# BRITISH CHEMICAL ABSTRACTS

## **B.**—APPLIED CHEMISTRY

SEPT. 25 and OCT. 2, 1936.\*

## I.—GENERAL; PLANT; MACHINERY.

Welded pressure vessels for low temperatures. E. POHL (Tech. Ind. Schweiz. Chem.-Ztg., 1935, 18, 91; Chem. Zentr., 1935, ii, 914).—The use of arc-welded Ni-steel (0.3% C, 0.6% Mn,  $\leq 0.25\%$  Si,  $\leq 2.5\%$  Ni) pressure vessels for liquid air plants etc. is described.

J. S. A.

Pendulum as a source of energy for plasticity measurements. I. WILLIAMS (Ind. Eng. Chem. [Anal.], 1936, 8, 304—306).—A pendulum of known potential energy is used to deform rapidly the plastic material. The damping effect is a measure of the energy expended on deformation. The elastic recovery following rapid deformation is much > that obtained with the parallel-plate instrument. Tests on rubber are described. E. S. H.

The Schiebler filter. H. ROTHERT (Fette u. Seifen, 1936, 43, 106–109).—Descriptive. The filter has been used for separating bleaching earths from oils.

F. C. B. M.

Use of solubility data to control the deposition of sodium sulphate or its complex salts in boiler waters. W. C. SCHROEDER, A. A. BERK, and E. P. PARTRIDGE (Amer. Soc. Test. Mat., Preprint 105, June, 1936).—Curves showing the solubility of  $Na_2SO_4$ from 150° to 350° in H<sub>2</sub>O and in various complex solutions representative of conc. boiler waters are given. If further investigation confirms the view that  $Na_2SO_4$ inhibits embrittlement of boiler steel, these curves will define the conditions which should be maintained.

R. B. C.

Boiling temperatures of solutions. I. A. TISCH-TSCHENKO (Sovet. Sachar., 1933, No. 11—12, 31—33).— The calculation of the b.p. of solutions, in connexion with the calculation of the heating surface of evaporators, is described. CH. ABS. (e)

Chemical works pumping and acid handling. E. A. REAVELL (Chem. & Ind., 1936, 586-597).—The different types of pumps and other apparatus available for handling corrosive liquids are discussed. D. K. M.

Importance of emulsions as forms of distribution. H. SCHRADER (Angew. Chem., 1936, 49, 473-475).—A survey of the importance of emulsions in foodstuffs, medicines, cosmetics, and in various industries. J. W. S.

Flue-gas corrosion. E. GENTE (Wärme, 1936, 59, 307–311).—The temp. of flue gas from S-containing coal should not be allowed to fall below the dew point (D), since the SO<sub>3</sub> contained therein forms a corrosive acid in presence of condensed H<sub>2</sub>O, even at relatively high temp. The presence of SO<sub>3</sub> slightly raises D, but the effect is negligible when the coal contains < 2%

S. A method for calculating the D of any flue gas by means of a total heat-dryness fraction diagram for a flue gas-H<sub>2</sub>O vapour mixture is described. Since the use of steam blowers may cause a considerable increase in the H<sub>2</sub>O content of the flue gas with a consequent rise of D the temp. of the gas should not be allowed to fall to the extent permissible when employing compressed air blowers. Automatic D indicators are described.

R. B. C.

New packing material [Sinterite] for socket joints. H. Vogr (Gas- u. Wasserfach, 1936, 79, 592— 594).—Sinterite prepared by heating at 1200—1350° in a reducing atm. an Fe powder poor in C or Fe-O compounds. The plastic Fe formed is coated with bitumen to prevent corrosion. Sinterite compares favourably (tests described) with other packing materials. J. W.

Wood for apparatus.—See IX. Cast irons for plant equipment. Steels for autoclaves.—See X.

#### PATENTS.

Manufacture of [heat-]insulating materials. COUTTS & CO., and F. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 446,442, 22.11.34).—Solutions of condensation products of ureas and aldehydes, *e.g.*, urea and  $CH_2O$ , are converted into foam-like masses with brisk agitation, preferably in presence of a foaming agent, *e.g.*, alkylnaphthalenesulphonic acids, *N*-methyloleyltaurine, and in presence of traces of acid, *e.g.*, SO<sub>2</sub>, followed by hardening, which is best conducted at about 60° in moist air or under H<sub>2</sub>O at 60—80°.

A. W. B.

Gas converter and cleaner.-See II.

## II.—FUEL; GAS; TAR; MINERAL OILS.

Gas evolution and rate of face advance. III. W. H. N. CARTER and A. HUDSON (Trans. Inst. Min. Eng., 1936, 91, 285-301).—The effect of roof condition and barometric changes on rate of gas emission is discussed. H. C. M.

Influence of method of preparation and fuel requirements on production of fine coal washings. O. SCHÄFER (Glückauf, 1935, 71, 437-445; Chem. Zentr., 1935, ii, 949).—The production of coal of specified ash, H<sub>2</sub>O content, etc. is discussed. J. S. A.

Contraction of brown coal on drying. B. G. ŠIMEK (Mitt. Kohlenforschungsinst. Prag, 1935, 2, 254— 261).—The average linear contraction of the coal substance between the moist and air-dried conditions is determined by measuring the distance between characteristic fixed points in X-ray photographs of coal sections, but both parallel and perpendicular to the bedding

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

plane. The contraction calc. from these data is approx. = vol. of  $H_2O$  removed from the coal. The capillary space left free from  $H_2O$  is very small; *e.g.*, in a mature brown coal from North Bohemia it is about 1.5%.

Measurement of coal temperature in hoppers of brown-coal briquette presses. O. Schöne (Braunkohle, 1935, 34, 321—327; Chem. Zentr., 1935, ii, 949). —Under normal conditions the temp. cannot rise high enough for spontaneous inflammation. J. S. A.

Production, consumption, and some chemical properties of Ube coal. A. TAWARADA (J. Fuel Soc. Japan, 1936, 15, 69—71).—Proximate and ultimate analyses for 4 seams of the Ube coalfield are listed, the coals have a high ash content and low calorific val. Their cokes have a very high electrical resistance, and are very reactive towards  $CO_2$ . H. C. M.

Colloidal fuel. D, BROWNLIE (Ind. Eng. Chem., 1936, 28, 839–842).—The various processes for preparing colloidal fuel are described. E. G. H.

Composition of phenol extracts of mineral coal. K. I. SISKOV and A. A. USCHAKOVA (Kolloid-Z., 1936, 76, 213-216).—Experiments on the methylation of the extracts and their adsorption capacity for Ba(OH)<sub>2</sub>, as a means of characterisation, are described. E. S. H.

Exhaustive chlorination of a bituminous coal. J. F. WEILER (J. Amer. Chem. Soc., 1936, 58, 1112— 1114).—The finely-divided dry coal (I) (Pittsburgh seam) in SbCl<sub>5</sub> with Cl<sub>2</sub> at 200°/6 days gives CCl<sub>4</sub>, C<sub>2</sub>Cl<sub>6</sub>, C<sub>6</sub>Cl<sub>6</sub>, and (after prolonged action of 20% HCl) a nonvolatile residue (A), containing more N and O (probably owing to hydrolysis of the more active Cl) than (I) and about 85 and 40%, respectively, of the original C and H. A is separated successively into Et<sub>2</sub>O- (21), CHCl<sub>5</sub>- (22), and PhCl-sol. fractions (18%), each having M about 160 [f.p. in o-C<sub>6</sub>H<sub>4</sub>(OH)<sub>2</sub>]; the residue has M about 250 (mean). It is concluded that approx. 85% of the C in (I) is part of a large condensed ring system containing the N of (I) as (probably) an integral part. H. B.

Smoke index : a quantitative measurement of smoke. R. J. PIERSOL (Dept. of Registration and Education, Illinois State Geol. Survey Rept. Invest. No. 41, 1936, 31-113).- A method for measuring the total quantity of smoke produced by the combustion under standard conditions of a weighed sample of fuel is described. The fuel sample is burned at a definite temp. in an electric muffle furnace with a specified supply of air. All the smoke produced is drawn through an absorption tube at a const. rate and the smoke density is measured by its absorption of a beam of light of known intensity passing lengthwise through the tube. The amount of light absorbed is determined by a Weston photronic cell and a galvanometer. The smoke index is the average % absorption of light  $\times$  the time (sec.) of smoke emission of 1 g. of fuel.  $C_{10}H_8$  is used as the standard fuel. Results obtained with various coals and coal briquettes are tabulated. A linear relationship between the smoke index and the % volatile matter was established in the case of Illinois and West Virginia bituminous coals. R. B. C.

Chemical nature of coke. H. L. RILEY (Gas World 1936, 105, Coking Sect., 17-22).-The amorphous forms of C are more reactive to O2 and CO2 than graphite (I), but the order of activity is inverted on oxidation with a solution of  $H_2CrO_4$  in  $H_3PO_4$  (cf. B., 1935, 789; A., 1936, 178, 689). Attention is directed to the similarity between (I) and CPh3 in structure and in chemical behaviour. The amorphous forms of C, e.g., charcoal, coke, soot etc., are regarded as (I) with variable amounts of H stably linked between the hexagon planes. The presence of such H retards the penetration of reagents to the interplanar surfaces. With respect to reactions dependent on such interpenetration, e.g., oxidation with  $H_2CrO_4 + H_3PO_4$ , therefore, (I) is more reactive than coke etc. Other evidence supporting this view of coke structure is discussed. A. B. M.

[Wet oxidation of carbon.] NORTHERN COKE RESEARCH COMMITTEE (Ann. Rept., No. 7, 1935, 10 pp.). -Investigation of the reaction between H2CrO4 in syrupy H<sub>3</sub>PO<sub>4</sub> and various forms of C has shown that H<sub>3</sub>PO<sub>4</sub> plays an important part in determining the reaction velocity and that the method offers a simple means of determining the chemical nature of C present in a sample. Samples of coal were carbonised both rapidly and slowly over a range of temp. and the properties of the resulting cokes examined, particularly with regard to their susceptibility to wet oxidation. The results show that there is a marked tendency for cokes from coals of high rank to be less susceptible to wet oxidation than those from low-rank coals. The experimental cupola has been employed to study the effect of blending coals on the properties of the cokes obtained. R. B. C.

Coking tests with stored coal dust. P. MICHAELIS (Glückauf, 1935, 71, 413—423; Chem. Zentr., 1935, ii, 628).—On storage, dust of "fat" coal slowly acquires the coking characteristics of a stack coal and may be used as such for admixture with gassy coking coal with a tendency to swell. H. N. R.

Dennstedt method for ultimate analysis of coke. B. G. ŠIMEK and F. COUFALÍK (Mitt. Kohlenforschungsinst. Prag, 1935, 2, 57-61).—A description is given of the Dennstedt combustion tube and of the procedure adopted for the ultimate analysis of coke. The method is claimed to be as accurate as, but simpler and more rapid than, that of Schulz. Results obtained by the two methods are compared. R. B. C.

Clinker formation in small underfeed stokers. Use of addition agents to promote clinkering of refractory ashes. R. A. SHERMAN and E. R. KAISER (Battelle Mem. Inst. Inf. Bull. No. 1, 1936, 10 pp.).— The effectiveness of Na metasilicate, Portland cement, Na<sub>2</sub>SO<sub>4</sub>, Na<sub>2</sub>CO<sub>3</sub>, Na silicate, and borax as agents for promoting the clinkering of coal ash was not as great as might be expected from the chemical compositions of the resultant mixtures. The two last-named were the most effective. The cost of treatment may be as high as 50 cents or more per ton of coal. R. B. C.

Alinement charts for calculating the calorific values [of fuels]. W. SCHREIBER (Wärme, 1936, 59, 324-325).—Four nomograms based on previously published data are given. The first enables gross

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calorific vals. (C) to be calc. for any desired  $H_2O$  and ash content when the C for a particular  $H_2O$  and ash content is known. The second and third are for calculating lower C, whilst the fourth is for calculating C from the ultimate analysis of the fuel. R. B. C.

Electro-graphite. Preparation, properties, and applications. R. STRAUSS (Chem.-Ztg., 1936, 60, 661-663).—A review. E. S. H.

Perfect gasification of coal. T. NAMIKAWA (J. Fuel Soc. Japan, 1936, 15, 73-74).-Two types of complete-gasification plants for production of water-gas low in CO2, N2, and hydrocarbons, as required for synthetic and hydrogenation purposes, are briefly described. One consists of an upper and a lower gasification zone, the low-temp, carbonisation occurring in the upper zone being induced by the sensible heat of the blow gases, which are mixed with the carbonisation gases. A certain amount of mixed hydrocarbon and water-gas is introduced by steam into the gasification zone to be decomposed and also to reduce  $CO_2$ . In the second plant, two such generators are combined and the watergas from one is passed into the gasification zone of the other. Steam is injected up and down in each generator alternately. Tables are included showing typical results obtained with both plants. H. C. M.

Corrosion problem in a city [gas] plant. C. F. TURNER (Nat. Gas, 1935, 16, No. 7, 10-12).—A discussion. CH. Abs. (e)

Coal gas as a basic product for the production of hydrogen. V. A. KARSHAVIN and A. G. LEIBUSCH (J. Chem. Ind. Russ., 1936, 13, 455-464).—Known processes for producing H<sub>2</sub> from coal gas are discussed. R. T.

Two-stage Thylox process for hydrogen sulphide removal [from gas]. A. R. POWELL (Gas J., 1936, 215, 277–280).—The chemical reactions occurring in the Thylox process are discussed. In the singlestage process only 90–95% of the  $H_2S$  can be economically removed. By scrubbing the gas in a separate washer with the very active, fresh make-up solution of Na<sub>2</sub>CO<sub>3</sub> and As<sub>2</sub>O<sub>3</sub>, complete removal of the residual  $H_2S$  is effected. The make-up solution, after it has been used for this secondary absorption, is then fed into the regular Thylox solution as the usual make-up reagent. Typical operating data and results obtained with a commercial plant are given. H. C. M.

