of mould on linseed oil films. ZnO and pigments containing Cu and Hg have a definite inhibiting action, ZnO being the most satisfactory, since it is the least poisonous to man. C black and other pigments (e.g., blanc fixe) containing adsorbed K or P compounds favour mould formation by providing nutriment for the moulds. G. H. C.

Mildew prevention on painted surfaces. III. H. A. GARDNER, L. P. HART, and G. G. SWARD (Sci. Sect. Nat. Paint, Varnish & Lacquer Assoc., Inc., May, 1934, Circ. No. 464, 135—137; cf. B., 1934, 209).— Particulars of further tests recently commenced are given. G. H. C.

Opaque and transparent colour lakes. G. ZERR (Farben-Ztg., 1934, 39, 442-443).—The manufacture and uses of the two types of lakes are described. The same dye may be obtained in either form by varying the substrate or the medium. G. H. C.

Oil derivatives as plasticisers for cellulose nitrate. A. W. VAN HEUCKEROTH (Sci. Sect. Nat. Paint, Varnish & Lacquer Assoc., Inc., Apr., 1934, Circ. No. 459, 101-103).—Et and Bu ricinoleates, Et acetylricinoleate, and acetylated castor oil showed little solvent action on nitrocellulose (I) though they imparted flexibility to the film. A special linseed oil exerted marked solvent action on (I) which had been wetted with PhMe. G. H. C.

Resins and plasticisers for cellulose acetate lacquers. A. W. VAN HEUCKEROTH (Sci. Sect. Nat. Paint, Varnish & Lacquer Assoc., Inc., Apr., 1934, Circ. No. 458, 97—100).—Very few natural or synthetic resins are compatible with cellulose acetate (I), which gives poor adhesion when used alone. The Santolites, some Rezyl balsams, and some Plasto resins give fairly satisfactory films with (I), particularly in presence of "phthalyl Et ethylglycolate" as plasticiser. G. H. C.

Lacquer development during 100 years. D. R. WIGGAM and W. E. GLOOR (Ind. Eng. Chem., 1934, 26, 551-553).—A brief review.

Bulking vals. of resins. Colour comparator.— See I. Hydrogenated naphthas.—See II.

See also A., May, 487, Colour lakes.

## PATENTS.

Manufacture of water paints, distempers, colour washes, and the like. W. J. TENNANT. From SINGA-PORE RUBBER WORKS, LTD. (B.P. 408,930, 11.7.32).— Better adhesion on plaster surfaces is obtained with  $H_2O$ -paints containing natural latex etc. by incorporating a silicofluoride (of Al, Mg, or Zn), Na<sub>2</sub>SiO<sub>3</sub>, or other permeating constituent. Vulcanisation of the rubber content may be effected by adding a vulcanising agent to the paint or by exposing the dried film to hot air, steam, or the sun. [Stat. ref.] S. M.

(A) Preparation of printing inks. (B) Dehydration of resins. W. J. TENNANT. From BAKELITE CORP. (B.P. 408,688—9, 15.10.32).—(A) The drying time is considerably reduced by incorporating a phenolic resin, e.g., a PhOH-CH<sub>2</sub>O product, with the linseed oil during the heat treatment. For rotary photogravure work a resin obtained by heating a phenol with a fatty oil and condensing with  $(CH_2)_6 N_4$  etc. is dispersed in tetrahydronaphthalene. (B) Heat-sensitive resins are dried without undergoing hardening by spraying the aq. mixture into a stream of air or other gas at 250— 300°; the dried particles are directed either into a cooled zone or upon a cooled metal surface. S. M.

[Phenol-aldehyde] air-drying [coating] composition. H. L. BENDER, Assr. to BAKELITE CORP. (U.S.P. 1,922,272, 15.8.33. Appl., 8.10.32).—The hardening by heat-treatment of phenol-aldehyde and other convertible resins is obviated thus: the initial reaction product is dehydrated by heat until it approaches the insol. stage; the reaction is checked by stirring in a cold solvent having b.p.  $> 100^{\circ}$ , e.g., BuOH, and the heating continued; low-b.p. solvents, e.g., COMe<sub>2</sub>, are then added. The solvents may be added initially with the resin ingredients. S. M.

Pyroxylin composition containing derivatives of ethylene glycol. R. H. VAN SCHAACK, JUN., and R. CALVERT, ASSTS. to VAN SCHAACK BROS. CHEM. WORKS, INC. (U.S.P. 1,922,646, 15.8.33. Appl., 29.10.26).— Compositions containing ethylene glycol phthalates or mixed esters of bis- $\beta$ -hydroxyethyl phthalate with org. acids  $< C_6 [o-C_6H_4(CO\cdot CH_2\cdot CH_2\cdot OAc)_2]$  as plasticisers are claimed. H. A. P.

Coating process and [nitrocellulose] material. H. V. DUNHAM, ASST. to CASEIN MANUFG. CO. OF AMERICA, INC. (U.S.P. 1,922,936, 15.8.33. Appl., 21.3.29).—An undercoat suitable for the application of pyroxylin lacquer (I) to wood, leather, etc. consists of an aq. glue or alkaline casein solution in which a small proportion of (I) has been dispersed in order to obtain greater adhesion of the finishing coat. S. M.

(A) Nitrocellulose coating composition containing low-boiling solvent vehicles. (B) Lacquer composition made from low-boiling solvents. (A) C. ELLIS, (B) T. F. BRADLEY, ASSTS. to ELLIS-FOSTER Co. (U.S.P. 1,923,714—5, 22.8.33. Appl., [A] 19.6.26, [B] 11.7.28).—(A, B) Pptn. of nitrocellulose (I) during evaporation of very volatile solvents and consequent "blushing" of the film are prevented by incorporating  $\leq 100\%$ [calc. on wt. of (I)] of a glyptal resin which, preferably, has been modified by heating with a fatty acid or with linseed or cottonseed oil acids, or (A) a monobasic aromatic acid, e.g., BZOH. (B) Balsams may be similarly used (cf. U.S.P. 1,824,757 and 1,863,264; B., 1932, 687; 1933, 478). S. M.

Viscosity-reduced [nitrocellulose] coating composition. T. F. BRADLEY, Assr. to ELLIS-FOSTER Co. (U.S.P. 1,923,704, 22.8.33. Appl., 6.5.27).—A mixture of nitrocellulose (I) and a glyptal resin with which has been incorporated cottonseed or castor oil fatty acids is dispersed in a mixture consisting of the solvent for (I), a hydrocarbon diluent (toluol), and  $Bu^{\beta}OH$  or  $Pr^{\beta}OH$ . S. M.

Non-gelling lacquers. (A, B) C. BOGIN and V. KELLY, (c) V. KELLY and C. BOGIN, Assrs. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,923,702—3, 1,923,732, 22.8.33. Appl., 29.7.31).—The marked livering tendency of nitrocellulose lacquers containing an aliphatic ester of lactic acid and a basic pigment is prevented by addition of 0.1-1.5% of (A) tartaric acid or salt, (B) malic acid or salt, (c) boric acid, salt, or ester. (Cf. B., 1931, 984.) S. M.

Mounting of decorations. T. S. REESE (U.S.P. 1,924,961, 29.8.33. Appl., 2.6.32).—A lacquer or varnish containing a cellulose ester, resin, and/or drying oil together with one or more solvents having b.p. 150—225° is used in a tacky condition as the binding medium for transferring a decoration to a metal, wood, or other base ; the ensemble is dried by baking. S. M.

Manufacture of organic cellulose ester moulding powders. KODAK, LTD., Assees. of D. E. NORTHRUP and A. W. CRANE (B.P. 408,952, 18.7.32. U.S., 17.7.31. Cf. B.P. 285,829; B., 1929, 167).—A solution of > 1 pt. by wt. of a cellulose ester in 3 pts. of COMe<sub>2</sub> or other org. solvent, which may also contain a plasticiser, is pptd. by addition of an org. non-solvent, *e.g.*, PhMe (I). Less (I) is required if some H<sub>2</sub>O is added first. The lower is the temp. of pptn. the more finely dispersed is the ppt. S. M.

Moulding composition and a resinous condensation product resulting from its conversion by heat and pressure. E. C. SHERRARD and E. BEGLINGER. Dedicated to U.S.A. (U.S.P. 1,923,756, 22.8.33. Appl., 22.8.31).—Finely-ground wood dust which, preferably, has been extracted with  $\text{Et}_2\text{O}$ ,  $C_6\text{H}_6$ , etc. is hot-moulded with PhOH and a catalyst, *e.g.*, HCl. S. M.

Resinous complex or composite of the ureaaldehyde condensation type and their solutions. B. N. LOUGOVOY, ASST. to ELLIS-FOSTER Co. (U.S.P.

1,922,690, 15.8.33. Appl., 23.8.30).—The use is claimed of a urea-aldehyde type of condensation product together with nitrocellulose and an org. solvent for varnishes, moulded substances, etc. S. M.

Preparation of [phenol-carbohydrate] resinous products. J. V. MEIGS, ASST. to PLASTIX CORP. (U.S.P. 1,923,321, 22.8.33. Appl., 15.1.27).—Fusible resins obtained by the process of U.S.P. 1,868,215 (B., 1933, 596) are rendered  $H_2O$ -resistant by heating to 240—270°, with or without a high-b.p. solvent, e.g., anthracene oil, until the mass becomes resistant to aq.  $NH_3$ , and then incorporating  $(CH_2)_6N_4$  or other hardening agent. S. M.

Production of [modified alkyd] synthetic resin. C. G. MOORE and E. H. DRAKE, ASSTS. to GLIDDEN CO. (U.S.P. 1,922,743, 15.8.33. Appl., 1.8.31).—A polybasic acid (phthalic anhydride), a polyhydric alcohol (glycerol), rubber-seed oil, and rubber-seed fatty acids are heated together until a tough, non-tacky resin is formed. S. M.

Manufacture of resin-like products from polybasic acids and polyhydroxy-compounds. R. BURNS, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 408,597, 6.10.32. Cf. B.P. 406,738; B., 1934, 413).—Hexahydroxycyclohexane or its Me<sub>1</sub> or Me<sub>2</sub> ether, e.g., quebrachitol, is heated with phthalic anhydride or other polybasic acid until the mass is homogeneous. The product may be modified by incorporation of drying oils or their fatty acids. S. M.

Artificial resinous compositions from polyhydric alcohols and polybasic acids. IMPERIAL CHEM. INDUS-TRIES, LTD., and H. A. HAMPTON (B.P. 408,667, 12.10.32). —In the prep. of alkyd resins modified with linseed oil fatty acids (I), very pale products are obtained if the (I) are redistilled under low pressure and only the more volatile portion is used. S. M.

Polymerisation of coumarone. G. K. ANDERSON, Assr. to NEVILLE Co. (U.S.P. 1,922,342, 15.8.33. Appl., 13.2.32).—Crude solvent naphtha (I) is considerably diluted with a petroleum or coal-tar distillate and heated in presence of  $H_2SO_4$  or other catalyst in a  $H_2O$ -cooled kettle so that the temp. rise is small and gradual. When cool, more (I) is added in batches and the polymerisation successively repeated. The resin recovered is useful for varnishes. S. M.