Susceptibility of asphalts to temperature change. H. E. SCHWEYER, C. E. COOMBS, and R. N. TRAXLER (Proc. Amer. Soc. Test. Mat., 1936, 36, Preprint 87, 8 pp.; Road Abs., 1936, 3, No. 314).—The log  $\eta$ -temp. graph for asphaltic materials is a straight line at processing temp. where  $\eta = 1$ —5 poises and also at service temp. (15—35°). A method of expressing susceptibility is suggested, based on the change in  $\eta$  (%) per 1° temp. rise, and is called the asphalt viscosity index. Data are given over the two temp. ranges for various paving asphalts. The susceptibility of a bitumen-filler mixture is nearly the same as that of the bitumen alone.

T. W. P.

Rheological properties of asphalt bitumens and working hypotheses on the inner structure of these products. J. P. PFEIFFER and P. M. VAN DOORMAAL (Kolloid-Z., 1936, 76, 95-111).--Bitumens show all grades of flow behaviour, from flow in accordance with Poiseuille's law to plastic flow, depending on the degree of peptisation of the disperse phase. Similarities in the properties of bitumens and typical gels are discussed.

Bitumens and humic acids. G. STADNIKOV (Brennstoff-Chem., 1936, 17, 270).—The substance extracted from brown coal by  $C_6H_6$ -EtOH, in addition to the bitumen (Voitova, B., 1936, 676), was probably not humic acid but a resin. A. B. M.

Composition of xylene from coal tar and petroleum. N. KISHNER and V. KRASOVA (J. Gen. Chem. Russ., 1936, 6, 748—756).—Coal-tar xylene contains o-3-5, m-68-70, and p-xylene 18—19, and PhEt 5-7%, whilst xylene from petroleum contains o-7-9, m-12-13, and p-xylene 36—38, PhEt 26—28, and nonaromatic hydrocarbons (including 1:3-dimethyl- and 1:2:4-trimethyl-cyclohexane) 15—18%. R. T.

Distillation of coal-tar phenols. H. G. SHATWELL (Chem. & Ind., 1936, 573—576).—Considerable improvement in efficiency of fractionation of tar acids can be effected by replacing the batch system of distillation using ring-packed columns by continuous stills with bubbling columns. A. B. M.

Hydrogenation of creosote oil. P. D. Scorr (Gas World, 1936, 105, Coking Sect., 102—106).—Hydrogenation was effected in a  $2\frac{1}{2}$ -litre, gas-heated converter. Each charge consisted of 250 c.c. of creosote (d 1.050, b.p. 220—340°; tar acids 9.8, bases 6, C<sub>10</sub>H<sub>8</sub> 7%), the initial pressure of H<sub>2</sub> was 84—110 atm., the reaction temp. 500—510° (reached in 2 hr. and maintained for a further 2 hr.), and max. pressure 177—225 atm. Catalysts employed were NH<sub>4</sub> molybdate or MoS<sub>3</sub>, supported on active charcoal, or on SiO<sub>2</sub> or Al<sub>2</sub>O<sub>3</sub> gel. The yields of recovered oil were 79.4—88.4 vol.-%, containing 38—55% of distillate to 195°. The light spirit had the composition : aromatic hydrocarbons 76.7, unsaturateds 3.5, paraffins and naphthenes 19.8%. The higherboiling fraction of the product could be further converted into spirit by repeated treatment. A. B. M.

Calculation of "equiviscous temperatures" of tar. G. BARR (Chem. & Ind., 1936, 576-577).--A method of constructing a suitable nomogram is suggested. A. B. M.

Nickel and its alloys in petroleum refining. D. R. W. MÜLLER (Petroleum, 1936, 32, No. 29, 3-6).-A description is given of the applications of alloys containing Ni in the construction of plant etc. for the petroleum industry. C. C.

Saponification values of asphaltic petroleum residues. Pressure-agitation method. J. H. BRUUN and L. W. CLAFFEY (Ind. Eng. Chem. [Anal.], 1936, 8, 255—256).—Modified procedure is recommended. E. S. H.

Gyro vapour-phase cracking process. K. KUDO (J. Fuel Soc. Japan, 1936, 15, 71-73).—The Gyro plant, the only vapour-phase cracking system in Japan, consists of 3 parts, the cracking (C), polymerising (P), and stabilising units (S). In C, the charge is fed to the middle of the fractionating column so that the

E. S. H.

raw material which is actually decomposed in the converter is the vapour of the fractionator bottom after separation of the heavy fraction in the evaporator. In P and S, the highly unsaturated cracked gas is first polymerised into liquid polymeride by heat and pressure, and the polymeride gasoline so produced is stabilised by heating at 200°/20 atm. Tables are given showing typical operating conditions, properties of the cracked distillates, and analyses of gas samples taken at various stages in the process. H. C. M.

Production of gasoline by polymerisation of gases. R. FUSSTEIG (Petroleum, 1936, 32, No. 29, 6—8). —Gases from vapour-phase cracking processes are polymerised at 480—540°/42—58 atm. An increased yield of gasoline is obtained by separating the gases of low mol. wt. (by an absorption process which removes gases of higher mol. wt.) and treating them separately at 700°/5 atm. Natural gas, containing a high proportion of saturated gases, must first be subjected to catalytic pyrolysis and then polymerised. C. C.

"Uni-Coil injection" process [for oils]. PETROL-EUM PROCESSES CO. (Petroleum, 1936, 32, No. 29, 1—2). —A process combining the reduction of the  $\eta$  of the treated oil with high-temp. cracking is described, for which special heat economy is claimed. C. C.

Photometric determination of iron in used engine oils. A. R. RESCORLA, E. M. FRY, and F. L. CARNAHAN (Ind. Eng. Chem. [Anal.], 1936, 8, 242—244). —The oil is ashed, the residue dissolved in HCl,  $NH_4CNS$ added, and Fe determined by means of a photometric colorimeter. The procedure is suitable for determining 0.00005-0.0015 g. of Fe. E. S. H.

Influence of water on the oxidation of hydrocarbons of high mol. wt. A. K. PLISSOV and E. MALEEFFA (Bull. Soc. chim., 1936, [v], 3, 1281–1290).— Oxidation of Grozni paraffin and solar oils is accelerated in presence of dehydrating agents such as  $CaCl_2$ ,  $CuSO_4$ , and  $Na_2SO_4$ ; when these are suspended in the oil, the acid val. of the oxidised oil is  $\leq$  that of a control. The rôle of  $H_2O$  in these oxidations is discussed. J. L. D.

Properties of peroxides formed during the oxidation of paraffin. A. K. PLISSOV (Bull. Soc. chim., 1936, [v], 3, 1274—1281).—Grozni paraffin when heated in O<sub>2</sub> at 140—160° forms peroxides (I) which are capable of oxidising KI and are fairly stable below 150°, but are quickly hydrolysed by acid and alkali and by H<sub>2</sub>O in presence of Ca naphthenate and Na stearate. (I) incapable of oxidising KI are also formed and are converted into the active (I) when heated for 1—2 hr. The acid val. of the oil increases as its (I) content diminishes. J. L. D.

Gasoline from ethylene by catalytic polymerisation. V. N. IPATIEV and B. B. CORSON (Ind. Eng. Chem., 1936, 28, 860).—1000 cu. ft. of  $C_2H_4$  yield 4.7 gals. of 204° end-point gasoline ( $C_8H_{18}$  no. 82) by polymerisation in presence of  $H_3PO_4$  at 520 lb. pressure. The rate of polymerisation is doubled by raising the temp. from 296° to 324°. The products at these two temp. were :  $C_2H_4$  recovered 13.7, 18.0; butene 0.7, 0.8; isobutane 11.6, 6.4; liquid polymeride 73.7, 74.5%. Susceptibility of gasolines to lead tetraethyl and aniline. C. O. TONGBERG, D. QUIGGLE, E. M. FRY, and M. R. FENSKE (Ind. Eng. Chem., 1936, 28, 792—794). —The Pb responses of different fractions of a gasoline measured by a Series 30B Ethyl Knock-testing engine vary considerably. Fractions containing  $\beta$ -methylpentane or n-C<sub>6</sub>H<sub>14</sub> are very responsive. Differences are believed to be due partly to S compounds. The increase in C<sub>8</sub>H<sub>18</sub> no. due to the addition of NH<sub>2</sub>Ph is practically linear for additions between 2 and 7%. When they are used together, NH<sub>2</sub>Ph acts independently of PbEt<sub>4</sub>. E. G. H.

Compressibility of butane-air mixtures below one atmosphere. F. W. JESSEN and J. H. LIGHTFOOT (Ind. Eng. Chem., 1936, 28, 870–871).—The additive rule for determining the compressibility of gas mixtures at low pressures does not hold for  $C_4H_{10}$  and air, deviations up to 23% being noted. E. G. H.

Smoke tendency of refined kerosene and its determination. J. B. TERRY and E. FIELD (Ind. Eng. Chem. [Anal.], 1936, 8, 293—295).—The flame height which can be tolerated without smoking is determined in a modified factor lamp. E. S. H.

Methane as propellent for automobiles. E. BÖRGER (Z. komp. flüss. Gase, 1934—1935, 31, 81—85; Chem. Zentr., 1935, ii, 950).—Compressed  $CH_4$  is as efficient as are the best liquid fuels for vehicular propulsion. The application of gaseous  $CH_4$  is discussed. J. S. A.

Liquid butane as motor fuel. Corrosion test methods. M. M. HOLM (Ind. Eng. Chem. [Anal.], 1936, 8, 299—300).—Qual. evidence of the presence of corrosive impurities is obtained by keeping the specimen in contact with mirrors of Cu and Ag on glass and observing the tarnish produced. E. S. H.

Testing the suitability of methyl alcohol for use in motor fuel blends. T. HAMMERICH (Oel u. Kohle, 1936, 12, 641-642).—The temp. at which a mixture of 100 c.c. of the MeOH with 100 c.c. of 80 vol.-% n-C<sub>7</sub>H<sub>16</sub> + 20% C<sub>6</sub>H<sub>6</sub> becomes clouded is recorded. The temp. varies from 21.60° for pure MeOH to 25.54° for MeOH containing 0.15 vol.-% of H<sub>2</sub>O. Addition of 1% of Bu<sup>g</sup>OH lowers the temp. by 1.9°. A. B. M.

Application of lignite derivatives as Diesel fuel. A. W. SCHMIDT (Braunkohle, 1935, 34, 49—53, 72—75; Chem. Zentr., 1935, ii, 633).—The influence of various properties of the fuel, and of engine design, on efficiency is discussed. Tests with creosote and paraffin admixtures indicate that cyclic compounds tend to inhibit combustion whereas straight-chain compounds promote it. H. N. R.

Vegetable oils and Diesel motors. M. GAUTIER (Rev. Combust. liq., 1935, 13, 129—136; Chem. Zentr., 1935, ii, 951).—Data are given for the (satisfactory) performance of vegetable oils as Diesel-engine fuels.

J. S. A.

New [German] regulations relating to the composition of motor fuels. K. R. DIETRICH and W. LOHRENGEL (Oel u. Kohle, 1936, 12, 637-641).—The regulations relating to the compulsory admixture of EtOH and MeOH with motor spirits are discussed,

E. G. H.

principally from the viewpoint of the stability of the mixture, as measured by the variation of the temp. of separation with the addition of small quantities of  $H_2O$ . A. B. M.

Diesel fuel specifications. C. H. BARTON (J. Inst. Petroleum Tech., 1936, 22, 508—514).—The significance of the tests normally employed in fuel specifications is discussed. The essential tests for a Diesel fuel of petroleum origin are  $\eta$  at 37.8°, 20°, or 50°, C-forming tendency, ignition quality (for high-speed engines), ash, and H<sub>2</sub>O. Pour point or  $\eta$  at low temp. may be required in special circumstances. C. C.

Different methods of assessing the ignition quality of Diesel fuels. F. H. GARNER (J. Inst. Petroleum Tech., 1936, 22, 503-507).—Recent work on the tating of Diesel fuels from the viewpoint of ignition quality is summarised. C. C.

Correlation of tests on ignition quality of Diesel fuels, carried out at Delft and Sunbury. G. D. BOERLAGE, J. J. BROEZE, L. J. LE MESURIER, and R. STANSFIELD (J. Inst. Petroleum Tech., 1936, 22, 455– 468).—Normal Diesel fuels, including cracked fuels, can be rated in terms of suitable mineral secondaries in almost any type of Diesel engine without serious risk that large differences will be found between one engine and another. C. C.

Modern testing of motor fuels for the high-speed type of automotive Diesel engine. A. HAGEMANN and T. HAMMERICH (J. Inst. Petroleum Tech., 1936, 22, 515-539).-In addition to the usual tests for Diesel oils, methods have been developed for filtering ability (difference in time of flow of 200 ml. of oil through a special apparatus, with and without a gauze filter), corrosion (of Cu and Zn), ignitability (using a singlecylinder Deutz engine), and coking properties (determining the asphalt and coke on a sample after heating at 155° in a bomb under 20 atm. air pressure). The effect of the fuel on the smoking point of the engine is important, but no entirely satisfactory method for determining the latter point has been described. C. C.

Lubrication : recent research. J. E. SOUTH-COMBE (J. Inst. Fuel, 1936, 9, 374—377).—A review.

Lubricants : some recent developments. J. S. S. BRAME (Proc. Inst. Automobile Eng., 1935—36, 30, 100—120).—A review covering petroleum refining, the production of lubricants from petroleum by hydrogenation, lubricants derived from coal, and extremepressure lubricants. R. B. C.

Determination of acidity in insulating oil. Use of the glass electrode in *n*-butanol. R. N. EVANS and J. E. DAVENPORT (Ind. Eng. Chem. [Anal.], 1936, 8, 287-291).—A potentiometric method, using the glass electrode in Bu<sup>a</sup>OH, is described. In absence of interfering substances, the precision is  $\pm 0.02$  c.c. of 0.01Malkali, E. S. H.

Temperature - sensitivity of highly viscous mineral oils. D. J. W. KREULEN (Chem. Weekblad, 1936, 33, 466–473; cf. B., 1935, 614, 888).—The changes in setting point and  $\eta$  resulting from preheating of the oil are not due to the presence of asphalts, since materials containing no asphalt, including honey, exhibit this phenomenon. Nevertheless, the presence of asphalts accentuates the effect. Preheated oils can be brought back almost to their original state by inoculation with untreated oil. Various mechanisms are suggested, but no complete explanation is offered. Simple retardation of crystallisation is no doubt an important factor where decrease in  $\eta$  occurs, whilst the formation of a network of fine crystals may account for the rise in  $\eta$  observed when the temp. of preheating is  $\gg 50^{\circ}$ . D. R. D.

Oil oxidation. R. W. DORNTE and C. V. FERGUSON (Ind. Eng. Chem., 1936, 28, 863—866).—The oxidation of lubricating oils unaffected by the reaction products is approx.  $\infty$  the partial pressure of O<sub>2</sub>. H<sub>2</sub>O and CO<sub>2</sub> but no peroxides are formed and inhibitors are practically without effect. Cu increases considerably the rate of oxidation but Fe, Sn, and Pb have only a slight accelerating influence. E. G. H.

Specific refraction of commercial paraffin waxes in liquid and solid states. J. M. PAGE, JUN. (Ind. Eng. Chem., 1936, 28, 856—859).—From measurements of  $n_p$  commercial paraffin waxes appear to be anisotropic uniaxial cryst. solids. Discrepancies between vals. for liquid and solid states are attributed to occluded air in the solidified waxes giving low vals. for the apparent d. E. G. H.

Gases for NH<sub>3</sub> synthesis.—See VII. Tars and bitumens. Asphalt pavements.—See IX. Lignite as fertiliser.—See XVI. Utilising cellulosic materials.—See XVIII.

#### PATENTS.

**Chamber ovens.** Dr. C. Отто & Co. G.M.B.H. (B.P. 448,861, 7.12.34. Ger., 14.5., 22.5., and 6.8.34).—The distillation gases are withdrawn not only from the gascollecting space (S) above the charge, but also through vertical ducts (D) made in the charge. D communicate with a horizontal gas-collecting channel (C) lying above the coking chamber. S communicates with D through regulable openings. C and S communicate with the hydraulic main through openings provided with dampers; certain of the D have apertures opening into S. The openings and dampers are so regulated that when suction is applied in the hydraulic main the pressure in S is maintained at atm. A. B. M.

**Regenerative coke oven.** W. REPPEKUS (U.S.P. 2,011,592, 20.8.35. Appl., 11.4.32. Ger., 16.4.31).— The coke-oven battery comprises alternate coking chambers and heating walls with vertical flues, below and parallel to which are the regenerators (R), below which again are channels (C) running longitudinally of the battery. C are arranged in two sets operable alternately for simultaneous separate inflow of fuel gas and air and concurrent outflow of waste gas. R are arranged correspondingly. The arrangement is designed to give uniform heating without individual control of the gas and air into the flues, and to avoid loss of combustion gas by leakage to the waste-gas channels. A. B. M.

Vertical coking retort bench. G. GOETZ, ASST. to L. KERN (U.S.P. 2,012,447, 27.8.35. Appl., 21.7.32).— The retorts are narrow, rectangular in cross-section, and slightly tapered upwards. They are heated by horizontal heating flues arranged along the two wider sides. The volatile products of carbonisation are withdrawn through a series of offtakes distributed vertically along the two narrower sides, and through offtakes in the top. Regenerators are provided for preheating the air for combustion. The retorts are arranged in sets of three which discharge into a common quenching chamber, whence the coke is delivered on to a discharge conveyor. A. B. M.

Refining of graphite. A. M. KENNEDY, Assr. to W. H. WELLER, JUN. (U.S.P. 2,012,684, 27.8.35. Appl., 21.4.34).—The conc. graphite ore is treated with aq.  $NH_4HSO_4$ , which dissolves the metallic impurities, e.g.,  $Al_2O_3 + 6NH_4HSO_4 = Al_2(SO_4)_3 + 3(NH_4)_2SO_4 + 3H_2O$ . The solution is regenerated by adding  $NH_3$  to ppt. the metallic impurities, separating the latter, evaporating, and reconverting the  $(NH_4)_2SO_4$  into  $NH_4HSO_4$  by heating. A. B. M.

Hydrogenation of carbonaceous materials. T. W. PFIRRMANN (U.S.P. 2,012,318, 27.8.35. Appl., 5.8.33. Ger., 10.8.32. Cf. B.P. 405,371; B., 1934, 391).—The material, e.g., coal, is heated with Zn and  $H_2O$  in a closed vessel under conditions such that the  $H_2O$  is decomposed by the Zn with the production of  $H_2$ , which converts the material into liquid products. The ZnO is reduced again to Zn by heating the residue with reducing gases; the Zn volatilises and is recovered and used again in the process. A. B. M.

Gas converter and cleaner of the liquid-contact type. J. F., F. W., and G. A. TECKEMEYER, Assrs. to L. L. LEWIS, C. BOSAU, C. MARQUIS, and N. R. WARD (U.S.P. 2,011,598, 20.8.35. Appl., 1.7.33).—An apparatus for saturating air or gas with the vapour of a volatile hydrocarbon comprises a lower chamber divided into a series of compartments (C) into which the liquid hydrocarbon and air are admitted, and an upper chamber (D) in which the saturated air or gas is dried and cleaned. C contains porous sheets suitably arranged to absorb the liquid and saturate the air therewith. In D the gas is passed through other porous sheets. A. B. M.

Production of gases for synthesis. E. T. OLSON, Assr. to CLEVELAND CLIFFS IRON CO. (U.S.P. 2,012,477, 27.8.35. Appl., 30.8.33).—Gases of high CO content, and free from catalyst poisons, are produced by distilling wood etc. and passing the gases evolved (except those produced during the first stages of the process) over C at a temp. sufficiently high to reduce the  $CO_2$ therein to CO. A. B. M.

Improving the odour and colour of tar acids. C. E. HARTWIG, ASST. to BARRETT Co. (U.S.P. 2,011,633, 20.8.35. Appl., 31.8.32).—The tar acids are dissolved in aq. alkali and treated, preferably at 50—90°, with  $< 1\frac{1}{2}$ % (calc. on the tar acids) of a gelatinous hydroxide of a heavy metal, *e.g.*, Fe(OH)<sub>3</sub>. The phenoxide solution is then separated from the hydroxide and the tar acids are liberated and recovered. A. B. M.

Breaking of [water-in-oil-type] petroleum emulsions. (A) C. H. M. ROBERTS, (B—F) C. N. STEHR, (G, I—K) M. DE GROOTE and B. KEISER, and (H) M. DE GROOTE and A. F. WIRTEL, Assrs. to TRETOLITE Co. (U.S.P. 2,023,976, 2,023,979—82, 2,026,195, 2,023,993—7, and 2,026,217—9, (A—E, G—K) 10.12.35, (F, L—N) 31.12.35.

Аррі., [А] 7.12.33, [В—Н] 12.11.34, [І—К, М] 31.12.34, [L] 25.6.34, and [N] 30.1.35).-The following agents are used : (A) a mixed ester (or mixed esters) of a polyhydric alcohol (or alcohols) and two dissimilar carboxylic acids, one detergent-forming and the other polybasic, e.g., phthalylmono-olein; (B) a product consisting of or derived from a superoxidised castor oil, oxidation being carried to a point just short of kerosene-insolubility; (c) an oxidation product of a mixture of castor oil and at least one semi-drying oil of the rape oil group, with or without addition of one member of the cottonseed oil group (castor oil; 40-80% of total); (D) a product derived from the oxidation of 40-80% of castor oil with 60-20% of sardine oil; (E) a mixture of conventionally-blown reactive fatty substances and dehydrated ricinoleic acid compounds; (F) an anhyd., oilsol. mixture containing blown oils, sulphonates of fatty acids, non-sulphonated fatty materials, and an aliphatic monohydric alcohol or alcohols, with < 5% of  $H_20$ ; (G) dehydration products of ricinoleic acid neutralised with a substituted basic amine and diluted with pine oil; (H) blown castor oil mixed with dehydrated ricinoleic acid compounds and alkylated naphthalenesulphonic acids; (I) an ester derived from a non-cyclic, non-fatty acid of the type  $(CH_2)_n(CO_2H)_2$ , (n = 5 - 8) and a hydroxylated fatty acid; (J) a hydroxyhendecenoic acid derivative : (K) a  $\Delta^{\theta \kappa}$ -hydroxyoctadecadienoic acid derivative; (L) a fatty acid (I) in which a hydroxylated, non-sulphonated aromatic residue is attached directly to the hydrocarbon radical of (I); (M) the sulphonation product of an unsaturated acid substance resulting from the thermal or pyrolytic decomp. of unsaturated hydroxyfatty acids; ((N) a sulphite or bisulphite additive compound of a fatty substance. D. M. M.

Cracking and coking hydrocarbon oils or tars and mixtures thereof with coal. H. A. BRASSERT & Co., LTD., and A. FISHER (B.P. 449,870, 1.1.35. Cf. B.P. 440,181 and 440,434; B., 1936, 259).—The tar, oil, or mixture thereof with coal, is coked, e.g., by the Knowles process, and the gases and vapours evolved are passed into a washing and fractionating column, from which fractions of different qualities are withdrawn at different levels. The heavier fractions are cooled and, after removal of the  $C_{10}H_8$  which crystallises out, are returned to the oven. A. B. M.

Distilling and coking mixtures of solid carbonaceous materials and hydrocarbon oils. N. CONSITT, J. SMITH, and R. E. COWELL (B.P. 449,619, 31.12.34).— Finely-divided coal, peat, shale, etc. is mixed with oil and the mixture heated, with agitation, under refluxing conditions and, if desired, in presence of N<sub>2</sub> or other inert gas, to about 325°. The permanent mixture so produced is discharged into a retort and carbonised at 500—600°. The vapours evolved pass to a fractionating column. The oils boiling above 200° are returned, without cooling, to the mixer. A. B. M.

Manufacture of hydrocarbons. V. IPATIEV, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 2,005,861, 25.6.35. Appl., 16.1.33).—Gaseous olefines in the gases from cracked hydrocarbon oil are caused to interact with  $C_6H_6$  hydrocarbons, of b.p. within the gasoline range, at about 60° in presence of  $H_3PO_4$ . The additive reaction yields alkylaryl hydrocarbons of high antiknock val. A. W. B.

Manufacture of hydrocarbons. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 2,010,948—9, 13.8.35. Appl., [A] 17.3.33, [B] 24.3.33).—(A) Aromatic hydrocarbons (I), e.g.,  $C_6H_6$ , are alkylated (e.g., with cracked gases and  $H_2SO_4$ ) and the products fractionated for motor fuel, the high-boiling fractions being dealkylated by treatment with a further quantity of (I) and a suitable catalyst (AlCl<sub>3</sub>, ZnCl<sub>2</sub>) and the product again distilled to give a fraction suitable for motor fuel. (B) Lower aromatic hydrocarbons are alkylated by condensation with gaseous olefines in presence of  $H_2SO_4$ . The product is fractionated to give a high anti-knock motor fuel and the high-boiling residue recycled with fresh starting materials through the converter. S. C.

Production of hydrocarbons of high b.p. Courts & Co., and F. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 449,274, 27.2.35).—A mixture of CO and  $H_2$  is heated at 150—300°/ > 20 atm. in presence of catalysts promoting the conversion of the gas mixture into hydrocarbons, e.g., metals and oxides of group VI and of the Fe group, preferably promoted by addition of alkali, rare earths, etc., and in a medium of aromatic character, e.g., anthracene oil, which remains liquid under the reaction conditions. Paraffin wax and lubricating oil are produced. A. B. M.

Rendering liquid hydrocarbons fluorescent and dyes therefor. WILMOT & CASSIDY, INC., Assees. of T. A. CASSIDY (B.P. 447,469, 19.9.34. U.S., 24.7.34).— Petroleum hydrocarbons are made fluorescent by addition of, *e.g.*, 6 : 12-dimethyl-cœroxene or -cœroxenol, or an org. ester of the latter, or diphenylbenzfuran.

C. H. **Production of** (A) gaseous fuel mixtures, (B) fuel. RUHRCHEMIE A.-G. (B.P. 446,493 and 449,013, [A] 27.7.34, [B] 10.12.34. Ger., [A] 31.7.33, [B] 9.12.33. Addns. to B.P. 444,315; B., 1936, 583).—(A) The process of the prior patent is operated to give a fuel containing 30—50% or < 20% of  $C_2H_4 + C_2H_6$ , the remainder except for a small proportion of  $H_2$ , CO, CH<sub>4</sub>, etc. consisting of  $C_3H_6$ ,  $C_3H_8$ ,  $C_4H_8$ ,  $C_4H_{10}$ , and higher-boiling hydrocarbons. (B) Coke-oven gas, after removal of the  $C_6H_6$  and  $H_2S$ , is compressed (1—5 atm.) and treated with active C. The fuel is recovered from the latter by heating under reduced pressure, and also periodically flushing with direct steam. The process is used advantageously in conjunction with pressure-transmission systems.

A. B. M.

Motor fuels. C. ARNOLD. From STANDARD OIL DEVELOPMENT CO. (B.P. 445,503, 4.6.35).—Ethers, ROR', where R and R' are hydrocarbon radicals either or both of which are branched, e.g.,  $Pr_2^{\rho}O$ , are added to motor fuels to reduce knocking tendencies. A. W. B.

Fuels for use in internal-combustion engines and for other purposes. R. G. TERAKOPOFF (B.P. 449,160, 20.11.34).—A composition containing Et<sub>2</sub>O, paraldehyde, tetrahydronaphthalene, a lubricant, and, if desired, NH<sub>2</sub>Ph or other aromatic amine is added to an internal-combustion engine fuel in the proportion of  $\frac{1}{2}$ —1%. A. B. M. Metallo - organo - derivatives [antiknock fuel]. S. SHAPPIRIO (U.S.P. 2,012,356, 27.8.35. Appl., 18.11.32). —An antiknock fuel is produced by halogenating petrol or other hydrocarbon oil, treating the product with Mg to form a Grignard compound, and then with a Pb salt or Pb-alkali metal alloy. S. C.