Production of esters of resin acids. A. C. JOHNSTON, Assr. to HERCULES POWDER Co. (U.S.P. 1,924,934, 29.8.33. Appl., 25.4.30).—Alkyl esters of abietic and other resin acids, *e.g.*, amyl abietate, are prepared by autoclaving the acid with an anhyd. monohydric alcohol ( $< C_6$ ) at 260—360°. S. M.

Production [in a press] of moulded articles from powdered or plastic materials. M. M. EATON (B.P. 409,123, 20.3.33).

Roll mill for paint.—See I. Fatty acid condensation products. Uses of octyl esters. Organo-Pb compounds.—See III. Improving cellulose derivatives. Turpentine from sulphite-cellulose digestion.—See V. Inulin nitrate [for lacquers]. —See XXII.

# XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Variations of smoked sheet rubber. J. YOUNG and E. H. RUCH (Trans. Inst. Rubber Ind., 1934, 9, 327—336).—In a lot of smoked sheet rubber the colour ranged from pale amber to dark brown. The pale sheets offered more resistance to milling and mixing than the dark rubber and, after compounding, the stock was less plastic and gave troubles in the extruding operation. D. F. T.

Solution properties of dried rubber. H. P. and W. H. STEVENS (Trans. Inst. Rubber Ind., 1934, 9, 367—368). Reply by T. H. MESSENGER (*Ibid.*, 368; cf. B., 1934, 212).—In a criticism of Messenger's paper it is suggested that the alteration in the solution  $\eta$  of rubber on extreme drying may be due to alteration in the hydrophilic protein rather than to mol. changes in the caoutchouc hydrocarbon. D. F. T.

Apparatus for extraction of samples of rubber and other materials with solvents. H. J. STERN and W. PUFFETT (Trans. Inst. Rubber Ind., 1934, 9, 363—366).—For continuous extractions, e.g., with COMe<sub>2</sub>, a modification of the A.S.T.M. type of apparatus is described. A small, siphon-fitted cup is suspended inside a small, wide-necked flask containing the solvent, a thimble-shaped condenser being supported in the flask neck. The flask rests on a perforated cylinder, inside which an electric C-filament lamp provides the necessary heat. D. F. T.

Organic [vulcanisation] accelerators as an aid to quality. M. JONES (Trans. Inst. Rubber Ind., 1934, 9, 337-350).-The two most widely used accelerators are mercaptobenzthiazole (I) and diphenylguanidine (II). (I) gives rather low tensile strength and low reinforcement, but permits the incorporation of high % of C black without undue hardness and gives a flat vulcanisation curve ; its products also exhibit good ageing and resistance to abrasion. (II) is slower but gives stronger resilient rubbers with poorer ageing qualities. Mixtures of accelerators often give results better than those expected from their individual influence: useful combinations comprise (I) and an aldehyde-amine, a thiuram, or (II). With (I) + (II) the ageing properties approach more nearly those of (I) and a tendency to pre-vulcanisation can be suppressed by using the (II) in the form of an acid salt. Using a thiuram disulphide as vulcanising agent without free S products can be obtained with flat curing properties, no risk of over-vulcanisation, and very good ageing D. F. T. qualities.

[Rubber-vulcanisation] accelerators derived from piperidine. M. W. PHILFOTT (Trans. Inst. Rubber Ind., 1934, 9, 351—362).—The vulcanisation properties of the additive compound of  $CS_2$  and piperidine (I) (piperidine pentamethylenedithiocarbamate) can be modified by replacing the (I) basic radical by a metal or by an acidic group, or by conversion into the corresponding thiuram sulphides. The characteristic features of the Zn, Cd, Pb, and Na pentamethylenedithiocarbamates are indicated and also those of the dipentamethylenethiuram disulphide, monosulphide, and tetrasulphide. These substances and also (I) itself are effective secondary activators for use with mercaptobenzthiazole. (I) is a

Chamical Abstracts-2.

548

relatively feeble accelerator, but is activated remarkably by a small proportion of the thiuram disulphide. For such activation by secondary accelerators the presence of free S appears to be essential, vulcanisation by Se or by a thiuram disulphide without free S showing no such advantage. D. F. T.

## PATENTS.

Rubber product and its manufacture. G. OENS-LAGER, Assr. to B. F. GOODRICH CO. (U.S.P. 1,925,879, 5.9.33. Appl., 9.10.30).—Rubber is treated with SO<sub>2</sub> in presence of O<sub>2</sub> or air as a catalyst. The product containing up to 10% of combined SO<sub>2</sub> resembles soft vulcanised rubber, at 20% of combined SO<sub>2</sub> resembles pure gutta-percha, whilst at 32% it is hard and horny. D. F. T.

Manufacture of articles from rubber dispersions. E. B. NEWTON, ASST. to AMER. ANODE, INC. (U.S.P. 1,924,214, 29.8.33. Appl., 30.12.31).—The mould on which deposition is to occur is previously coated with a H<sub>2</sub>O-insol. material which will act as a separating and venting layer; this material desirably consists of plateor needle-like particles, *e.g.*, of mica, fossil flour, or soapstone, and may be applied as a suspension in a volatile liquid such as EtOH. D. F. T.

Manufacture of goods of rubber or similar material [from aqueous dispersions]. DUNLOP RUBBER CO., LTD., ANODE RUBBER CO., LTD., and E. W. MADGE (B.P. 409,421, 12.11.32).—Microporous articles of vulcanised rubber are made by the wet vulcanisation of aq. dispersions containing 20—60% S (rubber 100), the only agent employed for the purpose of gelling by heat being  $\leq 2\%$  of a metallic oxide, particularly ZnO, MgO, or CaO, or a mixture of these. D. F. T.

Vulcanisation of rubber. G. H. STEVENS (U.S.P. 1,925,707, 5.9.33. Appl., 29.2.28).—An accelerator is used comprising a solid solution of a mono- and trisubstituted guanidine, the substituent groups in each including an aryl radical  $> C_6$ , e.g., monotolyl- and s-tritolyl-guanidine. D. F. T.

Rubber-impregnated fibres.-See VI.

# XV.-LEATHER; GLUE.

Comparison of the deliming action of hydrochloric, acetic, and boric acids [on pelts]. S. W. CALVER (J. Soc. Leather Trades' Chem., 1934, 18, 266— 275).—The amount of  $H_3BO_3$  required to reduce the  $p_{\rm H}$  val. of limed pelt to 9 was six times the amount of HCl or AcOH required. Only slightly more AcOH than HCl was required to reduce limed pelt to  $p_{\rm H}$  6.0. The relative costs to reduce limed pelt to  $p_{\rm H}$  9.5 were : HCl:AcOH: $H_3BO_3 = 1:6\cdot6:6\cdot0$ , and the ash contents of the pelt were respectively reduced by 81, 74, and 39%. The ash content was further reduced by washing after deliming. D. W.

Bacteriological studies on the red discoloration of salted hides. A. G. LOCHHEAD (Canad. J. Res., 1934, 10, 275-286).—Red halophilic sarcinæ have been isolated from the discoloration of Argentine hide. From Canadian hides, organisms similar to Serratia salinaria and S. cutirubra, n. sp., have been obtained, both of which owing to proteolytic action are considered to be capable of greater damage than the red sarcinal types. F. R. S.

Changes in the colour measurement of tannin infusions at analytical strength as a function of time. G. H. W. HUMPHREYS (J. Soc. Leather Trades' Chem., 1933, 17, 685-688).—The colour of solutions of pyrogallol tans was unaffected by exposure to air and light for 24 hr., but there was appreciable darkening of solutions of pyrocatechol tans even after 2 hrs.' exposure. Readings should be taken for a tannin analysis report > 75 min. after the prep. of the infusion. D. W.

Measurement of  $p_{\rm H}$  [in the leather trade]. F. C. THOMPSON (J. Soc. Leather Trades' Chem., 1933, 17, 680-684).-The H<sub>2</sub> electrode is poisoned by As, sulphides, etc.; it cannot be used where reducible substances, e.g., chromates, are present, and the passage of the H<sub>2</sub> through the solution causes loss of volatile The glass electrode requires repeated standardacids. isation, is affected by high alkalinities, and is unreliable in presence of salts. The quinhydrone electrode can be used for buffer mixtures of  $p_{\rm H} < 8.0$  and for onebath Cr liquors, deliming liquors, and pickling solutions only. Colorimetric methods are restricted to solutions of moderate acidity or alkalinity and are liable to errors due to combination of the indicator with proteins or tannins or to the presence of neutral salts. A table of recommendations is given, from which it is shown that there is no suitable method for sulphited extracts or for feebly buffered solutions, e.g., H<sub>2</sub>O-sol. matter from leather. D. W.

Apparatus for drying leather in the laboratory. T. F. G. HEPBURN (J. Soc. Leather Trades' Chem., 1933, 17, 688—691).—Constructional details are given of a conditioning chamber with automatic temp. and R.H. control, by means of which leather samples may be dried out under standardised conditions for comparative test purposes. D. W.

Determination of sulphuric acid in leather. I. K. BAKHTIYAROV (Ovlad. Tekh. Kozh. Proiz., 1931, No. 1, 33—34).—The Balland-Maljean (I) and Procter-Searle (II) methods give accurate results for free  $H_2SO_4$  added to hide powder, but for powder treated with sulphates much free  $H_2SO_4$  is found. Schröder's method correctly determines  $Na_2SO_4$ , and gives satisfactory results for small quantities of  $H_2SO_4$  in presence or absence of salts, but for powder treated with  $Na_2SO_3$  or  $NaHSO_3$ indicates an excessive amount of  $SO_4^{\prime\prime}$ . (I) and (II) cannot be used for leather tanned with sulphited or sulphite-cellulose extracts. Atkin and Thompson's "acid no." gives lower vals. with an increase in acid content; it is of use in evaluating the harmful effects of acid on the leather tissue. CH. ABS.

Casein adhesives and their applications. J. TAYLOR (Synth. Appl. Fin., 1934, 5, 34-36).

See also A., May, 531, Vegetable tannins and their behaviour towards proteins. Quebracho tannin. 540, Gelatin-tannin reaction. 571, Tannin from fresh tea leaves.

# PATENTS.

Insoluble cement [adhesive] solution. H. G. HALLORAN (U.S.P. 1,926,943, 12.9.33. Appl., 9.4.32).— A waterproof adhesive is manufactured by digestion of leather (with its tanning agent) in H<sub>2</sub>O at elevated pressure. Rubber latex and S may be added for impregnation of fibres. B. M. V.

Fatty acid condensation products. Alkylated phenols.—See III. Artificial leather. Shoe stiffeners.—See V. Gelatin blanks.—See XXI.

# XVI.—AGRICULTURE.

Measurement of imbibitional water [in soils]. E. W. RUSSELL and R. S. GUPTA (J. Agric. Sci., 1934, 24, 315—325).—Methods for determining imbibitional  $H_2O$  (I) by wt. and by vol. yielded concordant results except in the case of one soil containing a high proportion of easily decomposable org. matter. The swelling of wetted soils is probably influenced by the closeness of packing of the particles, and the direct vol.-measurement of (I) may be inaccurate. A. G. P.

Photomicrographic study of contact places in soils. G. I. POKROVSKI and S. I. SSINELSCHTSCHIKOV (Kolloid-Z., 1934, 67, 35—37).—An apparatus for measuring the swelling of soils in  $H_2O$  is described. Photomicrographs show that swelling occurs only with spherical, easily deformable particles, whilst the non-swelling particles have a structure like sand. E. S. H.

Determination of hydrolytic acidity [of soil]. O. K. KEDROV-ZIKHMAN and A. A. OSINA (Chem. Social. Agric., 1933, No. 4, 71–78).—Kappen's, Vageler and Woltersdorf's, and Csiky's methods are inadequate. A 25-g. sample of soil should be washed on a funnel.

CH. ABS.

Potassium in calcareous soils. I. Solubility and availability. II. Properties of replaceable potassium. W. T. McGeorge (Arizona Agric. Exp. Sta. Tech. Bull., 1933, No. 50, 1—19, 20—42).—I. Data for a no. of high-K soils are recorded. In some cases the H<sub>2</sub>Osolubility of the K was small, but adsorbed K was sufficiently readily sol. in carbonated H<sub>2</sub>O to be readily available to plants. In most cases the availability (Neubauer test) of soil-K and the % of H<sub>2</sub>O-sol. K declined with depth. Subsoils have a high capacity for fixing K, and cultivation tends to increase their K contents as a result of leaching from the surface layers.

II. Exchangeable K is readily displaced from calcareous soils (I) by 0.1N solutions of  $NH_4$  salts, but not by Ca salts. In non-calcareous soils (II) the two series of solutions are equally effective. Ca salts depress the hydrolysis of K zeolites and increase fixation of K in (I), but decrease it in (II). Liming materials depress the solubility of soil-K below the theoretical val. observed by Magistad. Neubauer vals. in (I) are > in (II) having similar proportions of replaceable K (III), whereas the solubility of the (III) in (II) is > in (I). Maintenance of (I) in a puddled condition for long periods did not change the replaceability of K. Leaching of (III) from (I) is small. Heating to 750° increased the (III) in calcareous sandy loams but not in silty clay loams. Grinding in a ball mill increased the (III) in (I) but not in peat soils. The exchange capacity of the inorg. complex is increased by grinding, but that of org. colloids is not affected. Evidence is presented indicating that a large proportion of K in (I) is absorbed by noncryst. colloids having forms resembling either synthetic zeolites or isoelectric ppts. A. G. P.

Distinction between magnesium absorbed and that exchangeable, four years after lysimeter incorporations of oxides and carbonates. W. H. MCINTYRE, W. M. SHAW, and B. ROBINSON (Soil Sci., 1934, 37, 289-303).-Absorption of Mg was > the exchange capacity of the soil as measured by customary methods, the absorbed Mg resisting 8 successive leachings or 4 successive extractions with N-NH<sub>4</sub>Cl. The exchange capacity of soil was not affected by treatment with Mg. Repeated extraction with 0.02N-HNO3 (maintained) recovered as much absorbed Mg as did digestion with conc. HCl. Applications of Mg increased the sol. Al in soils, probably as a result of partial decomp. of the Al complex. Neither K nor Ca", but only H, is displaced from the complex by Mg, although the exchange H of the colloid determined by usual methods was equiv. to only 10% of the Mg absorbed. Two types of clay acids in soils are indicated, one absorbing normal amounts of Mg in a replaceable form, and one reacting with much larger proportions of Mg to form Mg complexes not recorded by current methods for determining exchangeable bases. A. G. P.

Fixation of phosphates by clay soils. G. D. SCARSETH and J. W. TIDMORE (J. Amer. Soc. Agron., 1934, 26, 152—162).—On acid and calcareous soils very heavy dressings of superphosphate were necessary to obtain the max. crop response. The efficiency of P fertilisers decreased rapidly with the time of contact with soil. The availability (I) of phosphates examined was in the order: superphosphate,  $NH_4H_2PO_4$ ,  $Ca(H_2PO_4)_2$ ,  $Ca_3PO_4$ ,  $Fe^{III}$  phosphate. Treatment with  $CaCO_3$  reduced the (I) of readily sol.  $PO_4^{\prime\prime\prime}$  when applied immediately before planting on acid soils, but subsequently increased it after equilibrium was established and no more free  $CaCO_3$  remained. Fixation of  $PO_4^{\prime\prime\prime}$ was more rapid from salts of higher solubility. A. G. P.

Electrodialysis as a means of studying the nature of soil phosphates. L. A. DEAN (Soil Sci., 1934, 37, 253—266).—Electrodialysis (I) removes easily sol.  $PO_4^{\prime\prime\prime}$  from soils, but Fe phosphates are removed much less rapidly than by 0.002N-H<sub>2</sub>SO<sub>4</sub>. Difficultly sol.  $PO_4^{\prime\prime\prime}$  in all soils examined responded similarly to (I) and to acid extraction, and resembled the compound formed by addition of sol.  $PO_4^{\prime\prime\prime}$  to hydrated Fe oxides (dufrenite). The rate of removal of  $PO_4^{\prime\prime\prime}$  from soils by (I) furnishes information as to the nature of the P compounds present, but relative availability is more satisfactorily determined by acid extraction. A. G. P.

Potassium-lime problem in soils. H. JENNY and E. R. SHADE (J. Amer. Soc. Agron., 1934, 26, 162–170). —Displacement of adsorbed K (I) from soil colloids, permutits, natural and artificial soils by  $CaCO_3$  is general and extensive, and occurs in presence of Cl',  $SO_4''$ ,  $CO_3''$ ,  $HCO_3'$ , OH', and  $PO_4'''$ . Considerable amounts of (I) are liberated by  $CO_2$ , and further addition of  $CaCO_3$  increases these amounts. Micro-organisms reduce the leaching of K from colloid systems, especially in presence of  $CaCO_3$ . In artificial soils of low K content addition of  $CaCO_3$  may reduce the [K'] of the solution below that of unlimed controls as a result of biological resorption of K and disturbance of ionic exchange.

A.G.P.

Factors affecting the accumulation of nitratenitrogen in high-plain soils. H. H. FINNELL (Oklahoma Agric. Exp. Sta. Bull., 1932, No. 203, 47 pp.).— The effects of rainfall and cropping on the distribution of  $NO_3'$  in, and leaching from, these soils are examined. A. G. P.

Migration method for determination of replaceable bases in soils. L. C. WHEETING (Soil Sci., 1934, 37, 243—252).—Adsorbed bases are displaced from soil by migration of  $NH_4$  from  $NH_4OAc$ , the whole system being arranged in a column of agar gel placed between appropriate electrodes. The relative movement of ions is indicated by inclusion of  $Co(OAc)_2$  in the area adjacent to the positive electrode. Vals. obtained by this method were comparable with those by customary leaching methods in respect of Ca<sup>°</sup>, but were markedly higher for Mg<sup>°</sup>, K<sup>°</sup>, and Na<sup>°</sup>. A. G. P.

Culture of barley for brewing. E. S. BEAVAN (J. Inst. Brew., 1934, 40, 188-203).—A lecture.

Effects of soil temperature on absorption of water by plants. P. J. KRAMER (Science, 1934, 79, 371-372).—A rise in soil temp. increases the H<sub>2</sub>Osupplying capacity of soil and probably affects the movement of H<sub>2</sub>O from soil to root in a similar manner. L. S. T.

Fixation of potash in difficultly available form in soils. N. J. VOLK (Soil Sci., 1934, 37, 267–287).— Treatment of soil with K salts followed by alternate wetting and drying caused the fixation of much K in a non-replaceable form. Such fixation did not occur if soils were maintained in a moist condition. The fixation process is influenced by the nature and proportion of the soil colloids present. The amount of K fixed is increased by previous leaching with Na<sub>2</sub>CO<sub>3</sub> and decreased by leaching with HCl. Soils treated with Ca(OH)<sub>2</sub> and alternately wetted and dried fixed more K than unlimed controls under similar conditions. Synthetic mixtures of Al<sub>2</sub>O<sub>3</sub> gel, SiO<sub>2</sub> gel, Ca(OH)<sub>2</sub>, and sand did not fix K. The difficultly sol. form of fixed K is probably muscovite. A. G. P.

Comparison of the [fertiliser] action of ground phosphorite and superphosphate. D. V. DRUZHININ and Z. I. STROGANOVA (Min. Oudobr., 1932, 1, 53–70).— In soils of low alkalinity (50–60% of bases) application of a double quantity of ground phosphorite (I) gives better results with cereals than superphosphates (II); these soils also require heavy liming (>38%). In strongly acid soils (I) treatment can be followed by moderate liming. For soils containing > 70% of bases, (II) is better than (I) unless P<sub>2</sub>O<sub>5</sub> deficiency is pronounced. CH. ABS.

Determination of potassium in mixed fertilisers containing sulphate. C. KRÜGEL and A. RETTER (Z. anal. Chem., 1934, 96, 314—319).—The fertiliser is extracted with HCl, and  $K + NH_4$  in an aliquot portion are pptd. with Na<sub>3</sub>Co(NO<sub>2</sub>)<sub>6</sub>. The ppt. is dissolved in conc. HCl at 45° (whereby NH<sub>4</sub> is decomposed by HNO<sub>2</sub>), evaporated with HClO<sub>4</sub>, and washed out with EtOH.

J. S. A.

Calcium cyanamide as a nitrogenous fertiliser. T. R. MOYER (Soil Sci., 1934, 37, 305–331).—The N recovery in crops from added CaCN<sub>2</sub> (I) is, in general, = that from other high-grade N fertilisers. In very acid sands in which leaching was prevented, the effect of fertilisers was in the order  $NaNO_3 > (I) > (NH_4)_2SO_4$ . The rate of leaching of (I) was < that of other inorg. N fertilisers. The decrease in [H] following treatment of soil with (I) was > that produced by an equal wt. of Ca(OH)<sub>2</sub>. The former has also a permanent physiological alkalinity. The use of (I) as a weed killer is also examined. A. G. P.

Influence of soil reaction on fertilising effect of calcium cyanamide. J. KROTOWICZÓWNA (Polish Agric. Forestal Ann., 1931, 25, 235–272).—CaCN<sub>2</sub> had a good fertilising action in the range  $p_{\rm H}$  4–8. The crop increase in acidic was slightly > in neutral soils. CH. ABS.

Factors influencing the efficient application of nitrogenous fertilisers to soil. H. J. KOCH (J. S. African Chem. Inst., 1934, 17, 15–18).—When used in moist climates, the varying  $H_2O$  content of hygroscopic fertilisers greatly affects the amount delivered by a spreading machine. The size and shape of the particles, the apparent sp. gr., and the friction between particles also affect the ease of application. The best shape is spherical and there should be no very fine particles ; the best angle of repose is 40°. Once the fertiliser is in the soil absorption of  $H_2O$  is advantageous. A. G.