Lubricants. N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 449,311, 15.2.36. Holl., 19.3.35).—Lubricants suitable for use at high pressures, elevated temp., and high velocities are produced by adding to a lubricating oil a small proportion, e.g., 2%, of an org. cyclic compound containing groups in which S occurs linked to O and to a halogen, e.g.,  $p-C_6H_4Me\cdotSO_2Cl.$  A. B. M.

Production of viscous liquids [lubricating oils]. A. W. NASH, T. G. HUNTER, and W. R. WIGGINS (B.P. 447,778, 1.12.34).—Chlorinated petroleum, coal-tar products and hydrocarbons (C  $\lt$  10), e.g., chlorinated paraffin wax, are agitated in presence of Al with aromatic hydrocarbons, e.g., PhMe, with or without a nonaromatic diluent, e.g., light petroleum. The product is cooled, settled, centrifuged, washed, filtered through bleaching earth, and the diluent distilled (cf. B.P. 415,526; B., 1934, 1002). S. M.

Lubricating oil compositions. STANDARD OIL DEVELOPMENT CO. (B.P. 445,813, 26.4.35. U.S., 14.7.34).—Organometallic compounds (I) containing  $\bigstar$  2 metal atoms per mol., the atoms being of the 2nd, 3rd, 4th, and 5th groups and linked together, are added to lubricating oils to inhibit oxidation. (I) are prepared by reactions of the type MRNa + M'R'Cl  $\rightarrow$  MR·M'R'+ NaCl, where R and R' are org. radicals and M and M' are metals, usually conducted in liquid NH<sub>3</sub> or amines of low b.p. at  $\leq$  -20° to -30°. Examples are : SnMe<sub>3</sub>·SnPh<sub>3</sub>, Sn<sub>2</sub>Ph<sub>6</sub>, Bi<sub>2</sub>(C<sub>6</sub>H<sub>4</sub>Me)<sub>4</sub>, Hg<sub>2</sub>Me<sub>4</sub>, Pb<sub>2</sub>Ph<sub>6</sub>, SnMe<sub>3</sub>·PbPh<sub>3</sub>, SnEt<sub>3</sub>·BiPh<sub>2</sub>, SnPr<sub>3</sub>·Sb(C<sub>6</sub>H<sub>4</sub>Me)<sub>2</sub>, Bi(C<sub>10</sub>H<sub>7</sub>)<sub>2</sub>·As(C<sub>6</sub>H<sub>4</sub>Me)<sub>2</sub>, BiPh<sub>2</sub>·SnPr<sup>\$\vee\$-As(C<sub>6</sub>H<sub>4</sub>Me)<sub>2</sub>, SnMe<sub>3</sub>·TlPh<sub>2</sub>. A. W. B.</sup>

Purifying anthracene etc.—See III. Oil-sol. azo dyes. Oil colours.—See IV. Pickling metals.— See X. Rubber composition.—See XIV.

## **III.—ORGANIC INTERMEDIATES.**

Determination of carbon disulphide. W. A. DAMON (72nd Ann. Rept. on Alkali etc. Works, 1935, 29-32).-Exhaust gases from waterproofing works carrying out cold-curing of rubber contain CS<sub>2</sub>, SO<sub>2</sub>, and HCl. CS2 can be absorbed in KOH-EtOH and the xanthate titrated. Trial of the CuSO4 method gave unsatisfactory results. Titration with I, using pure CS2, gives very good results if fresh KOH-EtOH is used and a blank determination made. The SO<sub>2</sub> is too small in quantity seriously to affect the result. Another accurate method is to add H<sub>2</sub>O<sub>2</sub> and set the solution aside, with occasional shaking, for  $1\frac{1}{2}$  hr., the  $H_2SO_4$  produced being then titrated. It is slower but, as it can be combined with a normal acidity determination, enables the SO<sub>2</sub> and HCl to be allowed for. C. I.

Furans. F. N. PETERS, JUN. (Ind. Eng. Chem., 1936, 28, 755-759).—An historical review of industrial applications of furfuraldehyde and its derivatives.

R. S. C.

Xylene. Coal-tar phenols.—See II. Utilising cellulosic materials.—See XVIII. Anæsthetic  $Et_2O$ . —See XX.

#### PATENTS.

Manufacture of polymerisation products of olefines. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 447, 973,23.8. and 20.12.34).—Lower olefines (e.g., as present in coke-oven gases) are polymerised to liquid hydrocarbons by treatment with liquid (85%) H<sub>3</sub>PO<sub>4</sub> in presence of a Cu or Ag catalyst, preferably at 100—200° under pressure. C<sub>3</sub>H<sub>6</sub> gives mainly C<sub>9</sub>H<sub>18</sub>. S. C.

Treatment of aliphatic and cyclic saturated hydrocarbons. J. J. PELC (U.S.P. 2,011,199, 13.8.35. Appl., 17.8.34).—Such hydrocarbons are condensed with lower aldehydes and/or ketones (particularly COMe<sub>2</sub>, COEt<sub>2</sub>, and polymeride of MeCHO) either by heating to  $500-800^{\circ}$  or preferably by treatment with a condensing agent (H<sub>2</sub>SO<sub>4</sub>, NaOH) at about room temp. The products consist of alcohols, olefines, or polymerisation products according to the severity of the conditions. S. C.

Manufacture of chlorinated hydrocarbons. W. E. SHARP (U.S.P. 2,010,039, 6.8.35. Appl., 23.4.32).— A dry-cleaning fluid (mostly  $C_3H_5Cl_3$ ) is obtained by chlorinating pure or crude  $C_3H_8$  or a lower chlorination product thereof in stages at temp. rising from 50° to 400°, in presence of light and a metal halide. R. S. C.

(A) Chlorination process and product thereof. (B) Chlorination of propane and its partly chlorinated derivatives. H. B. HASS and E. T. MCBEE, Asses. to PURDUE RESEARCH FOUNDATION (U.S.P. 2,004,072-3, 4.6.35. Appl., [A] 1.2.32, [B] 1.5.34).-Chlorination is carried out at high temp. by separately preheating the starting material [(A) a hydrocarbon or chlorohydrocarbon of the paraffin, naphthene, or aromatic series] and  $Cl_2$  at  $> 250^\circ$  and mixing at a speed > that of flame propagation (A) in absence of light and catalysts. Apparatus is described. E.g., (A) chlorination of isobutane or Bu<sup>B</sup>Cl at 250-600° gives the ay-Cl2derivative, b.p. 136.4°, as main product; similarly, (B) chlorination of  $C_3H_8$  or PrCl at  $> 250^\circ$  gives mainly  $CH_2(CH_2Cl)_2$ . H. A. P.

Chemical product [mixtures of proteins with the additive product of vinylacetylene and hydrochloric acid]. A. M. COLLINS, ASST. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 2,010,012, 6.8.35. Appl., 9.1.34).—Acid and alkaline dispersions (prep. described) of polymerised  $\beta$ -chlorobutadiene are compounded with acid and alkaline dispersions, respectively, of proteins, *e.g.*, casein, gelatin, glue. The products are employed as interlayers in safety glass, paper size, vehicle for cold-H<sub>2</sub>O paints, casein plastics, impregnating and coating compositions, and finishes for leather. P. G. C.

(A) Fluorination of carbon compounds. (B) Fluorine compounds. (C) Halogenated ethane derivatives containing fluorine. (D)—(G) Organic fluorine compounds. (H) Fluorinated acyclic hydrocarbons. H. W. DAUDT and (A) E. L. MATTI-SON, (B) M. A. YOUKER and H. L. B. JONES, (C)—(F) M. A. YOUKER, (G), (H) L. C. HOLT and E. L. MATTISON, ASSTS. to KINETIC CHEMICALS, INC. (U.S.P. 2,004,931—2 and 2,005,708–13, 18.6.35. Appl., [A] 20.3.31, [B] 24.3.31, [c] 24.8.33, [D] 7.10.33, [E] 26.3.34, [F] 15.6.34, [G] 31.8.32, [H] 2.2.33).—Replacement of halogen attached to acyclic C in org. compounds by F by, in general, treatment with fluorinating agents, e.g., HF, heavy-metal fluorides (ZnF<sub>2</sub>, PbF<sub>2</sub>), Sb fluorochlorides of the type SbCl<sub>5-x</sub>F<sub>x</sub>, and halogenation catalysts, e.g., SbCl<sub>5</sub>, is claimed. Typical reactions are CCl<sub>4</sub>  $\rightarrow$  CCl<sub>2</sub>F<sub>2</sub> + CCl<sub>3</sub>F, and CH<sub>2</sub>Cl<sub>2</sub>  $\rightarrow$  CH<sub>2</sub>ClF + CH<sub>2</sub>F<sub>2</sub>. (A) deals mainly with treatment of CCl<sub>4</sub>, (B) S compounds, e.g., CS<sub>2</sub>, CSCl<sub>2</sub>, (c) halogenoethanes, (F) CH<sub>2</sub>Cl<sub>2</sub>, and (G) aralkyl halides. Apparatus is described in (C), (D), and (E). A. W. B.

Stabilisation of halogenated hydrocarbons. P. J. CARLISLE and C. R. HARRIS, ASSTS. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,008,680, 23.7.35. Appl., 3.3.31).—Halogenated hydrocarbons (e.g.,  $C_2H_2Cl_4$ ,  $Ccl_2$ :CHCl,  $CH_2Cl_2$ ,  $C_2Cl_4$ ,  $C_2H_2Cl_2$ ) which develop acidity on storage are stabilised by addition of 0.0001— 1% of a phenol, e.g., PhOH, o-NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·OH, p-cresol, o-, m-, and p-C<sub>6</sub>H<sub>4</sub>(OH)<sub>2</sub>,  $\alpha$ -C<sub>10</sub>H<sub>7</sub>·OH, 1:2:3-C<sub>6</sub>H<sub>3</sub>(OH)<sub>3</sub>, thymol. P. G. C.

Effecting organic vapour-phase reactions. J. C. WOODHOUSE, Assr. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 2,003,477, 4.6.35. Appl., 28.2.31).—The use of halogens, H halides, gaseous alkyl halides, or oxides of N as gaseous exciters for additive reactions of CO, *e.g.*, ROH + CO  $\rightarrow$  RCO<sub>2</sub>H, is claimed. The addenda, in concns. of 0.1—5 vol.-%, are mixed with the CO prior to or during admixture with the other reactant. Examples are : HCN from CO and NH<sub>3</sub> + NO ; AcOH and MeOAc from CO and MeOH + HCl (Cu chloride catalyst) ; CO<sub>2</sub> and H<sub>2</sub> from CO and steam + Br.

A. W. B. Separation of chlorohydrins from hydrochloric acid solution. Soc. CARBOCHIM., Soc. ANON. (B.P. 445,011, 9.9.35. Fr., 26.12.34).—An aq. solution of HCl and chlorohydrin, e.g., such as is obtained from an olefine and  $Cl_2$ -H<sub>2</sub>O, is neutralised with NH<sub>3</sub> or an NH<sub>4</sub> salt of a volatile weak acid, and the chlorohydrin is separated by distillation, NH<sub>4</sub>Cl being recovered from the residue. H. A. P.

Preparation of olefine derivatives [ethyl alcohol]. W. B. SCOTT, L. S. BOVIER, and E. D. MATTHEWS, ASSTS. to HOOKER ELECTROCHEM. Co. (U.S.P. 2,004,084, 4.6.35. Appl., 18.4.32).—C<sub>2</sub>H<sub>4</sub> or gases containing it are passed into 83—88% H<sub>2</sub>SO<sub>4</sub> at 60—100° (80°)/75—300 lb. per sq. in. until the mol. ratio C<sub>2</sub>H<sub>4</sub>: SO<sub>3</sub> is  $\lt$  0.8 (1·25). The product (containing EtHSO<sub>4</sub> and Et<sub>2</sub>SO<sub>4</sub>) is hydrolysed by addition to H<sub>2</sub>O at the b.p., the EtOH being removed by distillation as formed. The amount of H<sub>2</sub>O used is adjusted so that the final concn. of aq. H<sub>2</sub>SO<sub>4</sub> is  $\lt$  50%. H. A. P.

Condensation of alcohols. N. D. Scorr, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,004,350, 11.6.35. Appl., 2.12.31).—The alcohol is heated in the liquid phase with a dehydrogenating catalyst (Cu-Al<sub>2</sub>O<sub>3</sub> gel, Cu chromite) with or without a condensing catalyst (alkalis, Cs<sub>2</sub>CO<sub>3</sub>, K<sub>2</sub>SiO<sub>3</sub>) and a dehydrating catalyst (Al<sub>2</sub>O<sub>3</sub>, ThO<sub>2</sub>); esters and higher alcohols are formed, the mol. wt. of the latter depending on the ratio of dehydrogenating to condensing catalyst. *E.g.*, EtOH with Cu-Al<sub>2</sub>O<sub>3</sub> at 220° gives mainly EtOAc; in presence of  $K_2CO_3$  in addition the main products are BuOH and hexyl and higher alcohols. H. A. P.

Conversion of olefines into sulphuric esters. E. I. DU PONT DE NEMOURS & Co. (B.P. 448,457, 7.12.34. U.S., 7.12.33).—An equimol. mixture of a normally gaseous olefine, *e.g.*,  $C_3H_6$ , under sufficient pressure to ensure liquefaction, and  $\lt$  75% (90—95%)  $H_2SO_4$ is passed through a narrow tube under conditions of turbulent flow at  $> 30^\circ$  (25°). High yields of sulphuric esters are claimed. N. H. H.

Method for minimising polymerisation [in reaction between olefines and sulphuric acid]. K. B. LACY, ASST. to VAN SCHAAK BROS. CHEM. WORKS, INC. (U.S.P. 2,009,062, 23.7.35. Appl., 21.4.33).—Olefines  $(C_4-C_7)$  are treated with  $H_2SO_4$  (preferably 75%) at  $< 30^\circ$  until the mixture consists essentially of AlkHSO<sub>4</sub>. Sufficient primary alcohol (EtOH) is then added to react with the acid to form Et alkyl sulphate, which is subsequently hydrolysed to form primary and sec. alcohols or ethers. S. C.