Use of small amounts of nitrogen for maize in addition to phosphorus and potassium. S. R. MLES (J. Amer. Soc. Agron., 1934, 26, 129–137).— Additions of small amounts of a N fertiliser (2—6 lb. of N per acre) to a P-K mixture applied at sowing reduced the yield of maize on the soils examined. The effect was independent of season, texture of soil, yield level of the crop, use of cattle manure, or applications of NaNO<sub>3</sub> later in the growing season, but was less marked when the maize crop was preceded by clover or lucerne. In general, org. manutes appeared preferable to artificial fertilisers. A. G. P.

Available phosphorus in soil and the phosphorus content of grain as influenced by phosphorus applications to soil. A. G. WEIDEMANN (J. Amer. Soc. Agron., 1934, 26, 170–178).—In a soil showing slight deficiency of  $PO_4^{\prime\prime\prime}$  (Truog test), applications of superphosphate produced no consistent increase in crop yields or  $PO_4^{\prime\prime\prime}$  content of grain, and the increased  $PO_4^{\prime\prime\prime}$  contents of chaff and straw were irregular

A. G. P.

Effect of sodium nitrate on growth and nitrogen content of a lucerne and grass mixture. H. G. THORNTON and H. NICOL (J. Agric. Sci., 1934, 24, 269– 282).—In sand cultures, treatment with NaNO<sub>3</sub> did not affect the yield or N content of lucerne (I) except in the case of the larger amounts, which somewhat restricted root growth. In mixed cultures of (I) and rye grass (II) the growth of (II) varied directly and of (I) inversely with the amount of NaNO<sub>3</sub> applied. Reduced growth of (I) is associated with root competition with (II). The N contents of the combined tops and the combined roots of (I) and (II) were inversely related to the NO<sub>3</sub>' dosage. Evidence is presented indicating that (II) utilised N fixed by the roots of (I). A. G. P. Influence of mineral fertilisers on botanical composition of meadow vegetation. В. D. Омоянко (Chem. Social. Agric., 1933, No. 4, 46—51).—Nitrates stimulate grain-bearing grasses; K is important for legumes. Сн. Авs.

Source of nitrogen and active acidity of the medium as factors in the growth of meadow grasses. V. V. TZERLING (Chem. Social. Agric., 1933, No. 4, 52—57).—The active acidity of the soil is influenced by the type of fertiliser-N. CH. ABS.

Cultivation of grasses and change in the quantity of available nutrients in the soil. A. V. VLADIMIROV (Chem. Social. Agric., 1933, No. 5, 49–55).—Crops following grasses responded to K, P, and N fertilisation in that (decreasing) order. CH. ABS.

Fertiliser experiments on "run-out" hay lands. F. S. PRINCE, P. T. BLOOD, T. G. PHILLIPS, and G. P. PERCIVAL (New Hamps. Agric. Exp. Sta. Bull., 1933, No. 271, 17 pp.).—Of the principal nutrients K was the most effective in increasing the yield and prolonging the life of lucerne (I) on these soils. Good crops of (I) were produced on soils having  $p_{\rm H} \ge 5.6$ . The composition of (I) was not appreciably altered by fertiliser treatment. For timothy N fertilisers produced the greatest improvement. "Worn-out" hay soils are deficient in available plant nutrients as a result of a decline in the supply of rapidly decaying org. matter and lowered bacterial activity. This condition is frequently intensified by the development of high acidity. A. G. P.

Five years fertiliser experiments with Irish potatoes in eastern North Carolina. C. B. WIL-LIAMS, H. B. MANN, and J. J. SKINNER (N. Carolina Agric. Exp. Sta. Bull., 1933, No. 283, 21 pp.).—Amongst different salts of K used in mixed fertilisers KCl gave best returns.  $(NH_4)_2SO_4$  (I) and NaNO<sub>3</sub> (II) were equally efficient, but produced smaller yields than either used in conjunction with org. N manures. Urea and Leuna saltpetre were somewhat inferior to (I) or (II). Best results were obtained with mixtures of (II) and cottonseed meal (III) with 65% NO<sub>3</sub>-N or with (I) and (III) containing 80% NH<sub>4</sub>-N. A. G. P.

Copper injury in relation to time of spraying. E. L. LOEWEL (Z. Pflanzenkr. Pflanzenschutz, 1934, 44, 71–76).—Pre-blossom spraying of apples causes little injury. During the flowering season  $\frac{1}{2}$ % Cu preps. do not injure flowers, and the proportion of scorched fruit is small. The risk of injury increases from petalfall until mid-June, but is much lessened after the end of June. A. G. P.

Zinc sulphate for tung oil groves. H. A. GARDNER (Sci. Sect. Nat. Paint, Varnish & Lacquer Assoc., Inc., Apr., 1934, Circ. No. 452, 47–48).—ZnSO<sub>4</sub> is a cure for "bronzing" of tung oil trees. G. H. C.

Rotenone and its importance in plant protection. H. KRIEG (Zentr. Bakt. Par., 1934, II, **89**, 475–476).— Comparison is made of the insecticidal uses of rotenone and pyrethrin. A. G. P.

Contact insecticides. VI. (1) Reactions of certain insects to controlled applications of various concentrated chemicals. (2) New technique for initial appraisal of proposed contact insecticides. W. C. O'KANE, G. L. WALKER, H. G. GUY, and O. J. SMITH (New Hampshire Agric. Exp. Sta. Tech. Bull., 1933, No. 54, 23 pp.; cf. B., 1933, 37).—(1) Physiological responses of a no. of insects to pyrethrum and nicotine and some supplementary spray materials are recorded.

(2) The above reactions are utilised in determining the efficiency of insecticides. Results thus obtained indicate that customary methods involving dilution of the poison in solutions or emulsions may lead to the masking of certain lethal properties. A. G. P.

Determining Na [in fertilisers].—See VII. CaOsludge disposal.—See XXIII.

See also A., May, 500, Colorimetric determination of  $NH_3$  in small amounts, and of  $PO_4^{\prime\prime\prime}$  in plant ashes. 507, Soils, and soil mapping. 569, Dormancy in *Tilia* seeds. 570, Nutrients used by leaves and growth of apple trees.

#### PATENTS.

Germicide, fungicide, etc. and method of using same. M. S. KHARASCH, ASST. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,920,311, 1.8.33. Appl., 30.7.28). —A fungicide for cereal grains comprises a mixture of 10% of a condensation product of a Hg<sup>II</sup> salt and NH<sub>2</sub>Ph or one of its derivatives with 90% of an inert filler, e.g., talc. A. R. P.

Organo-Pb compounds.-See III.

# XVII.—SUGARS; STARCHES; GUMS.

Effect of the so-called harmful space of the [sugar-juice] diffusion battery. I. Theory. E. POKORNÝ and J. VONDRÁK. II. Practice. J. VONDRÁK and E. POKORNÝ (Z. Zuckerind. Czechoslov., 1934, 58, 241—245, 249—253; 257—262, 265—268).—I. Calculations are reproduced leading to the conclusion that the "harmful space" is not actually disadvantageous, but that, on the contrary, it can lead to the production of denser juices and better-exhausted slices.

II. This view is confirmed by experiments made in an experimental plant, and later in factory practice. Even when in the design of the battery such space was unusually large it was possible to obtain juices of ordinary density. There is no reason why wider connecting pipes than are general should not be used, so that the rate of circulation of the juice can be improved. J. P. O.

Control of sugar boiling by means of electrical conductivity. IV. O. SPENGLER, F. TÖDT, and J. WIG-AND (Z. Ver. deut. Zucker-Ind., 1934, 84, 93-111; cf. B., 1933, 1075).—Reports from 11 German factories in which this system was tried last campaign, for raw- or white-sugar strikes, are discussed. Only two were unfavourable, and these were due largely to special conditions. In most factories the vac. maintained throughout boiling is sufficiently const. to render correction of  $\kappa$  for temp. variations unnecessary. Provision for temp. compensation, where necessary, can be made in a new indicating or recording apparatus by Siemens und Halske. Relative merits of different types of apparatus are discussed. Cu electrodes in some pans have become bent by the vigorous circulation of massecuite, and in other cases a deposit has formed on them. These defects are avoided by the adoption of rustless Ni-Cr steel instead of Cu. J. H. L.

Inversion of sucrose in concentrated solutions and in marzipan pastes by means of technical invertase preparations. O. SPENGLER and R. WEIDEN-HAGEN (Z. Ver. deut. Zucker-Ind., 1934, 84, 218—227).— Invertase hydrolyses sucrose (I) at all concns. up to saturation. In 100 c.c. of solution containing 60 g. of (I) and 0.1 c.c. of "Convertit" or Merck's "Invertin," and buffered at  $p_{\rm H} 4.62$ , inversion was complete in 3—5 days at 50° and almost complete in 14 days at 20°. In saturated solutions at these temp. the same enzyme concn. effected about 40% inversion in a fortnight and was still acting. In marzipan pastes containing 33—37% of (I) 0.125% of the same enzyme preps. inverted about a third of the (I) in a week at 20°. J. H. L.

Dextrin (British gum) formed by heating dry starch. J. R. KATZ [with F. J. F. MUSCHTER, JUN., and A. WEIDINGER] (Rec. trav. chim., 1934, 53, 555—560).— When potato or tapioca starch, previously dehydrated by  $P_2O_5$  at 20° in vac., is heated at 165—190° or 180— 200°, respectively, the X-ray pattern changes, and the solubility of the product in 10 pts. of hot H<sub>2</sub>O changes from 5—10 to 90%. The solutions thus obtained do not "retrograde" (give ppts. when kept). This is due either to oxidation or anhydride formation, with shortening of the chain. A second change, of unknown nature, occurs at 210—220°. R. S. C.

Bulking vals. of gums.—See I. Fermentable sugars and bread-making. Fodder sugar.—See XIX.

# PATENT.

Inulin nitrate.-See XXII.

# XVIII.—FERMENTATION INDUSTRIES.

Comparison of the Windisch-Kolbach and Pollak-Egloffstein methods of determining the diastatic power of malt and malt extracts. A. HEIDUSCHKA and E. BISCHOFF (Z. ges. Brauw., 1933, 56, 53-55; Chem. Zentr., 1933, ii, 3060).—The Windisch-Kolbach method is preferable. H. J. E.

Fermentation of (A) red, (B) white, wines. W. V. CRUESS (Fruit Products J., 1934, 13, [A] 199-204, 207, 212, 214, 217; [B] 230-232, 249).—Methods of prep. of white and red wines are described and compared. E. B. H.

Manufacture of poi from taro in Hawaii, with special emphasis on its fermentation. O. N. ALLEN and E. K. ALLEN (Hawaii Agric. Exp. Sta. Bull., 1933, No. 70, 32 pp.).—Two stages of the fermentation of taro root are distinguished : (a) an initial period (3 days) of rapid acid production ( $p_{\rm H}$  4.5), during which bacterial activity predominates ; (b) a second phase in which yeasts, mycoderma, and oidia increase steadily and acid formation is retarded. A min.  $p_{\rm H}$  (3.8) is reached after 6 days and there is little subsequent change. A. G. P.

Solvents.—See III. Barley for brewing.—See XVI. Fermentable sugars and bread-making.— See XIX. Lymphatin.—See XX.

See also A., May, 559, Purification of malt amylase.

## PATENTS.

Preparing a wort. H. C. Gore and C. N. Frey, Assrs. to Standard Brands, Inc. (U.S.P. 1,922,730, 15.8.33. Appl., 11.3.30) .- To avoid clogging of the filters, the solid matter of a cereal mash is rapidly compacted in thin layers, slimy matter being thus uniformly retained. A finer grind than usual is necessary, with the additional advantages of accelerated mashing and increased yield of extract. Preferably, > 5-15% of the grist should remain on a 60-mesh sieve. On a commercial scale, and especially to facilitate continuousfiltration processes, e.g., in connexion with countercurrent washing, woven wire or, better, perforated metal sheets may replace the filter cloth. Suction and/or pressure filters may be used, particularly suction filters of the rotating-drum type, the deposit being removed by a scraper, with or without washing. The filter-cake holds substantially less H<sub>2</sub>O than normal draff, whilst the filtrate, which is usually slightly turbid, may if necessary be obtained bright by adding, e.g., kieselguhr I. A. P. and filtering through cloth.

Manufacture of absolute alcohol. J. RENNOTTE (B.P. 409,053, 18.11.32).—The EtOH content of vapours from the rectifiers is reduced (substantially below 96° Gay-Lussac) so that the heat supplied by them corresponds to the heat necessary for dehydration (I), thus effecting an economy of fuel. EtOH passes into the dehydrating apparatus in the form of vapour. The ternary mixture used for (I) is stirred vigorously, additions of  $H_2O$  being regulated so that the alcoholic portion is completely dissolved in  $H_2O$ , thus isolating the carrier (C) (e.g.,  $C_6H_6$ ). The mixture then separates into two layers, viz., dil. EtOH and pure C. If the lower layer remains cloudy it may be cleared by electrical means. I. A. P.

Manufacture of materials [beverages] containing ethanol [ethyl alcohol]. L. LILIENFELD (B.P. 408,989 and Addn. B.P. 409,018, 15.7.32) .- Inebriating and other unfavourable effects due to beverages containing EtOH are reduced or eliminated by adding suitable simple or mixed alkyl, alkoxy-, or OH-acid derivatives of carbohydrates (D) of the type  $(C_6H_{10}O_5)_n$ , either during prep. or to the finished beverage, depending on the method of manufacture. D reduce the amount of EtOH passing into the blood, and are themselves harmless. Examples of D are Me, Et, or Pr derivatives of cellulose, starch, dextrin, etc. ; they must be sol. in aq. EtOH or in H2O and aq. EtOH, and compatible with special constituents in particular beverages. D are used in amounts equiv. to 10-25 or 30-60 pts. (wt.-%) of EtOH. Methods of incorporating D in potable spirits, liqueurs, wines, beers, etc. are described, together with methods of prep. of D. In a modified process, D may be replaced by suitable simple or mixed esters of cellulose, starch, dextrin, etc.

I. A. P.

Production of stabilised mixture of spirit and [sparkling] water. F. MUSSET (B.P. 408,906, 15.11.33. Fr., 3.10.33).—Brandy, whisky, etc. are diluted by introducing H<sub>2</sub>O charged with CO<sub>2</sub> into the bottles under pressure (2—3 atm.). Distilled H<sub>2</sub>O is used and/or detrimental impurities are removed previously from the CO<sub>2</sub> by bubbling it through an alcoholic liquor identical with that to which it is later to be added, to eliminate risk of development of turbidity on storage.

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## XIX.—FOODS.

Spontaneous heating of grain. B. L. ISATSCHENKO, M. M. ONTSCHUKOVA, A. A. PREDTETSCHENSKAJA, and T. V. LIPSKAJA (Compt. rend. Acad. Sci. U.R.S.S., 1934, 1, 507—509).—The development of micro-organisms, especially in presence of moisture and O<sub>2</sub>, is primarily responsible for the spontaneous heating of grain. Under favourable conditions the temp. can rise to 58°, sterilising the seed and yielding a black mass. Loss of carbohydrate and fat occurs during the heating, and the activity of the amylase is diminished. J. W. S.

Are the ferments of grain injured by conditioning ? L. HOPF (Mühlenlab., 1934, 4, 47-50) .- A brief review is given of investigations into the effect of conditioning (C) on the diastatic activity (A) of grain and related subjects [e.g., effect of temp. (T) and timeof moistening on germination (G), and of T on A, both with and without moisture, loss of substance by the endosperm (E) during respiration]. A is lower shortly after C, but has increased 24 hr. after C to > its original val. The grain T should be  $> 50^\circ$ . The scutellum forms the main seat of the diastase (D), but the E contains sufficient D to solubilise its contents. It appears that no, or very little, D is present in the aleurone cells. Increase in A can be attained without G. In wheats having high A (e.g., sprouted German wheats) hulling may be carried out before C; in this case removal of the germ seems to be an advantage, but this is counterbalanced by contamination of E with crease dirt and absorption of H<sub>2</sub>O during washing. E. A. F.

Maturing [ageing] of wheat flour and its biochemical principles. N. P. KOSMIN [with K. A. ALAKRINSKAJA and V. D. BONDAREV] (Mühlenlab., 1934, 4, 17–32).—The decrease in  $p_{\rm H}$  of wheat flour (F) during storage is not great enough to account for the changes in the condition (e.g., elasticity and extensibility) of the gluten (G). The direct cause of ageing (A) is the hydrolysis of the fats (H), which leads to the accumulation of 1% of free unsaturated fatty (oleic, linoleic, and linolenic) acids (I) in the F. Removal of (I) by Et<sub>2</sub>O extraction produced a complete "rejuvenation" of the F, the Gacquiring the properties of G from freshly milled F. Inversely, addition of (I), but not saturated fatty acids containing a similar no. of C atoms, to fresh F produced a marked A effect. Rise in temp. and in moisture con-tent accelerates H. H is independent of the presence of  $O_2$ , but with  $O_2$  rancidity develops. The sp. effect of (I) on the colloidal G complex cannot at present be explained. It appears likely that the oxidising effect of maturing agents (e.g., Novadel, Agene) is not the cause of their A effect, but is only an accompanying phenomenon.

E. A. F.

Digestibility of wheat and millet proteins. W. H. ADOLPH and T. WANG (Chinese Med. J., 1934, 48, 59-61).—White bread has an apparent coeff. of digestibility of 88%, compared with 74.4% for steamed millet cake. No account was taken of metabolic N, the N intake and fæcal N alone being determined.

P. G. M.

Fermentable sugars, alcoholic fermentation, and gas production during bread-making. R. GUILLEMET, C. SCHELL, and P. LE FUR (Compt. rend., 553

1934, 198, 1083—1085).—Glucose (I), maltose (II), sucrose (III), and raffinose (IV) with brewers' top yeast (pressed) (V) give 70—73% of EtOH, the CO<sub>2</sub>/EtOH ratio (A) being about 0.96. Wheat-flour dough contains (I) (<0.15%), (II) (1.5—3%) and (III) (1—1.8%) but no (IV). Fermentation of (III) and, more so, of (II) is more rapid in the dough than in aq. solution, and 3 times as rapid at 27° as at 20°. During breadmaking A increases from 0.7 at the beginning to 0.8 at the end, the total deficiency of CO<sub>2</sub> being 20%. Only the gas evolved after the last "turning" of the dough determines the size of the loaf. When 0.2% of (V) is used at 27°, the flour used for bread-making should contain  $\ll 3\%$  of fermentable sugars; not all flours contain this amount. R. S. C.

Does butter fat change during the baking process? W. STOLDT (Pharm. Zentr., 1934, 75, 297– 298).—Fat extracted from brown cakes prepared with  $K_2CO_3$  (? KHCO<sub>3</sub>) as raising agent was unchanged (e.g., in sap., Reichert-Meissl, Polenske, and I vals., and in refractometer no.) by baking. E. A. F.

Characteristics of an abnormal type of eggshell. H. J. ALMQUIST and B. R. BURMESTER (Poultry Sci., 1934, 13, 116—122).—"Glassy" shells have lower breaking strength and porosity, are thinner, and have smaller proportions of shell membrane than normal eggs. They are also characterised by higher proportions of shell-protein and slightly lower  $CaCO_3$  content than normal. The % of protein does not affect the breaking strength of the shell and tends to vary inversely as the thickness of the shell. A. G. P.

The  $p_{\rm H}$  of fresh and storage eggs. S. E. ERIKSON, R. E. BOYDEN, J. H. MARTIN, and W. M. INSKO, JUN. (Kentucky Agric. Exp. Sta. Bull., 1932, No. 335, 359— 394).—In determinations of  $p_{\rm H}$  of yolks and whites, dilution with H<sub>2</sub>O (1:1) is permissible. Observations should be made within 30 min. of breaking the eggs. No significant change in  $p_{\rm H}$  occurred in unbroken eggs within 9 hr. of collection. Average variations in fresh eggs were small (< 0.05  $p_{\rm H}$  unit), mean vals. being 6.32 for yolks and 7.92 for whites. Corresponding vals. for storage eggs were 6.98 and 8.90, but variations were under in this case (> 0.1  $p_{\rm H}$  unit). A. G. P.

Physico-chemical changes produced by the cooking of potatoes. M. D. SWEETMAN (Amer. Potato J., 1933, 10, 169—173).—Cooking causes mechanical disintegration by separation of cells (not bursting), partial gelatinisation of starch, dissolution of some of the pectic substances, coagulation of most of the protein, and some caramelisation of the sugar. The cellulose is rendered more digestible. CH. ABS.

Dried apricot paste. A. S. SHALLAH and P. F. NICHOLS (Fruit Products J., 1934, 13, 238-240, 246, 251).—An account is given of the prep. of dried apricot paste in Syria, and of the method worked out by the Food Products Laboratory in order to utilise small, very ripe apricots unsuitable for canning. The paste can be used as a sweet instead of dried apricots, and to make a beverage. E. B. H.

Refrigeration in the food industry. P. BILHAM (Chem. & Ind., 1934, 436-440).-A lecture.

Refrigerated [food] transport. W. H. GAUNT (Chem. & Ind., 1934, 440-442).—A lecture.

Applications of solid carbon dioxide. F. HEYWOOD (Chem. & Ind., 1934, 443—446).—A review, dealing mainly with food preservation.

**Poultry-feeding experiments.** H. EMBLEDON (Arizona Agric. Exp. Sta. Bull., 1933, No. 143, 79—103).— Rations having narrow nutrient ratios (1:3.6) and containing a high proportion of concentrates gave best results. A. G. P.

Cod-liver oil for laying pullets. J. H. MARTIN and W. M. INSKO (Kentucky Agric. Exp. Sta. Bull., 1933, No. 337, 3—22).—The hatchability of eggs was improved by supplements of cod-liver oil to diets for confined hens, or by direct exposure to sunshine, or by allowing grass range. A. G. P.

Comparative values of some commercial protein supplements in the rations of growing chicks. O. JOHNSON and D. BRAZIE (J. Agric. Res., 1934, 48, 183—186).—The effect on the growth of chicks of various protein concentrates as supplements to their diet bore no relationship to the biological val. of the protein. Growth was greatest with Alaska herring meal and least with Argentine meat scrap, whilst fish shreds, skim-milk powder, Vico meat scrap, and various blends held intermediate positions. W. O. K.