Production of aliphatic organic compounds. CARBIDE & CARBON CHEMICALS CORP., Assees. of J. N. WICKERT and B. T. FREURE (B.P. 446,026, 21.12.34. U.S., 5.1.34).—COMeBu<sup> $\beta$ </sup> is condensed with an aliphatic aldehyde  $\measuredangle$  C<sub>8</sub>, particularly  $\alpha$ -ethylhexaldehyde or α-ethyl-β-propylacraldehyde, in presence of an alkali (KOH) in an inert solvent (MeOH, EtOH) at  $> 25^{\circ}$ . The reaction may be continued (15 hr.) until the ketol first formed is dehydrated to the corresponding unsaturated ketone, which is purified (distillation); this is reduced by H<sub>2</sub>, e.g., over Ni at 100-125°/900-1000 lb. per sq. in., to the saturated ketone and alcohol  $\lt$  C<sub>14</sub>, which are separated by distillation, and the alcohol is monosulphated (by 90-95% H<sub>2</sub>SO<sub>4</sub> and Ac<sub>2</sub>O, ClSO<sub>3</sub>H, etc.). The salts of the product with various bases are claimed as detergents. E. J. B.

Production of sulphonated [alcohol] products. A. BEYER (B.P. 448,804, 18.12.34. Ger., 18.12.33).— Crude or purified polysulphonic acids of carboxyl-free aromatic or hydroaromatic hydrocarbons or their derivatives are partly esterified by treating with  $\leq$ stoicheiometric amounts of higher aromatic alcohols having  $> C_{10}$  or hydroaromatic OH-compounds, e.g., lauryl, cetyl, or naphthenyl alcohol, at room or slightly raised temp. The products are wetting, dispersing, etc. agents. The esters may be further caused to react with a nuclear combining agent, e.g., CH<sub>2</sub>O, to produce tanning agents, mordants, etc. N. H. H.

Manufacture of sulphonation products [wetting etc. agents, from phosphatides]. C. E. EVERY-CLAYTON. From CHEM. FABR. STOCKHAUSEN & Co. (B.P. 449,132, 25,10.35).—Vegetable or animal phosphatides (I), e.g., soya- or egg-lecithin, are sulphonated, either alone or in presence of fats or fatty acids, with  $\leq 60$  wt.-% of H<sub>2</sub>SO<sub>4</sub>, SO<sub>3</sub>, or CISO<sub>3</sub>H at  $> 10^{\circ}$  in presence, if desired, of a diluent, e.g., benzine or CCl<sub>4</sub>, and a dehydrating agent, e.g., Ac<sub>2</sub>O. Superior products are claimed when (I) is sulphonated in presence of substances capable of condensation, e.g., aliphatic, aromatic, or hydroaromatic alcohols or aldehydes, or substituted aromatic hydrocarbons. Examples of the use of the products in textile operations are given. N. H. H. Esters of acids of phosphorus. G. DE W. GRAVES, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,005,619, 18.6.35. Appl., 10.11.34).—Mixtures of esters or mixed esters of acids of P, containing at least one alkyl of  $\ll C_8$  and at least one other alkyl or aryl, are prepared by esterification of appropriate mixtures of OH-compounds. *E.g.*, a mixture of 60% of lauryl alcohol (I) with 40% of myristyl alcohol, in  $C_8H_6$ , treated with POCl<sub>3</sub> under reflux, gives lauryl myristyl phosphate; (I) with  $(C_6H_4Me)_2POCl$  gives ditolyl lauryl phosphate. A. W. B.

Manufacture of wetting, washing, emulsifying, softening, and like agents. E. A. MAUERSBERGER (B.P. 448,668, 31.10.35).—Equimol. mixtures of the boric esters of wool-fat alcohols or cholesterols derived therefrom and the boric ester of an aliphatic alcohol, *e.g.*, oleyl alcohol, are sulphonated in  $H_2SO_4$  or oleum at 25—70°. The two esters are said to condense prior to sulphonation. The neutralised reaction products may be bleached, *e.g.*, by  $H_2O_2$ . N. H. H.

Production of thiosulphate esters. A. KIRSTAHLER and W. J. KAISER (U.S.P. 2,004,873, 11.6.35. Appl., 5.12.33. Ger., 4.1.32).—An alkyl, cycloalkyl, or aralkyl ester of a reactive halogenocarboxylic acid in which the alkyl etc. has  $\langle \mathcal{L}_6$  is caused to interact with a H<sub>2</sub>O-sol. thiosulphate. *E.g.*, CH<sub>2</sub>Cl·CO<sub>2</sub>·C<sub>12</sub>H<sub>25</sub>-n is heated with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> to give Na dodecyl glycollate thiosulphate, C<sub>12</sub>H<sub>25</sub>O<sub>2</sub>C·CH<sub>2</sub>·S·SO<sub>3</sub>Na; similar products are obtained from octadecenyl,  $\beta$ -dodecoxyethyl, and naphthenyl chloroacetates. The products are sol. in H<sub>2</sub>O and have soap-like properties. H. A. P.

Manufacture of cyclic esters. E. I. DU PONT DE NEMOURS & Co. (B.P. 448,708, 12.12.34. U.S., 12.12.33). —Monomeric cyclic esters and lactones containing >C<sub>7</sub> are obtained in good yields by carrying out the prep. in presence of  $\lt$  20 and preferably  $\lt$  100 vols. of an inert solvent. In absence of the large amount of diluent, the products are mainly polymerides (cf. B.P. 433,632; B., 1936, 684). S. C.

Esterification process. J. P. BURKE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,010,426, 6.8.35. Appl., 28.1.32).—> 90% yields of pure esters are obtained by distilling  $CH_2Cl_2$ , ( $\cdot CH_2Cl)_2$ , or  $CHMeCl \cdot CH_2Cl$  with the alcohol, acid, and catalyst (e.g.,  $H_2SO_4$ ) until the theoretical amount of  $H_2O$  is removed as binary  $H_2O$ -dichloride mixture, provided that the b.p. of the alcohol is > 30° above that of the dichloride. If this difference is  $< 30^\circ$ , a ternary mixture of alcohol,  $H_2O$ , and dichloride distils. The distillate separates into two layers, allowing recovery of the dichloride. The prep. of Bu<sub>2</sub>, di(methoxyethyl), and amyl phthalate is described. R. S. C.

Production of halogenated alcohols and halogenated ethers. N. V. DE BATAAFSCHE PETROLEUM MAATS., Assees. of H. P. A. GROLL and J. BURGIN (B.P. 448,659, 22.6.35. U.S., 23.6.34).—Normal and branchedchain unsaturated halides ( $\ll C_4$ ) containing an olefinic linking, which may be attached to a *tert*. C, are converted into esters by treatment with approx. 1 mol. of an inorg. oxy-acid (H<sub>2</sub>SO<sub>4</sub>, H<sub>3</sub>PO<sub>4</sub>, HNO<sub>3</sub>, HClO<sub>4</sub>), HOHal excepted. In the preferred method 80—90% H<sub>2</sub>SO<sub>4</sub> is used at  $\leq 25^{\circ}$  to prevent polymerisation etc. Halides of the vinyl type react more slowly than those in which the halogen and olefine groups are further removed. The esters are converted into halogenated alcohols by dilution with  $H_2O$  at low temp. to give 10-15%  $H_2SO_4$ ; reaction with mono- or poly-hydric alcohols gives the corresponding ethers. The method is not successful with olefines containing *tert*. or quaternary C  $\beta$  to the double linking. The examples include the prep. of OH·CMe<sub>2</sub>·CH<sub>2</sub>Cl, [CH<sub>2</sub>Cl·CMe(OH)·CH<sub>2</sub>]<sub>2</sub>, and OH·CPhMe·C<sub>6</sub>H<sub>4</sub>Br. S. C.

Preparation of lævulic esters. E. F. IZARD and P. L. SALZBERG, ASSTS. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,004,115, 11.6.35. Appl., 7.5.32).— Lævulates of esters or ethers of polyhydric alcohols are claimed. Examples are : ethoxyethyl, b.p. 108°/5 mm., butoxyethyl, b.p. 150—175°/11 mm., "lauroxyethyl," b.p. 170—190°/1·5 mm., phenoxyethyl, b.p. 165—166°/ 2 mm., ethoxyethoxyethyl, b.p. 139—140°/2 mm., and butoxyethoxyethyl lævulate, b.p. 175—195°/10 mm., and lævulates of hydrogenated castor oil and the monoand di-glycerides of the fatty acids of coconut oil. The products are plasticisers for cellulose derivatives etc.

H. A. P.

Lævulic esters. W. E. Lawson and P. L. SALZBERG, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,008,720, 23.7.35. Appl., 3.5.32).—Esters of lævulic acid with aliphatic alcohols ( $C_7$ — $C_{18}$ ) are obtained by heating at 80—250° with removal of H<sub>2</sub>O as it is formed ; catalysts (HCl, H<sub>2</sub>SO<sub>4</sub>, p-C<sub>6</sub>H<sub>4</sub>Me·SO<sub>3</sub>H, Na alkoxides, ZnCl<sub>2</sub>) may be added. Lauryl b.p. 175—190°/5 mm., benzyl, b.p. 160—170°/8 mm., and stearyl lævulate, b.p. 180—200°/1·5 mm., are described. The products are highly compatible with cellulose esters and ethers and may be used as plasticisers. P. G. C.

Ether acid ester of [esters of alkoxy-acids and] polyhydric alcohols. [Plasticisers.] M. H. Hu-BACHER, Assr. to H. S. KREIGHBAUM (U.S.P. 2,010,154, Appl., 27.6.33).—Glycerol tri-β-n-butoxyethoxy-6.8.35. acetate, b.p. 255-275°/15 mm., and -β-methyl-n-amyloxyacetate, b.p. 245-290°/20 mm., diethylene glycol di-\beta-methyl-n-amyloxyacetate, b.p. 226-234°/15 mm., and - \Beta-n-butoxyethoxyacetate (I), b.p. 219-263°/8 mm., triethylene glycol di-β-methyl-n-amyloxyacetate, b.p. 238-264°/8 mm., and nine similar esters are good plasticisers for cellulose esters and ethers and for resins, (I) being the best. Some, particularly (I), are solvents for cellulose nitrate. The requisite acids are prepared by heating the Cl-acid with the appropriate Na alkoxide. Thus are obtained \$\$'-methyl-n-amyloxy-, b.p. 156-161°/28 mm., β-n-butoxy-, b.p. 177-179°/25 mm., 281–288° (decomp.)/760 mm., β-methoxy-, b.p. 121–122°/4 mm., β-ethoxy-, b.p. 125–126°/4 mm., β-npropoxy-, b.p. 131°/4 mm., ββ'-n-butoxyethoxy-, and ββ'-methoxyethoxy-ethoxyacetic acid, b.p. 155-156°/4 mm. R. S. C.

Recovery of acidic and basic reagents. BRIT. CELANESE, LTD., H. F. OXLEY, and E. B. THOMAS (B.P. 445,108, 2.10.34).—A salt of a volatile org. acid and a volatile base (e.g.,  $NH_4$  or an amine acetate) is separated into its constituents by thermal decomp. in presence of a substance which forms with the acid a stable compound at the temp. employed (NaOAc, KOAc), this compound itself being resolved into its constituents by heating at a higher temp. E.g., aq.  $\rm NH_4OAc$  is heated with KOAc-NaOAc (equimols.) to 150°;  $\rm NH_3$  and H<sub>2</sub>O distil off. On distilling the residue up to 250°/vac. AcOH and a little unchanged  $\rm NH_4OAc$  distil. H. A. P.

Manufacture of condensation products. I. G. FARBENIND. A.-G. (B.P. 449,185, 21.12.34. Ger., 23.12.33).—Citric acid is heated with mannitol (1 mol.) at  $125^{\circ}/18$  mm. or sorbitol (1 mol.) at  $120^{\circ}/15$  mm. until 1 CO<sub>2</sub>H is esterified. The products are dibasic and give sol. Mg and Ca salts, the latter suitable for internal administration. Tartaric acid or any alcohol with > 2 OH may be used. R. S. C.

Production of formic acid. E. W. FAWCETT, R. O. GIBSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 447,492, 19.11.34).—Conc. solutions of  $HCO_2H$ are obtained by interaction of  $H_2O$  and CO alone or in presence of catalysts (halides of H and metals, metal oxides, and non-volatile acidic oxides) at  $175-250^\circ/\ll$ 2000 atm. S. C.

Production of halogenated organic acids. N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 447,876, 8.11.35. U.S., 19.11.34) .- Halogenated org. acids are produced in excellent yields by treating halogenated ketones, containing at least one halogen in an alkyl group, with an oxidising agent (usually HNO<sub>3</sub>), in presence of H<sub>2</sub>O and an inert diluent, which is preferably the halogenated acid produced. The oxidising agent is introduced in a very finely-divided state, e.g., by vaporisation in air, and arrangements are made for the rapid dissipation of the heat of reaction. The prep. of CHMeCl·CO.H from CHMeCl·COMe is described in detail. CH\_Cl·CO\_H is obtained from CH\_Cl·COMe or CH2Cl·CO·CH2·CH2Cl (high yield), CHCl2·CO2H from CHCl<sub>2</sub>·COMe, and CMe<sub>2</sub>Br·CO<sub>2</sub>H from CMe<sub>2</sub>Br·COMe. The corresponding OH-acids are obtained by hydrolysis with 1 mol. of dil. NaOH under pressure. S. C.

Production of acid anhydrides. DEUTS. GOLD- U. SILBER-SCHEIDEANSTALT VORM. ROESSLER (B.P. 447,756, 19.11.34. Ger., 17.11.33).—Anhydrides substantially free from mixed anhydrides are obtained by heating fatty acids (>  $C_2$ ) with 1 mol. of Ac<sub>2</sub>O and removing the AcOH continuously as formed by distillation, *e.g.*, in a column fitted with a suitable dephlegmator. Esters may be employed instead of fatty acids and the corresponding acetate removed continuously. The prep. of (EtCO)<sub>2</sub>O, (PrCO)<sub>2</sub>O, and (C<sub>5</sub>H<sub>11</sub>·CO)<sub>2</sub>O is described. S. C.

Manufacture of lower aliphatic anhydrides. H. DREYFUS (B.P. 448,452, 7.12.34).—Lower aliphatic acid anhydrides are obtained by thermal decomp. of esters of the corresponding acids with unsaturated, *e.g.*, vinyl or allyl, alcohols (I) containing substantially no free carboxylic acid, at 250—650°. Catalysts, *e.g.*, pumice, and/or dehydration catalysts, *e.g.*, H<sub>3</sub>PO<sub>4</sub>, may be used. The unsaturated ethers corresponding to (I) are produced simultaneously. N. H. H.

Manufacture of aliphatic anhydrides and aldehydes. H. DREYFUS (B.P. 447,690, 24.11.34).—Alkylidene di-esters of lower fatty acids are subjected to thermal decomp., preferably at  $300-400^\circ$ , in presence of an inert diluent (CO<sub>2</sub>, N<sub>2</sub>, CH<sub>4</sub>) and an acid or acid salt as catalyst  $(H_3PO_4, H_2SO_4)$ , to give the corresponding acid anhydride and aldehyde. S. C.

Oxidation of aliphatic alcohols to aldehydes. H. A. BOND and L. B. SMITH, Assrs. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 2,005,645, 18.6.35. Appl., 15.4.30).—Porous, pelleted reduced Ag, which may be activated by presence of  $\geq 10$  ( $\geq 1$ )% of an oxide of W, V, Ce, Th, Al, Cr, Zn, or Mo, is claimed as a catalyst. The process applies particularly to lower alcohols, *e.g.*, MeOH  $\rightarrow$  CH<sub>2</sub>O. A. W. B.

Manufacture of formaldehyde and other oxidation products from paraffins having more than one carbon atom in the molecule. GUTEHOFFNUNGS-HÜTTE OBERHAUSEN A.-G. (B.P. 448,733, 8.4.35. Ger., 7.4.34).—Paraffins of  $\langle C_2, e.g., C_2H_6, C_3H_8, C_4H_{10}$ , are oxidised by means of gases containing  $O_2, e.g.$ , air, and about 1% of NO at 500—800° in presence of a catalyst, *i.e.*, one or more elements of group II or IV or one or more of these elements with at least one of the elements Cr, V, Mo, Al, W, or Ni (optionally as oxide or carbonate), to CH<sub>2</sub>O and other products. Examples are :  $C_2H_6 \rightarrow$ CH<sub>2</sub>O + MeCHO,  $C_3H_8 \rightarrow$  CH<sub>2</sub>O + MeCHO + HCO<sub>2</sub>H,  $C_4H_{10} \rightarrow$  CH<sub>2</sub>O + MeOH + MeCHO. N. H. H.

Chemical compound. [Condensation products of vinyl ketones.] H. S. ROTHROCK, ASST. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,010,828, 13.8.35. Appl., 18.8.32).—A vinyl ketone, e.g., COMe·CH·CH<sub>2</sub> (I), is condensed with a compound RXH (R = alkyl, aryl, or acyl; X = 0, S, or SO<sub>2</sub>). The examples include the prep. of the following from (I), the appropriate reagent being given in parentheses : Et, b.p.  $56-60^{\circ}/24$  mm. (EtOH), and Ph  $\gamma$ -ketobutyl ether, b.p.  $160-180^{\circ}/6$  mm. (PhOH);  $\gamma$ -ketobutyl mercaptan, b.p.  $92-96^{\circ}/53$  mm. (H<sub>2</sub>S); Pr<sup>\*</sup>, b.p. 107—111°/20 mm., (Pr<sup>\*</sup>SH), and Ph  $\gamma$ -ketobutyl sulphide, b.p. 145—148°/2 mm. (PhSH);  $\gamma$ -ketobutyl acetate, b.p. 98—102°/30 mm. (AcOH); p-tolyl  $\gamma$ -ketobutyl sulphone, m.p. 70—71° (p-C<sub>6</sub>H<sub>4</sub>Me·SO<sub>2</sub>H). S. C.

Manufacture of water-soluble salts of imidoethers, imidothioethers, or amidines. I. G. FARB-ENIND. A.-G. (B.P. 448,469, 13.12.34. Ger., 13.12.33).— Sol. salts of imido-ethers and -thioethers are obtained by treating a mixture of nitriles, one of which contains  $\bigstar C_8$ , e.g., such as is obtained from commercial stearic acid or coconut oil fatty acids, with an acid (HCI) and a compound RX in which R is an aliphatic, alicyclic, or aromatic radical and X is OH, SH, NH<sub>2</sub>, or NHAlk. In the detailed examples RX = Bu<sup>a</sup>OH, EtOH, EtSH, NH<sub>2</sub>Bu<sup>a</sup>. The sol. salts of the imidoethers are converted into sol. salts of amidines by reaction with NH<sub>3</sub>, or a primary or *sec.* amine. 14 samples are described. S. C.

Preparation of acid amides and anhydrous or substantially anhydrous alcohols. N. V. DE BATAAF-SCHE PETROLEUM MAATS., Assees. of W. J. HUND and L. ROSENSTEIN (B.P. 445,148, 9.5.35. U.S., 14.5.34).—A carboxylic acid having  $\ll C_2$ , or its anhydride, halide, or ester (glyceride), is treated with anhyd. NH<sub>3</sub> under pressure at room temp. *E.g.*, coconut oil with excess of NH<sub>3</sub> gives the amides of the mixed fatty acids and glycerol. H. A. P. Manufacture of amines and diamines. J. F. OLIN, ASST. to SHARPLES SOLVENTS CORP. (U.S.P. 2,006,058, 25.6.35. Appl., 14.5.31).—The monochloropentanes, or mixtures thereof, obtained by chlorination of  $C_5H_{10}$  from natural gas are treated, *e.g.*, at 100—110° in an autoclave, with NH<sub>3</sub> or an org. base in aq. alcohol. Mixtures of mono-, di-, and tri-amylamines are obtained, according to the conditions.

A. W. B.

Manufacture of washing, wetting, emulsifying, and like agents. Courts & Co., and F. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 449,081, 10.12.34).-Primary or sec. amines, free from sulphuric ester radicals and containing at least one aliphatic or cycloaliphatic radical of  $\ll C_6$ , which may be interrupted by heteroatoms, attached directly to N, are caused to react with polycarboxylic acids  $(C_2 - C_6)$  or derivatives, e.g., esters or anhydrides, at 60-250° (100-200°), with or without a solvent or diluent, to yield, if necessary after hydrolysis, carboxylic amides which contain at least one CO<sub>2</sub>H. 16 examples are given of, e.g., the prep. of Na oleylmethyloxamate from oleylmethylamine and Et<sub>2</sub>C<sub>2</sub>O<sub>4</sub>, product from maleic anhydride and the amines resulting from NH<sub>2</sub>Me and the bromides of the alcohols derived from coconut oil fatty acids, and the product from diglycollic anhydride and methyldodecylamine.

N. H. H.

Manufacture of guanyl and biguanyl [diguanyl higher aliphatic] compounds. I. G. FARBENIND. A.-G. (B.P. 448,796, 14.12.34. Ger., 16.12.33) .-- Guanyl and diguanyl compounds in which the C is linked to a higher-mol. radical by an NH<sub>2</sub> or ether (S or O) group are prepared by condensing aliphatic C10-C16 primary or sec. amines (I), mercaptans (II), or alcohols (III) with CN·NH<sub>2</sub>, dicyanodiamide, an alkyl or aralkyl ether of isourea, isothiourea, guanylisourea, or guanylisothiourea, or a salt thereof, or by condensing the reactive esters of (III), e.g., with hydrohalide and aromatic sulphonic acids, with a guanidine, diguanidine, CS(NH2)2, guanylthiourea, or a salt thereof. (I), (II), and (III) may contain other substituents, e.g., OH, NH<sub>2</sub>, and the C chain may be interrupted by other atoms or groups, e.g., O, S, NH, or a cyclic group. Examples are : n-C12H25 NH2 and S-ethylisothiourea hydrobromide yield n-dodecylguanidine, m.p. 96° (hydrobromide, m.p. 82°); dodecanol, CN·NH<sub>2</sub>, and HCl yield dodecylisourea, m.p. 78° (hydrochloride, m.p. 93°),  $CS(NH_2)_2$  and  $C_{12}H_{25}Br$ yield s-dodecylisothiourea, m.p. 82° (hydrochloride, m.p. 133°: hydrobromide m.p. 111°); in like manner are obtained S-decylisothiourea hydrobromide, m.p. 99°, and N-methyl-S-dodecylisothiourea, m.p. 66-67°, C12H25 NH2 and S-ethylguanylisothiourea hydrobromide yield the dodecyldiguanide, m.p. 96° (Cu complex, m.p. 219°); guanylthiourea and C<sub>12</sub>H<sub>25</sub>Br yield S-dodecylguanylisothiourea hydrobromide, m.p. 140°; CS(NH<sub>2</sub>)<sub>2</sub> and chloro-methyl dodecyl ether, b.p. 126°/4 mm., yield S-dodecyloxymethylisothiourea hydrochloride, m.p. 134°; aminoethyl dodecyl sulphide, b.p. 156°/3 mm., and S-ethylisothiourea hydrobromide yield dodecylthioethylguanidine hydrobromide, m.p. 58°; aminoethyl dodecyl sulphide and S-ethylguanylisothiourea hydrobromide yield dodecylthioethyldiguanide hydrobromide, m.p. 90°; and m-aminophenyl dodecyl ether hydrochloride, m.p. 115° (free base,

Stat. ref.]

m.p. 63°), and dicyanodiamide yield 3-diguanidinophenyl ex dodecyl ether, m.p. 175° (dihydrochloride, m.p. 207°). 2

Manufacture of diaryImethanes and their derivatives. W. BLYTHE & Co., LTD., W. H. BENTLEY, and B. CATLOW (B.P. 446,450, 29.12.34).—The use of aliphatic alcohols, *e.g.*, EtOH, methylated spirit, as diluents, instead of AcOH, in the condensation of aromatic hydrocarbons with CH<sub>2</sub>O in presence of H<sub>2</sub>SO<sub>4</sub> is claimed to give improved yields. By-products are less in quantity and more easily removed. The prep. of CH<sub>2</sub>Ph<sub>2</sub>, CH<sub>2</sub>(C<sub>6</sub>H<sub>4</sub>Cl)<sub>2</sub>, CH<sub>2</sub>(C<sub>6</sub>H<sub>4</sub>Me)<sub>2</sub>, CH<sub>2</sub>Ph·C<sub>6</sub>H<sub>4</sub>Me, CH<sub>2</sub>(C<sub>6</sub>H<sub>4</sub>·OMe)<sub>2</sub>, and CH<sub>2</sub>(C<sub>10</sub>H<sub>7</sub>-1)<sub>2</sub> is described.

A. W. B.

N. H. H.

Manufacture of perylene. W. W. GROVES, Assee. of I. G. FARBENIND. A.-G. (B.P. 445,896, 20.10.34. Ger., 2.11.33. Addn. to B.P. 425,363; B., 1935, 443).— The process of the prior patent is improved by the use of the mixed three isomeric dinaphthyls obtainable from  $C_{10}H_8$  as starting material. Oxidising agents, *e.g.*, KMnO<sub>4</sub>, accelerate the reaction. If the AlCl<sub>3</sub> is mixed with alkali chloride a temp. of 100—110° may be used. A. W. B.

Purification of organic solids [anthracene etc.]. S. P. MILLER, Assr. to BARRETT Co. (U.S.P. 2,011,724, 20.8.35. Appl., 28.10.32).—Repeated washing of crude 40% anthracene with liquid  $NH_3$  at 25—50°/100—125 lb. per sq. in. in a closed system gives 80% anthracene. The  $NH_3$  is recovered from the washings after addition of a solvent for the dissolved solids ; the first washings contain mostly phenanthrene, the later mostly carbazole. The process is also applicable to crude fluorene etc. The necessary plant is described. R. S. C.

**Reduction of hydrocarbons.** E. I. DU PONT DE NEMOURS & Co. (B.P. 446,013, 22.10.34. U.S. 20.10.33).— 1 mol. of acenaphthene,  $C_{10}H_8$ , or a homologue is allowed to react with 2 atoms of an alkali metal (Na) in an inert solvent containing "active" ethers (cf. B.P. 443,361 and 445,417; B., 1936, 537, 684) to yield the corresponding dimetal compound. If this is hydrolysed in situ by, e.g., H<sub>2</sub>O, EtOH, AcOH, at (a) -20° to -30° after considerable amounts of it have been formed, or (b) at room temp. as soon as it is produced, monomeric H<sub>2</sub>derivatives (I) are obtained. If it is hydrolysed in bulk at 20-30° by H<sub>2</sub>O etc. or by further (I), resinous polymerides of (I) are obtained in good yield. E. J. B.

[Preparation of] chloro-derivatives of aromatic isoalkyl hydrocarbons. K. JÜLICHER (B.P. 448,851, 10.10.34).—isoAlkylated aromatic hydrocarbons, e.g., PhPr<sup>\$\vee\$</sup> (1), PhBu<sup>\$\vee\$</sup> (II), are chlorinated at about the b.p. with Cl<sub>2</sub> in presence of metal catalysts, e.g., Al, Ni-Co-Cu. Cl is attached only to one or more C connected to the intermediate CH of the isoalkyl chain. Examples are:  $\beta$ -mono- and  $\beta\beta$ -di-chloroisopropylbenzene from (I), and  $\gamma\gamma$ -dichloroisobutylbenzene from (II). N. H. H.