Nutritive value of legume husks. R. G. LINTON, A. N. WILSON, and S. J. WATSON (J. Agric. Sci., 1934, 24, 260—268).—Chemical analyses and digestibility trials with sheep are recorded. Pea, bean, and lentil husks have, in general, low protein contents, but moderately high starch equivs. In rations for sheep the inclusion of pea husks permits suitable adjustment of the Ca: P ratio of the ration. A. G. P.

Biological value of rations containing fish meal. J. L. ST. JOHN, J. S. CARVER, O. JOHNSON, S. A. MOORE, and H. GERRITZ (J. Nutrition, 1934, 7, 13-25).—The val. of rations supplemented with herring meal was high, but decreased somewhat as the level of total protein intake was raised. Optimum utilisation of protein (I) was attained with rations containing 15% of (I). A. G. P.

Feeding of fodder sugar and [dried beet] slices to horses. F. HONCAMP, A. BÖMER, P. RINTELEN, and K. HÖTZEL (Z. Ver. deut. Zucker-Ind., 1934, 84, 112— 163).—Comparative 3-month trials with stallions and working geldings and mares showed no disadvantage in any respect from the partial or complete replacement of the normal full ration of oats by rations, equiv. in starch val. and protein, consisting of fodder sugar (I) (raw sugar denatured with beet powder, polarisation val. 83%) or dried beet slices (II) (50—66% of sugar) with soyabean meal or other nitrogenous adjuncts. No aversion was shown to the sugar rations, which amounted to 2—2.5 kg. of (I) or 2.5—4 kg. of (II) per head per day. J. H. L.

Solid  $CO_2$ .—See VII. Sucrose inversion in marzipan pastes.—See XVII.

See also A., May, 500, Micro-ashing process. 501, Determination of PO<sub>4</sub><sup>'''</sup> [in foods]. 511,

Synthesis of vitamin-C. 553, Nutritive val. of alfalfa leaves and stems. 565, Effect of NaNO<sub>3</sub> on anaërobic bacteria [in meat]. 568—9, Vitamins (various). 571, Phosphatides of wheaten meal.

#### PATENTS.

**Preparation of yeast-raised dough for baking.** H. JUNG (U.S.P. 1,922,096, 15.8.33. Appl., 22.6.31).— The dough is prepared in 2 portions (A, B), both containing yeast, but all the NaCl being in B. Portion A is kept at  $\gg 15 \cdot 5^{\circ}$ , while B is kept at 32—38° for  $\ll 1$  hr. as required. A and B are then mixed, the resulting temp. being about 27°, and the dough allowed to ferment until ready. The low temp. in A and the high [NaCl] in B check yeast activity, while allowing the gluten to mature. E. B. H.

Pasteurising apparatus [for milk etc.]. C. E. MITCHUM (U.S.P. 1,925,877, 5.9.33. Appl., 6.4.31).— The apparatus comprises an outer tank  $(\mathcal{A})$  and an inner tank  $(\mathcal{B})$ . After charging the milk (e.g.) into  $\mathcal{B}$  and fastening the cover,  $\mathcal{A}$  is filled until it entirely submerges  $\mathcal{B}$  in heating medium, which is kept in rapid circulation by tangential injection. B. M. V.

Preparation of whey concentrate. E. E. EL-DREDGE, Assr. to KRAFT-PHENIX CHEESE CORP. (U.S.P. 1,923,427, 22.8.33. Appl., 13.2.29).—The difficulty of using dried whey caused by caking is overcome by allowing it to absorb  $H_2O$  (to cake) and then mechanically grinding it. E. B. H.

Egg preservation. P. F. SHARP, ASST. to CORNELL UNIV. (U.S.P. 1,922,143, 15.8.33. Appl., 11.7.28).— Improved quality of the whites of storage eggs is effected by maintaining the  $p_{\rm H}$  of the white at 7.6 by storage in an atm. of CO<sub>2</sub> of suitable concn., e.g., 3% at 2.8°.

E. B. H.

Drying apparatus [for grain].—See I. Baking powder.—See VII. Packaging edible oils.—See XII. Stabilised egg products. Fish-liver oil.—See XX.

# XX.-MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Size of colloidal particles in homeopathic grinding. C. A. ROJAHN and H. WIEDER (Arch. Pharm., 1934, 272, 198-221).-The amounts (E) of the element passing into colloidal solution when it is centrifuged with H<sub>2</sub>O and gum arabic after grinding in a machine mortar for varying periods (T) with different amounts (L) of lactose (Neugebauer, A., 1927, 1137) have been determined (methods described). In agreement with Neugebauer (loc. cit.) E increases only slowly with progressive increase in T, and to effect a noteworthy increase a disproportionately large increase in Tis required. The figures in parentheses give the extreme total % increase in E (T, 3-96 hr.): Sb (2930), As (4500), Pb (4420), Fe (18,500), graphite (12,800), vegetable C (4370), Cu (2840), Hg (2370), Se (3060), Ag (2170), S (4350), and Bi (5000). L is as important as T in determining E. J. W. B.

Incompatibility of sodium hydrogen carbonate and mercurous chloride. V. LUCAS (Bol. Assoc. brasil. Pharm., 1933, 14, 301–303; Chem. Zentr., 1933, ii, 3593).—The reaction  $2 \text{HgCl} + 2 \text{NaHCO}_3 = \text{HgCl}_2 + \text{Hg} + \text{Na}_2\text{CO}_3 + \text{CO}_2 + \text{H}_2\text{O}$  occurs. A. A. E.

Testing of calcium glycerophosphate. G. BÖMMING and S. KROLL (Arch. Pharm., 1934, 272, 297-300).— On the basis of trial determinations under varying conditions the following alterations in the D.A.B. VI tests for Ca glycerophosphate are suggested: (1) the indicator colour change in titration with N-HCl should be specified; (2) in the determination of the residue on ignition, evaporation with HNO<sub>3</sub> should be specified; (3) in the molybdate test the temp. should be stated and a suitable reagent prescribed; and (4) in the test for H<sub>2</sub>SO<sub>4</sub> the quantities of added HNO<sub>3</sub> and Ba(NO<sub>3</sub>)<sub>2</sub> should be accurately specified. J. W. B.

Alcohol limits of the B.P., 1932. T. T. COCKING (Quart. J. Pharm., 1934, 7, 76–107).—The limits of the EtOH content of alcoholic tinctures and fluid extracts of the B.P., 1932, are frequently > those obtained by the use of the methods given in the B.P., 1932.

F. O. H.

Bromometric determination of phosphorus in phosphorus solutus, D.A.B. VI. B. STEMPEL (Pharm. Zentr., 1934, 57, 281).—The determination is made with excess of Br in aq. NaBr, adding KI, and back-titrating with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. A blank determination is run.

G. H. C.

Determination of mercury content of pharmaceutical mercury preparations. Separation of mercury from bismuth. E. SCHULEK and S. FLODERER (Z. anal. Chem., 1934, 96, 388–398).—Hg is converted into  $HgSO_4$  or  $Hg(NO_3)_2$  and titrated with 0.1N-NH4CNS. Hg org. compounds free from Cl', Br', CN', CNS', CNO' are destroyed by oxidation with  $H_2SO_4 + 30\% H_2O_2$ . HgSO<sub>4</sub> formed is dissolved in aq. HNO<sub>3</sub>, and Hg oxidised by KMnO<sub>4</sub>. Hg or HgO in ointments or suspensions is extracted by refluxing with 30% HNO3 and washing out with dil. aq. HNO3. Preps. of HgCl<sub>2</sub>, Hg(CN)<sub>2</sub>, etc. are dissolved in H<sub>2</sub>O (+ HCl if necessary) and HgS is pptd. The ppt. is dissolved in  $H_2SO_4 + H_2O_2$ . Gelatin preps. may require refluxing with 10% HCl.  $Hg_2Cl_2$ ,  $HgNH_2Cl$ , etc. in presence of Bi are extracted with 5% HCl + Br, brought to 20% HCl concn., and treated with H2S. HgS free from Bi is pptd. and treated as above. Resins, fats, etc. may be first destroyed by oxidation with  $H_2SO_4 + H_2O_2$ . J. S. A.

Possible decomposition of alkaloids in aqueous solution, especially during sterilisation. R. DIETZEL and G. W. KÜHL (Arch. Pharm., 1934, 272, 369—379).— Examination of the ultra-violet absorption spectra in 0.1-0.01% aq. solution shows that  $\beta$ -eucaine hydrochloride (I) suffers no appreciable change even by prolonged (8-hr.) sterilisation at 97°. A sample kept in the dark for 1 year gives a similar absorption curve, but displaced, corresponding with a 39.7% decrease in conce. This is not due to oxidative degradation since 8-hr. treatment of (I) with O<sub>2</sub> causes no changes in the spectrum, but is due to hydrolysis and separation of the H<sub>2</sub>O-insol. base, the  $p_{\rm H}$  changing from 6.98 to 5.26 over this period. J. W. B.

Composition and solubility of acriflavine and other derivatives of 2:8- [3:7-]diaminoacridine used in therapeutics. M. GAILLIOT (Quart. J. Pharm., 1934, 7, 63-75).—Methylation of diacetamidoacridine to the methochloride is generally incomplete. Separation of the methochloride (I) and hydrochloride of 3:7diaminoacridine (II) is effected by treatment with Ag<sub>2</sub>O followed by crystallisation from MeOH. The prep. of acriflavine (III) and of the neutral and basic (II) and the analysis of their mixtures are described. Solubility data for the pure substances and for their mixtures do not indicate the formation of mol. compounds of (I) and (II) (cf. B., 1930, 741). The description of "acriflavine B.P." corresponds to a mixture (approx. 1:1) of (II) and (III). F. O. H.

Lymphatin. S. TRAWKOWSKA (Wiad. Farm., 1933, 60, 661—662, 675—677).—Lymphatin, the dry extract of lymphatic glands, contains  $H_2O$  11·7, ash 4·633 (sol., 1·833), NaCl 0·975% (Na<sub>2</sub>O 1·23, K<sub>2</sub>O 1·45, CaO 0·058, MgO 0·184, Fe<sub>2</sub>O<sub>3</sub> 0·028%; Mn 3·04 mg. per 100 g. of dry substance); Cu 0·064, Ni 0·0025%, Zn 16·9, TiO<sub>2</sub> 0·011 mg. per 100 g., S 0·45, P 1·043, lipin-P 0·3026, nuclein-P 0·2032, mineral P 0·4696%, fatty acids 5·5, unsaponifiable matter 7·36, cholesterol 1·021%, I val. 57, sap. val. 230, m.p. of acids 31·2°, acid val. 25·2, total N 9·8, sol. N 2·45, protein-N 7·35, NH<sub>3</sub> 1·8, total protein 45·92%. Arginase, arbutase, lecithase, and glycolase are present. CH. Abs.

Change and stabilisation of phenol water. G. VERGEZ (Bull. Soc. Pharm. Bordeaux, 1933, 71, 56–59; Chem. Zentr., 1933, ii, 2855).—The chemical change in PhOH shown by the development of a red colour depends less on the action of light and air than on the presence of traces of Cu.  $H_2O$  redistilled from quartz gave a PhOH solution which remained colourless during 8 months. The catalytic action of Cu increases in alkaline and diminishes in acid solution. The addition of a small quantity of  $H_3BO_3$  improves stability.