Manufacture of N-nitroamines of nuclear halogenated primary aromatic amines. Soc. CHEM. IND. IN BASLE (B.P. 447,514, 23.1.35. Switz., 23.1.34).— Diazo compounds from nuclear-halogenated arylamines are converted into the corresponding N-nitroamines by oxidation with NaOCl in 5% NaOH solution. A large

excess of alkali is unnecessary. Examples are: 3-chloro-, 2 : 5-dichloro-, 5-chloro-2-methoxy-, 4- and 5-chloro-2--methyl-phenylnitroamine. S. C.

Production of substituted benzylcarbinamines [ $\beta$ -phenylethylamines]. L. MELLERSH-JACKSON. From F. P. NABENHAUER (B.P. 447,792, 11.3.35).— $\beta$ -Phenyl $\alpha$ -alkylethylamines are obtained by electrolytic reduction of benzyl alkyl ketoximes in H<sub>2</sub>SO<sub>4</sub> (d 1·4) at 25°. The crude base is liberated by basification and purified via the hydrochloride. The prep. of CH<sub>2</sub>Ph-CHMe·NH<sub>2</sub> is described in detail. The products are remedies for colds etc. S. C.

Manufacture of 2'-nitro-2-carboxydiphenylamine. L. SPIEGLER, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,003,842, 4.6.35. Appl., 23.11.33).—The interaction of o-NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·CO<sub>2</sub>H (I) and alkali carbonate (K<sub>2</sub>CO<sub>3</sub>) (II) and o-C<sub>6</sub>H<sub>4</sub>Cl·NO<sub>2</sub> (III) in presence of a catalyst (Cu) is carried out with sufficient of (III) to maintain the reaction mixture in a fluid condition at >160°. Preferably, a mixture of (I) and (II) (equimols.) and a little Cu is added gradually to (III) at 150—160°, and the mass is then heated at 185—205°. H. A. P.

Amino-derivatives of hydroxydiphenyls. W. D. WOLFE, Assr. to WINGFOOT CORP. (U.S.P. 2,004,914, 11.6.35. Appl., 18.12.30).—Additive compounds of  $C_6H_4Ph\cdotOH$  with primary aliphatic mono- and diamines, primary aromatic monoamines of the  $C_6H_6$  and  $C_{10}H_8$  series,  $CH_2Ph\cdot NH_2$ , cyclohexylamine (I), and  $(CH_2)_6N_4$  (II) are claimed. Examples are the compounds : o- $C_6H_4Ph\cdotOH$  (III) with  $(CH_2\cdot NH_2)_2$  (2:1) (formed in EtOH), m.p. 78—80°; (III) with (II), m.p. 161—162°;  $p-C_6H_4Ph\cdotOH$  (IV) with  $\beta$ - $C_{10}H_7\cdot NH_2$  (1:1), m.p. 130— 131°; 2(IV),  $C_2H_4(NH_2)_2$ , m.p. 137—138°; (IV), (I), m.p. 108—109°, and (IV), (II), m.p. 152°. H. A. P.

Preparation of  $\beta$ -form[alky1]amino- $\alpha$ -p-anisylpropanes. G. A. ALLES (U.S.P. 2,011,790, 20.8.35. Appl., 13.3.34).—p-OMe·C<sub>6</sub>H<sub>4</sub>·CH<sub>2</sub>·COMe, NH<sub>3</sub> or NH<sub>2</sub>R (or a salt; > 2 mols.), and HCO<sub>2</sub>H (or HCO<sub>2</sub>Na; > 2 mols.) at 115—170° give  $\beta$ -form-amido-, b.p. 195— 205°/6 mm., -benzylamido-, b.p. 190—200°/6—10 mm., -methylamido-, b.p. 210—220°/8 mm., and -ethylamido- $\alpha$ p-anisylpropane, b.p. 205—215°/4 mm., hydrolysed to the amines, the hydrochlorides of which have m.p. 203—205°, 223°, 176—177°, and 154—155°, respectively. R. S. C.

Manufacture of substituted aminochrysenes. I. G. FARBENIND. A.-G. (B.P. 449,182, 21.12.34. Ger., 22.12.33).—Dinitrochrysene (modified prep.) with hot Na<sub>2</sub>S-EtOH gives 2:8-diaminochrysene (I), m.p. 284— 286°, and with less Na<sub>2</sub>S and S in hot EtOH yields 2nitro-8-aminochrysene (II), m.p. 228—229°, hydrogenated (Ni) to (I) in  $C_5H_5N$  at 70—80°. 2-Acetamidochrysene and HNO<sub>3</sub> (d 1.5) in AcOH at 70—80° give the Ac derivative, m.p. 322—325°, of (II), which yields (II) by NaOH-EtOH, and by hydrogenation (Ni) in  $C_5H_5N$  at 70—90° affords 2-amino-8-acetamidochrysene, m.p. 266— 268°, hydrolysed to (I). R. S. C.

[Preparation of] amidines. J. LEE, Assr. to E. R. SQUIBB & SONS (U.S.P. 2,004,994, 18.6.35. Appl., 6.9.33).—Bis-(2-ethoxy-5-diphenylyl)acetamidine [hydrochloride, m.p. 120—125° (decomp.)] is prepared by

interaction of 5-amino-2-ethoxydiphenyl, its Ac derivative, and  $PCl_5$  in  $C_6H_6$ ; p-phenetyl- and p-carboxyethylphenyl-2-ethoxy-5-diphenylylacetamidine are similarly prepared. The products are claimed to have therapeutic properties. H. A. P.

Preparation of aromatic hydrazo compounds. H. J. WEILAND, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,012,234, 20.8.35). Appl., 2.3.34).—Hydrazo compounds, obtained by the Zn–NaOH–EtOH process, can be directly extracted with  $C_6H_6$  or PhMe if the  $H_2O$  content of the liquor is adjusted so as to coagulate the Zn residues. R. S. C.

Water-soluble diazoamino-compounds. M. A. DAHLEN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,008,750, 23.7.35. Appl., 27.7.32).-Stable, sol. diazoamino-compounds for use in dyeing and printing are obtained by causing diazonium compounds in slightly alkaline solution to react with a pyrrolidine derivative containing at least one solubilising group [e.g., the  $\alpha$ -CO<sub>2</sub>H- (I),  $\alpha$ -SO<sub>3</sub>H- (II), or  $\beta$ -hydroxya-carboxy-derivative (III)] and evaporating the solution to dryness at 40°. The examples include  $2:5-C_6H_3Cl_2\cdot NH_2 \rightarrow$ (I), (II), (III); 3:1:4- $NO_2 \cdot C_6 H_3 Me \cdot NH_2 \rightarrow (I).$ S. C.

Manufacture of *m*-cresol [-tolyl] methyl ether. W. V. WIRTH, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,005,815, 25.6.35. Appl., 23.2.34).—Me<sub>2</sub>SO<sub>4</sub> (0.55—0.6 mol.) is added gradually at 30—33° to an aq. solution of *m*-C<sub>6</sub>H<sub>4</sub>Me·ONa (1.0 mol.) The temp. is then raised to 97—100° and 30% aq. NaOH (0.2 mol.) is added at a rate just sufficient to maintain  $p_{\rm H}$  11.5— 12.5; the yield of *m*-C<sub>6</sub>H<sub>4</sub>Me·OMe is > 95%.

A. W. B. Manufacture of diphenyl ether. K. MARX and H. WESCHE, ASSTS. to WINTHROP CHEM. Co. (U.S.P. 2,008,987, 23.7.35. Appl., 16.5.33. Ger., 4.6.32).— Improved yields of Ph<sub>2</sub>O are obtained from PhCl (2 mols.) and aq. NaOH (1 mol.) by adding sufficient PhOH (> 3.5 mols.) to form a homogeneous reaction mixture; Cu is used as catalyst and the reaction effected at 340°/> 100 atm. P. G. C.

Manufacture of unsymmetrical dialkyl or alkylaralkyl derivatives of resorcinol. W. E. AUSTIN, Assr. to Redro Labs., Inc. (U.S.P. 2,006,039, 25.6.35. Appl., 14.9.32). -m-C<sub>6</sub>H<sub>4</sub>(OH)<sub>2</sub> is treated, in two stages, with acylating agents, e.g., acid chlorides, in presence of a condensing agent, e.g., ZnCl<sub>2</sub>, and the resulting diketones are reduced, e.g., with Zn-Hg, to dialkyl derivatives. The second acylation stage may be effected after reduction of the monoketone. Methylbutyl-, b.p. 160-170°/6-8 mm., -amyl-, b.p. 170-178°/ 6 mm., -hexyl-, b.p. 170-180°/5 mm., ethyl-propyl-, b.p. 140°/3 mm., -butyl-, 140-150°/3 mm., -amyl-, b.p. 170-175°/3 mm., -hexyl-, b.p. 172-182°/2 mm., -heptyl-, b.p. 175-185°/3 mm., benzylethyl-, b.p. 210-220°/6 mm., propyl-butyl-, b.p. 165-170°/3 mm., -amyl-, b.p. 170-180°/4 mm., -hexyl-, b.p. 180-190°/2 mm., -heptyl-, b.p. 185-200°/4 mm., butyl-amyl-, b.p. 175—190°/5 mm., -hexyl-, b.p. 195—205°/5 mm., -heptyl-, b.p. 170-190°/2 mm., and hexylheptyl-, b.p. 205-220°/3 mm., -resorcinol are described. A. W. B.

Manufacture of *p*-tolyl *p*-anisyl sulphide. T. B. JOHNSON, ASST. to SHARP & DOHME, INC. (U.S.P. 2,011,582, 20.8.35. Appl., 12.1.34. Cf. U.S.P. 1,976,732; B., 1935, 841).—*p*-Anisyl and *p*-hydroxyphenyl *p*-tolyl sulphide have b.p. 181—184°/4 mm. and 178—180°/3 mm., respectively. R. S. C.

Manufacture of aromatic or heterocyclic carboxylic acid amides of high mol. wt. Courts & Co., and F. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 448,788, 13.12.34).—An org. compound containing  $\checkmark 4$ condensed nuclei, e.g., pyrene (I), chrysene, perylene (II), fluoranthene (III), triphenylene (IV), naphthanthracene, or brascane (V), or their halogen, alkyl, aralkyl, or aryl substitution products (1 mol.), is caused to react with a carbamyl (2 mols.), or a mixture which acts as such, e.g., KCNO and gaseous HCl, and a catalyst of the Friedel-Crafts type (1 mol.), if desired in an inert solvent or diluent, e.g., CS2, to yield carboxylic amides, which may be hydrolysed to the free acids by KOH-EtOH. Examples are: (I), 3-chloro-, 3- or 4-methylpyrene, (II), naphthofluorene, isochrysofluorene, 1:2benzanthracene, naphthacene, or picene with NHMe COCl, and (III), (IV), or (V) with NH2 COCl. (IV) gives triphenylenecarboxylamide, m.p. 286°, hydrolysed to triphenylenecarboxylic acid, m.p. 325-326°

N. H. H.

Manufacture of a new hydroxycarboxylic acid and derived amides. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 449,045, 11.3.35).—An alkali salt (Na or K) of 5-hydroxyhydrindene is heated with  $CO_2$  at > 1 atm. to yield the o-carboxylic acid, m.p. 198° (anilide, m.p. 187°;  $\beta$ -, m.p. 229°, and  $\alpha$ -naphthylamide, m.p. 210°; p-chloroanilide, m.p. 230°; 4-chloro-2:5-, m.p. 195°, and 3-chloro-4:6-dimethoxyanilide, m.p. 210°; 4-phenylanilide, m.p. 225°; benzidide, m.p. 285°; 2:2' dichlorobenzidide, m.p. > 300°). N. H. H.

Manufacture of nitro-derivatives of 1-sulpho-2hydroxynaphthalene-3-carboxylic acid. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 445,857, 8.10.34).—Nitration of 1-sulpho-2: 3-hydroxynaphthoic acid gives the 6- and 8-NO<sub>2</sub>-derivatives. The corresponding *Me* and *Et carboxylic esters* are obtained by nitrating the appropriate esters. A. W. B.

(A) Halogenated polyphenyl ketone [benzophenone]. (B) Halogenated material [diphenylmethane]. F. M. CLARK, and W. M. KUTZ, ASSTS. to GEN. ELECTRIC CO. (U.S.P. 2,012,301-2, 27.8.35. Appl., 4.4.33).-(A) COPh<sub>2</sub>, prepared by interaction of C<sub>6</sub>H<sub>6</sub> (1 mol.), CCl<sub>4</sub> (2 mols.), and AlCl<sub>3</sub> ( $\frac{1}{2}$  mol.) and hydrolysing the product, is chlorinated at 150-160° in presence of Fe to give non-cryst. solid or liquid Cl<sub>2</sub>- to Cl<sub>6</sub>derivatives. The Cl<sub>5</sub>-derivative has  $\varepsilon$  8.2 at 50° (1000 cycles). (B) Chlorinated derivatives (Cl<sub>2</sub>--Cl<sub>5</sub>) of CH<sub>2</sub>Ph<sub>2</sub> are prepared by condensing Cl-derivatives of CH<sub>2</sub>PhCl and C<sub>6</sub>H<sub>6</sub> in presence of AlCl<sub>3</sub>. The products of (A) and (B) have valuable electrical properties. S. C.

**Preparation of alkylated aromatic ketones.** H. A. BRUSON and O. STEIN, ASSTS. to RÖHM & HAAS Co. (U.S.P. 2,004,069, 4.6.35. Appl., 14.5.31).—An aromatic hydrocarbon having  $\not < 2$  free nuclear positions is condensed simultaneously with an alkyl halide or olefine

and an acyl halide or acid anhydride in presence of  $AlCl_3$ etc. *E.g.*, interaction of *n*-amyl chloride (I),  $C_6H_6$ ,  $Ac_2O$ (or AcCl), and  $AlCl_3$  in  $C_2H_4Cl_2$  gives a *C*-amylacetophenone, b.p. 121—123°/3 mm. A similar product is prepared by using monochlorinated pentane (from natural gas) in the place of (I). H. A. P.