L. S. T. **Constitution of carrot essence**. L. PALFRAY and A. M. LEPESQUEUR (Compt. rend., 1934, 198, 1365— 1367).—Physical properties of the essence are given. By distillation under reduced pressure, *l*-pinene and carotol have been isolated. H. G. R.

Terpenes of Java citronella oil. J. TABUTEAU (Bull. Inst. Pin, 1933, 237—246).—Raman spectra of fractions obtained by distilling this oil show the presence of methylheptenone, limonene, citronellal, geraniol, citronellol, a mixture of alcohols and acids, mixed diterpenes, and a diterpene oxide. R. S. C.

Examination of Algerian geranium oil for esters. Analytical data. B. ANGLA (Parfums de France, 1933, 11, 137—143, 180—184; Chem. Zentr., 1933, ii, 3056—3057).—Samples of doubtful origin or purity are examined by determinations of the total acid and of the acid combined as esters and comparison with vals. for pure oil. Physical and chemical data are recorded. H. J. E.

**Neroli oil.** G. LOUVEAU (Rev. Marques Parfum. Savonn., 1933, **11**, 245—246, 277—278; Chem. Zentr., 1933, ii, 2905).—1933 oil had  $d^{15}$  0.8745, 0.8731;  $\alpha_{\rm D}$  +0° 48', +0° 30';  $n_{\rm D}$  1.4709, 1.4716; acid val. 1.2, 0.9; ester val. 65.1, 59.5; ester content 22.8, 20.8%; ester val. after acetylation 159.6, 163.8; Me anthranilate 0.52, 0.60%. A. A. E.

Western American peppermint oil. H. STRAUSZ (Riechstoffind., 1933, 8, 119–120; Chem. Zentr., 1933, ii, 2905).—The oil had  $d^{15}$  0.904,  $\alpha_{\rm D}$ —25° 20′, and contained esters 5.7, ester-menthol 4.5, total menthol 55.85, menthone 19.45%. A. A. E.

**Rosmarinus officinalis** and its Italian essential oils. G. ROVESTI (Riv. ital. Essenze Prof., 1933, 15, 227—252; Chem. Zentr., 1933, ii, 2905).—A compilation of the physical properties of rosemary oil (I) of different origins according to various authors shows terpene-free (I) to have  $d^{15} 0.925$ —0.960,  $\alpha_{\rm D} 0^{\circ}$  to  $+10^{\circ}$ ,  $n_{\rm D} 1.470$ — 1.472, and solubility in 70% EtOH 2—3 vols. An abs. rosemary extract oil has  $d^{15} 0.9267$ ,  $\alpha_{\rm D}^{15} + 1.74^{\circ}$ ,  $n_{\rm D}^{15}$ 1.4791, sap. val. 28.32. L. S. T.

Determining fat etc. [in ointments etc.].-See XII.

See also A., May, 474, Luminescence of ZnO. 511, Synthesis of vitamin-C. 515, Quaternary NH<sub>4</sub> salts. 516, Reactions of betaine. 529, Indian essential oils. 531, Chemical constituents of tobacco. 533, Mercurated substitution products of di-*p*-hydroxyphenylisatin. 538—9, Alkaloids (various). 541, Action of Hg<sub>2</sub>Cl<sub>2</sub> on alkaloid salts. 556—7, Nicotine in tobacco smoke. 564, Recovered diphtheria anatoxin. 568, Separation of sexual hormones. 568—9, Vitamins (various). 571, Constituents of Cascara sagrada extract. Odorous substances of green tea. Essential oils in desert plants. 573, Prep. of pollen extracts.

## PATENTS.

Stabilised egg products [cosmetics]. W. KRIT-CHEVSKY, B. R. HARRIS, and C. J. BECKERT (U.S.P. 1,924,947, 1,924,972, and 1,924,990, 29.8.33. Appl., [A, c] 8.4.29, [B] 15.4.29).—Cosmetics for external application comprise egg material stabilised by (A) glycols, polyglycols, their ethers or esters (ethylene or diethylene glycol), (B) monohydric alcohols, their ethers or esters (Pr<sup>8</sup>OH), and (c) trihydric alcohols, their ethers or esters (glycerol and derivatives), and a sulphonated oil. H. A. P.

Pretreatment of fish livers for subsequent production of medicinal oil. A./S. FERROSAN (B.P. 409,182, 11.8.33. Denm., 13.5.33).—Deterioration of the fat-sol. vitamins is prevented by storing the rinsed fresh livers in an aq. solution of a  $H_2O$ -sol. fat solvent, e.g., 1:3 COMe<sub>2</sub>-H<sub>2</sub>O. E. L.

Anæsthetic composition. S. D. GOLDBERG, ASST. to NOVOCOL CHEM. MFG. CO., INC. (U.S.P. 1,924,685, 29.8.33. Appl., 6.12.30).—A solution of an anæsthetic (I), e.g., procaine hydrochloride, a vaso-constricting material (II), e.g., adrenaline, a weak acid to dissolve (II), a buffer [compatible both with body-fluids and (I)] to maintain the  $p_{\rm H}$  at 5.7—7.0, and an anti-oxidising agent to preserve (II), e.g., NaHSO<sub>3</sub>, is specified.

Preparation of diphenylphenolphthalein [2-hydroxydiphenylphthalein] and substitution products. S. E. HARRIS and W. G. CHRISTIANSEN, ASSTS. to E. R. SQUIBB & SONS (U.S.P. 1,922,240, 15.8.33. Appl., 23.5.31).—Interaction of  $2 \cdot C_6 H_4 Ph \cdot OH$  with phthalic anhydride and  $H_2SO_4$  at 130—140° gives diphenylphenolphthalein [ $Br_2$ -, m.p. 110—111°, and ( $NO_2$ )2derivative, m.p. 135°]. H. A. P. Condensation products of cinchona-bark alkaloids and thiouram sulphides. [Inhibitors for metal-pickling baths.] R. E. LAWRENCE, Assr. to GRASSELLI CHEM. Co. (U.S.P. 1,923,056, 15.8.33. Appl., 22.11.32).—Cinchona alkaloids (quinoidine) are heated with thiouram sulphides (tetramethylthiouram mono- or di-sulphide) at  $> 100^{\circ}$  until reaction is complete.

H. A. P. Aldehyde-amine-cinchona-bark alkaloid (A) con-

densation product, (B) inhibitors, [for metal-pickling baths]. R. E. LAWRENCE, ASST. to GRASSELLI CHEM. Co. (U.S.P. 1,923,057, and 1,923,055, [ $\triangle$ , B] 15.8.33. Appl., [ $\triangle$ ] 22.11.32, [B] 19.12.31).—( $\triangle$ ) Aldehyde-amines and cinchona alkaloids (quinoidine) are condensed by heating together at > 100° (130—140°). (B) The products are used as inhibitors in acid pickling baths for metals.

H. A. P.

Alkylated phenols. Amylcresols. Ozonides.— See III. Packaging cod-liver oil.—See XII.

# XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Manufacture of photographic films. J. EGGERT (Chem. -Ztg., 1934, 58, 397—400).—A review of recent advances.

Sensitive materials for aërial photography. L. P. CLERC (Bull. Photogrammétrie, 1934, 4, 1-6).—Emulsions (I) should be fast and of high contrast, and give fine-grain images. Panchromatic (I) are recommended and a pale-yellow filter should always be used. The use of thin metal plates as (I) supports is suggested, printing being done by projection with an epidiascope. The differentiation of objects of similar brightness by taking two pictures with different colour-sensitive (I), or with different colour filters, is suggested. J. L.

Choice of emulsions for aërial photography. M. LEBELLE and G. CORDONNIER (Bull. Photogrammétrie, 1934, 4, 7—19).—The difficulties of obtaining sufficient contrast are discussed, with regard to the various types of emulsions and filters available. J. L.

Calculation of the exposure for photographic negatives using artificial sources of light. J. A. M. VAN LIEMPT (Rec. trav. chim., 1934, 53, 471-475).—A formula has been deduced which expresses the time of exposure for a given source of light, such as a glow lamp, as a function of the distance of the source (I), the sensitivity of the plate (II), and the aperture of the lens (III). For a given flashlight a relation has been found between (I), (II), and (III). Both formulæ are in satisfactory agreement with experiment. H. S. P.

New technique of photographic development, with regard to the various characters of negative emulsions. F. HAGELSTEIN (Pharm. Zentr., 1934, 75, 253-257, 270-273).—The properties of various kinds of emulsions are discussed. The author's two-stage method of fine-grain development is described, using first a quinol-glycine developer, and then a  $Na_2CO_3$ - $Na_3PO_4$  bath. J. L.

Photographs etc. and textile chemistry.-See V.

See also A., May, 496, Effect, on the latent image, of post-heating of gelatin. 504, Electron diffraction photographs.

E. H. S.

## PATENTS.

Sensitisation of photographic emulsions. KODAK, LTD. From EASTMAN KODAK Co. (B.P. 408,570, 30.6.32).— $\psi$ -Cyanines derived from 2-iodoquinoline alkylo-salts and 2-methyl-oxazole, -thiazole, or -selenazole alkylo-salts (of. B.P. 408,569; B., 1934, 537) are claimed as sensitisers. H. A. P.

Preparation of light-sensitive layers on foils of regenerated cellulose. J. KARAFIAT and J. CERNY (B.P. 409,134, 18.4.33. Ger., 18.4.32).—An aq. solution of a  $H_2O$ -sol. Ag or Fe salt, without precipitant, developing agent, or colloid, is dried on the foil, which is stretched flat under tension during the drying operation. J. L.

Preparation of gelatin blanks [for photographic films]. L. T. TROLAND, Assr. to TECHNICOLOR, INC. (U.S.P. 1,924,890, 29.8.33. Appl., 12.6.29).—The gelatin (I) layer is made containing  $K_2Cr_2O_7$  (e.g., 5%), and before it is completely set (while still moist) is treated with AcOH or citric acid, and then hardened by heating to 32—38°. Alternatively, the celluloid base may first be treated with, e.g., 10% alcoholic AcOH solution, and the dichromated (I) coated on afterwards. The characteristic acidity of the layers obtained is especially effective for the accurate transference and reproduction of dyes such as Fast Red S. J. L.

Restraining dye diffusion [in gelatin films]. L. T. TROLAND, ASST. to TECHNICOLOR, INC. (U.S.P. 1,923,043, 15.8.33. Appl., 24.5.28).—Diffusion of dyes after their application to gelatin surfaces is restrained by bathing the film in an aq. solution of  $\text{CuSO}_4$  ( $\frac{1}{2}$ %), containing preferably a little AcOH (5%), after the dye application. If three colours are used, the treatment is repeated after the application of the second dye. J. L.

Methods of and sensitive materials for photographic printing. N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 409,164, 6.7.33. Ger., 13.7.32).—Printing material which is sensitive only to ultra-violet light is made by using a mixture of a phenol (I) (other than an aminophenol), e.g., pyrocatechol,  $\alpha$ -naphthol, quinol, with a colourless oxidising compound, e.g., HNO<sub>3</sub>, KNO<sub>3</sub>, collodion solution, etc., applied to a suitable base. The carrier of the negative printed must be transparent to ultra-violet light, being, e.g., "Cellophane." Images of various colours are obtainable. After exposure the (I) is washed out with H<sub>2</sub>O. J. L.

Photographic gelatin relief. L. T. TROLAND, Assr. to TECHNICOLOR, INC. (U.S.P. 1,923,764, 22.8.33. Appl., 30.1.31).—An exposed emulsion is partly developed with a non-hardening developer, then treated with a hardening developer, and finally bleached and hardened with a selectively hardening solution (CrO<sub>3</sub>). The combined advantages of both methods of hardening are claimed. Details of suitable formulæ etc. are given. J. L.

Manufacture of photographic pictures [colloid reliefs]. C. ROEHRICH, ASST. to SILFREE A.-G. (U.S.P. 1,922,982, 15.8.33. Appl., 9.2.31. Ger., 14.2.30).— The incompletely hardened (though fully exposed) parts of an exposed dichromated gelatin layer are hardened through by first moistening the film (by spraying, bathing, etc.) and then passing the film for > 1 min. over heated rollers at 40—60° ( $> 100^\circ$ ). The unexposed, swollen parts may be coloured on the surface with one dye (from a colouring roller), and by imbibition the inner layers may be dyed with another colour. A third colour may be applied to the fully hardened parts. The heat-treatment should be applied before or directly after the colouring processes. J. L.

**Production of multi-colour photographs and** kinematograph pictures. B. GASPAR (B.P. 408,991, 18.7.32. Ger., 20.7.31).—Three-coloured emulsion layers are superimposed on one base, the top (a), middle (b), and bottom (c) layers being dyed yellow, purple, and blue-green, respectively, and sensitive to (a) blue and green, (b) green, (c) infra-red. Colour pictures are made on these films by printing from a coloured master-image, or from ordinary colour-selection negatives with appropriate colour filters. The dyes are destroyed or dissolved at the non-image portions. Details are given of suitable dyes, spectral divisions, etc. The process as applied to two-colour pictures is also described. [Stat. ref.]

J. L.

## Cyanine dyes.—See IV.

## XXII.—EXPLOSIVES; MATCHES.

# PATENTS.

Manufacture of military propellent explosives. E. WHITWORTH, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 408,659, 14.9.32).—Irregularity and difficulty in ignition of granular cordite M.D., due to exudation of mineral jelly at high temp., is overcome in manufacture by increasing the speed of the incorporator blades to 20 and 10 r.p.m., respectively, the temp. of the dough being maintained at  $\geq 30^{\circ}$ . After incorporation, the dough is allowed to mature for  $\lt$  40 hr. and then further incorporated for 1 hr. W. J. W.

Production of inulin nitrate. W. DE C. CRATER, Assr. to HERCULES POWDER CO. (U.S.P. 1,922,123, 15.8.33. Appl., 6.8.31).—Inulin nitrate, prepared by nitrating inulin with  $HNO_3$ - $H_2SO_4$ , softens at 90°, has m.p. 102°, and decomposes at 110°. It is not very sensitive to shock and has a flash point of 228—230°. It is sol. in COMe<sub>2</sub>,  $Et_2O$ -EtOH, conc.  $H_2SO_4$ , and EtOAc. It may be used as a sensitiser in dynamites and as a constituent of detonator compositions and, being compatible with nitrocellulose and nitro-starch, it may be used in lacquers. W. J. W.

NO<sub>2</sub>-compounds.—See III. Ni-Au [bridge] wire. See X. Pyroxylin composition.—See XIII.

# XXIII.—SANITATION; WATER PURIFICATION.

Semi-micro-method for determination of carbon monoxide in air. L. W. WINKLER (Z. anal. Chem., 1934, 97, 18—26).—250 c.c. of air are exposed for 4 hr. to 10 c.c. of aq. PdCl<sub>2</sub> (0·1% Pd + 5% KBr), 2 c.c. of CCl<sub>4</sub> are added, and then 2 c.c. of 0·02N-KBrO<sub>3</sub> (I) followed by 5 c.c. of 10% HCl + 1 c.c. of saturated aq. I. Br converts reduced Pd into PdBr<sub>2</sub>. Excess Br is back-titrated with 0·02N-Na<sub>3</sub>AsO<sub>3</sub>. The vol. of (I) used × a factor varying from 1·2 in absence of H<sub>2</sub> to 0·90 if H<sub>2</sub>: CO = 10:1 gives the CO in pts. per 1000. Unsaturated hydrocarbons must first be removed by Br-H<sub>2</sub>O, followed by NaOH. Aq. PdCl<sub>2</sub> on quant. filter paper detects 0·02% of CO; 0·1% produces a

British Chemical Abstracts-B

visible colour within 5 min.  $SO_2$ ,  $C_2H_4$ ,  $C_2H_2$ , etc. interfere and must be removed. J. S. A.

Lime sludge [in water softening] and its disposal. H. T. CAMPION (J. Amer. Water Works Assoc., 1934, 26, 488—494).—Lime sludge produced in the softening of Grand River  $H_2O$  at Grand Rapids, Mich., has for many years been returned to the river below the intake. In recent periods of low flow it has been drawn back towards the intake and has caused disagreeable tastes. Various other methods of disposal have been considered but rejected owing to cost. An attempt is being made to market the dry product for agricultural use. C. J.

Design and care of rapid sand filters. A. B. MORRILL and W. M. WALLACE (J. Amer. Water Works Assoc., 1934, 26, 445—460).—The size of the material forming the upper surface of the filter medium of rapid sand filters is the most important factor in determining efficiency. It should be carefully adjusted to secure the max. rate of operation which will effectively remove turbidity, colour, odour, and taste. Sterilisation is not primarily a function of such filters, but requires separate treatment. C. J.

Chemical action between filter media and hot alkaline waters. H. G. TURNER and G. S. SCOTT (J. Amer. Water Works Assoc., 1934, 26, 527-531).— Under like conditions the initial rate at which  $SiO_2$  is dissolved from Ottawa sand by NaOH solutions is about 10 times that at which it is dissolved from Anthrafilt (I) (a filter medium containing 3.6% SiO<sub>2</sub>). In the case of SiO<sub>2</sub> sand the dissolution rate (R) increases up to a max, of 4 times its initial rate before it starts to decrease, whereas in the case of (I) R immediately begins to decrease and reaches zero exponentially. C. J.

Modern water-chlorination practice. L. H. ENSLOW (J. New England Water Works Assoc., 1934, 48, 6-22).—A review. C. J.

Chlorine-fixing velocity [in water purification] and bactericidal chlorine quantity. G. NACHTIGALL and M. ALI (J. Amer. Water Works Assoc., 1934, 26, 430—444).—The rate at which  $Cl_2$  is fixed is a more important sterilisation factor than the total  $Cl_2$  demand. This rate varies considerably in different waters, so that it is not always possible to ensure sterility by the addition of 0.1 or 0.2 p.p.m. of  $Cl_2$  in excess of the total demand. C. J.

Experiments on the Newton water and treatments for the control of corrosion. F. W. GILCREAS (J. New England Water Works Assoc., 1934, 48, 105—116).—The H<sub>2</sub>O supply from Newton, Mass., is drawn from ground-H<sub>2</sub>O supplemented by infiltration from a neighbouring river. It is of good sanitary quality, but owing to its O<sub>2</sub> and CO<sub>2</sub> contents readily corrodes the mains. As a result of experimental work described, this objection has been largely removed by a gradual increase in the  $p_{\rm H}$  from the normal 6·2 to 7·6—8·0. Complaints of green stains due to Cu have ceased and the length of life of brass pipe is expected to be considerably prolonged. C. J.

Bleaching clays. J. R. BAYLIS (Water Works and Sewerage, 1933, 80, 287–288).—The odour-removing power is < 2% of that of activated C. When used with  $Al_2(SO_4)_3$  the clay assists the formation of a rapidly settling ppt. CH. Abs.

Radium poisoning in water. L. B. LOEB (J. Amer. Water Works Assoc., 1934, 26, 461-476).— Ra is a cumulative poison the complete ultimate elimination of which from the body is impossible and  $2 \times 10^{-6}$  g. of which are known to be fatal. Detection is difficult owing to its delayed action causing death by secondary effects. No cases are on record due to drinking natural radioactive H<sub>2</sub>O, but attention is called to the danger arising from the persistent use of certain much-advertised radioactive preps. C. J.

Improvements on Winkler's method for determining oxygen in sea-water. Z. NAKAI (Bull. Fishery Exp. Sta. Gov. Gen. Chosen, 1933, D, No. 3, 25-42, 43-48, 49-62).-100-c.c. samples obtained by dissolving  $Mn(OH)_2$  in HCl, and a table for converting observed vals. into  $O_2$  vals., are used. CH. ABS.

# Corrosion of brass in H<sub>2</sub>O.-See X.

See also A., May, 497, Prep. of distilled  $H_2O$ . 500, Determination of F' in natural waters. 505, "Atmos" distillation apparatus for  $H_2O$ .

#### PATENTS.

Substance for indicating the exhaustion of gaspurifying masses and its use [in respirators]. O. NEUMANN, ASST. to DEUTS. GASGLÜHLICHT-AUER GES. M.B.H. (U.S.P. 1,925,905, 5.9.33. Appl., 21.9.29. Ger., 17.11.28).—To the later parts of an absorbent in a gas mask is added Mg nitride, which will react with CO and warn the wearer by the odour of NH<sub>3</sub> that the absorbent is nearing exhaustion. B. M. V.

Sewage-treating apparatus. J. F. SKINNER, Assr. to DORR Co., INC. (U.S.P. 1,925,679, 5.9.33. Appl., 21.10.29. Renewed 15.2.32).—A shallow clarifier (C)is superposed on an anaërobic digester (D); the inlet to and overflow from C are both circumferential at the opposite ends of a diam. The sludge is raked over the flat bottom of C to a central outlet and is distributed in the upper part of D by arms on the same shaft, and after digestion raked along the bottom of D to a central outlet. The only other outlets from Dare circumferential ones for gas. B. M. V.

Garbage incinerator. W. L. and H. L. McLAUGH-LIN (U.S.P. 1,925,875, 5.9.33. Appl., 3.9.31).—A steeply inclined rotary kiln is described. The claims relate to the method of taking the large end-thrust, and other mechanical devices. B. M. V.

Treatment of liquids [waste water from sulphur mining]. W. JUDSON, ASST. to KOPPERS CO. OF DELAWARE (U.S.P. 1,924,861, 29.8.33. Appl., 10.3.31). —Waste  $H_2O$  containing Ca and S compounds is treated with a small amount of  $Na_2HPO_4$  and passed down a packed tower up which hot flue gases are circulated to expel  $H_2S$  and saturate the  $H_2O$  with  $CO_2$ , thereby forming Ca(HCO<sub>3</sub>)<sub>2</sub> and avoiding deposition of scale. A. R. P.

Gas-works' effluents.—See II. Organo-Pb compounds.—See III. Pasteurising apparatus.— See XIX.