Preparation of terpene esters of dicarboxylic acids. J. N. BORGLIN, Assr. to HERCULES POWDER Co. (U.S.P. 2,011,707, 20.8.35. Appl., 4.6.32).—Mixed and *s*-neutral esters are obtained by heating *sec.*-alcohols, *e.g.*, borneol, fenchyl alcohol, with dibasic acids, *e.g.*,  $o-C_6H_4(CO_2H)_2$ , in PhMe and completing the esterification (if necessary) with BuOH. R. S. C.

Production of sulphonated terpene products. A. L. RUMMELSBURG, Assr. to HERCULES POWDER Co. (U.S.P. 2,003,471, 4.6.35. Appl., 28.5.32).—A polymerised terpene is sulphonated in presence of another org. compound capable of undergoing sulphonation, *e.g.*, unsaturated higher fatty acids, higher aliphatic alcohols, aromatic compounds ( $C_{10}H_8$ ). The products are wetting agents. H. A. P.

Manufacture of benzanthrone derivatives. IMP-ERIAL CHEM. INDUSTRIES, LTD., I. M. HEILBRON, and F. IRVING (B.P. 447,546, 20.11.34).— $\alpha\beta\gamma\gamma$ -Tetrachloropropylene is condensed with anthrone, anthranol, or a chloro- or bromo-derivative of either, in presence of an acid condensing agent (conc. H<sub>2</sub>SO<sub>4</sub>). Anthrone gives 2-chlorobenzanthrone, m.p. 192°; 4-chloro-10-anthrone gives 2:6- and 2:8-dichlorobenzanthrone, m.p. 176° and 240°, respectively; 2:6:11- (m.p. 190—191°) and 2:8:11- (m.p. 180—181°) -benzanthrones are obtained from 1:5- and 1:4-dichloroanthrones. C. H.

Purification of (A, C) 4-halogeno-1-N-methylanthrapyridones, (B) 4-bromo-1-N-methylanthrapyridone. (A) E. C. BUXBAUM, (B) H. R. LEE and E. C. BUXBAUM, (C) H. J. WEILAND and E. C. BUXBAUM, Assrs. (A—C) to E. I. DU PONT DE NEMOURS & Co. (U.S.P. [A] 2,012,228, [B] 2,012,233, [C] 2,012,235, 20.8.35. Appl., 22.10.34).—4-Halogeno- (e.g., -bromo-) 1-methylanthrapyridone is purified by (A) treatment with NaNO<sub>3</sub> or NO·SO<sub>3</sub>H in 93% H<sub>2</sub>SO<sub>4</sub> at  $< 160^{\circ}$  (best 25—80°), (B) pptn. from H<sub>2</sub>SO<sub>4</sub> by dilution to 78% acid with or without treatment with NaNO<sub>2</sub>, or (C) oxidation of the impurities (mainly 4-bromo-1-methylaminoanthraquinone) by MnO<sub>2</sub> at 80—100° or Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> at 120° in 93% H<sub>2</sub>SO<sub>4</sub>. R. S. C.

Production of aromatic compounds containing selenium, nitrogen, and sulphur. E. and P. THEO-BALD (B.P. 447,646, 22.5.36. Ger., 30.8.34).—The compound, NHPh·NH·C<sub>6</sub>H<sub>4</sub>·Se·C<sub>6</sub>H<sub>4</sub>·NH<sub>2</sub>,PhSO<sub>3</sub>H, m.p. 211—212°, is obtained by allowing PhSO<sub>2</sub>·NHPh to react with liquid H<sub>2</sub>SeO<sub>3</sub> at room temp. The process is applicable generally to benzenesulphonarylides. S. C.

Manufacture of 3-aminoquinoline derivatives. I. G. FARBENIND. A.-G. (B.P. [A] 448,502, 28.11.34, and [B] Addn. B.P. 448,504, 30.11.34. Ger., 15.12.33).— (A) 3-Hydroxyquinolines (I) are heated with anhyd. or aq. NH<sub>3</sub> at 150—300°/>1 atm. in presence or absence of  $(NH_4)_2SO_4$ . Examples are : 3-amino-2-methyl-, 3-amino-6-phenyl-2-methyl-, m.p. 217—218°, and 3-amino-2-phenyl-, m.p. 120—121°, -quinoline. When (I) contains a 4-CO<sub>2</sub>H.

 $CO_2$  is eliminated during the reaction. (B) (I) are heated with aliphatic, aromatic, or alicyclic primary or sec. bases at 180—300°/>l atm., with or without a catalyst. Examples are: 3-hydroxy-2-methylquinoline (or its 4-carboxylic acid) with (a) aq. NH<sub>2</sub>Me gives 3-methylamino-2-methyl-, m.p. 93—94° (picrate, m.p. 233—234°), (b) aq. NHMe<sub>2</sub> gives a product, m.p. 90—91° (picrate, m.p. 230—231°), and (c) NH<sub>2</sub>Ph gives 3-anilino-2-methyl-, m.p. 120—121° (hydrochloride, m.p. 235—236°), -quinoline. N. H. H.

Preparation of N-p-aminophenylmorpholine. (A) H. A. LUBS, (B) H. A. LUBS and G. C. STROUSE, ASSIS. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,004,763-4, 11.6.35. Appl., [A] 11.6.32, [B] 14.6.32).-1-p-Nitro- or 1-p-nitroso-phenylmorpholine is reduced (A) with Zn and a "non-oxidising" acid (HCI), and (B) with an alkali sulphide (I) or (I) + NH<sub>4</sub>Cl. 1-p-Aminophenylmorpholine has m.p.  $128\cdot8-130\cdot2^{\circ}$  (Ac derivative, m.p.  $211\cdot2-212\cdot4^{\circ}$ ). H. A. P.

Production of N-p-β-naphthylaminophenylmorpholine. G. C. STROUSE, ASST. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,005,571, 18,6.35. Appl., 25.6.32).—Interaction of β- $C_{10}$ H<sub>7</sub>·OH, 1-p-aminophenylmorpholine, and aq. NaHSO<sub>3</sub> at the b.p. (70 hr.) gives 1-p-β-naphthylaminophenylmorpholine, m.p. 188—189°. H. A. P.

Heat-insulating materials.—See I. Tar acids. Hydrocarbons. Metallo-organo-derivatives. Lubricants.—See II. Anthraquinone derivatives. Cœroxene and cœrbioxene compounds.—See IV. Washing etc. agents.—See XII. Aliphatic-aromatic amines. *iso*Butylchlorophenols. Glyoxalines.— See XX.

#### IV.—DYESTUFFS.

Vat dyes containing only carbon, hydrogen, and oxygen. R. F. THOMSON (J. Soc. Dyers and Col., 1936, 52, 247-252).—A lecture. S. C.

Anthraquinone dyes. R. F. THOMSON (J. Soc. Dyers and Col., 1936, 52, 237-247).—A lecture. S. C.

Solubility and density of some malachite-greens. M. T. SALAZAR and A. SOSA (Anal. Fís. Quím., 1935, 33, 861-863).,  $d^{99}$  (by displacement of PhMe or vaseline) and solubility in CCl<sub>4</sub>, Et<sub>2</sub>O, CS<sub>2</sub>, PhMe, and vaseline of tetramethyldiaminotriphenylmethane sulphate, oxalate, and chlorozincate (malachite-green) are recorded. F. R. G.

#### PATENTS.

Manufacture of [mono]azo dyes. Soc. CHEM. IND. IN BASLE (B.P. 447,561, 23.2.35. Switz., 23.2.34 and 22.2.35).—4-Nitro- or 4 : 6-dinitro-2-cyanoaniline is diazotised and coupled with an arylamine of the  $C_6H_6$  or  $C_{10}H_8$  series. Examples are : 4-NO<sub>2</sub>-compound  $\rightarrow$ ethyl- $\beta$ -methoxyethylaniline (reddish-violet on acetate silk), NN-di- $\beta$ -methoxyethylcresidine (I) (violet), or 1 : 5-aminonaphthol (greenish-blue); 4 : 6-(NO<sub>2</sub>)<sub>2</sub>-compound  $\rightarrow$  (I) (blue), NN-di- $\beta$ -methoxyethyl-2 : 5dimethoxyaniline (blue); 4-NO<sub>2</sub>-compound  $\rightarrow$   $\gamma$ -acid in acid medium (violet on woof). C. H.

Manufacture of monoazo dyestuffs. W. W. GROVES. From J. R. GEIGY A.-G. (B.P. 448,601, 4.10.34).— Dyestuffs giving bright red-violet shades fast to fulling. light, and sea-H<sub>2</sub>O on wool, silk, and lacquers are obtained by coupling 1-amino-2: 4-diphenoxybenzene, b.p. 240— 250°/18 mm. [made by reducing the product from  $C_6H_3Cl(NO_2)_2$  and PhOH at 130—150°], or the corresponding 2': 2''- or 4': 4''-Me<sub>2</sub> and 4': 4''-Cl<sub>2</sub>-derivatives with 1-benzyl-, 1-phenyl-, or 1-p-tolyl-sulphonamido-8-hydroxynaphthalene-3: 6- or -4: 6-disulphonic acids.

Manufacture of [acid] (A) monoazo, (B) azo, dyes. I. G. FARBENIND. A.-G. (B.P. 447,522 and 448,131, [A] 12.7.35, [B] 3.12.34. Ger., [A] 14.7.34, [B] 1.12.33).-(A)  $\gamma$ -Acid is coupled (acid) with a diazotised aniline*m*-sulphonic acid or amide carrying in *p*-position ·NRAc or  $\cdot$ NRBz, where R = alkyl, aralkyl, Ph, or cyclohexyl, e.g., 4-amino-N-cyclohexylacetanilide-2-sulphonic acid (scarlet on wool or silk), the N-Me compound (scarlet) or its methylamide (yellowish-red). (B) A 1: 3-diaryl-5-pyrazolone carrying solubilising groups is coupled with a diazo component. Examples are: 3-phenyl-1-psulphophenyl-5-pyrazolone (I)  $\leftarrow$  p-toluidine-3-sulphonanilide (orange-yellow) or Cleve acid (brown); 3-pcarboxyphenyl-1-o-chloro-p-sulphophenyl-5 - pyrazolone < sulphanilic acid (reddish-yellow); 3-phenyl-l-(2'-hydroxy-3'-carboxy-5'-sulphophenyl)-5-pyrazolone + anthranilic acid (yellow); (I)  $\leftarrow$  o-aminophenol (+ Cr, brownish-red); 3-phenyl-1-p-phenoxy-m-sulphophenyl-yellow). C. H.

[Manufacture of] oil-soluble azo dyes. R. B. PAYNE, ASST. to NAT. ANILINE & CHEMICAL CO., ING. (U.S.P. 1,986,116, 1.1.35. Appl., 21.12.32).— $\beta$ -C<sub>10</sub>H<sub>7</sub>·OH is coupled with an aminoazoxylene derived substantially from o-xylidines. C. H.

Manufacture of heavy-metal complex compounds of azo dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 446,746, 31.10.34).-Heterocyclic amines containing the grouping .N.C(NH2).NH., e.g., aminotetrazole (I), phenylguanazole (II), 3-amino-1:2:4triazole-5-carboxylic acid (III), 3-amino-5-methyl-1:2:4-triazole (IV), or 3-aminotriazole (V), are diazotised and coupled with suitable components. The heterocyclic ·NH· is sufficiently acidic to confer solubility and (with the azo grouping) chromability. Examples are: (I)  $\rightarrow$  1-o-chlorophenyl-3-methyl-5-pyrazolone (yellow on wool; + Cr); (II)  $\rightarrow \gamma$ -acid (yellowish-red; + Cr, reddish-brown); [(II)  $\rightarrow$  J-acid]<sub>2</sub>  $\leftarrow$  benzidine-3:3'-dicarboxylic acid (VI) (violet on cotton; + Cu, navy-blue); (I)  $\rightarrow 2:$  3-hydroxynaphthoic  $\beta$ -naphthylamide (orange on cotton; + Ni, red);  $(I) \rightarrow$  resorcinol]  $\leftarrow$  benzidine (reddish-brown on cotton; + Cu);  $(I) \rightarrow J$ -acid  $\leftarrow$  benzidine  $\rightarrow$  salicylic acid (VII) (blackishbrown; + Cu); (III)  $\rightarrow$  (VII) (+ Cr, yellowish-brown on wool); (III)  $\rightarrow$  8-aminoquinaldine  $\rightarrow$  phenyl-J-acid (violet on cotton; + Cu, greenish-blue); [(IV)  $\rightarrow$  J-acid]<sub>2</sub>  $\leftarrow$  dianisidine (+ Cu, navy-blue); [(V)  $\rightarrow$  $m - C_6 H_4 (NH_2)_2]_2 \leftarrow (VI) (+ Cu, brown).$ C. H.

Manufacture of [azo] dyes containing chromium. COMP. NAT. DE MAT. COL. ET MANUF. DE PROD. CHIM. DU NORD RÉUNIES ETABL. KUHLMANN (B.P. 446,820, 31.10.34. Fr., 2.12.33).—Chromed o-hydroxyazo dyes (other than o-aminophenols  $\rightarrow$  pyrazolones), or mixtures containing such dye or dyes, are treated with acid, before or after isolation from the chroming solution, until wholly or partly converted into "sec. chromed compounds" which are acid-resistant. Suitable chromed dyes are those from 1:2:4-aminonaphtholsulphonic acid and  $\beta$ -C<sub>10</sub>H<sub>7</sub>·OH, with or without 1-phenyl-3-methyl-5-pyrazolone. C. H.

Production of o-hydroxyazo dyes containing chromium. F. STRAUB and W. WIDMER, Assrs. to Soc. CHEM. IND. IN BASLE (U.S.P. 1,959,507, 22.5.34. Appl., 22.5.33. Switz., 9.6.32).—An o-aminophenol carrying in position 4 halogen, alkyl, or alkoxyl is diazotised and coupled with a 1-sulphoaryl-3-methyl-5-pyrazolone, 1 or 2 further SO<sub>3</sub>H groups being present, and chromed. Examples are : 4-chloro-o-aminophenol-5-sulphonic acid  $\rightarrow$  1-m- or -p sulphophenyl-3-methyl-5-pyrazolone, + Cr (red on wool), or 1-(4'-sulpho  $\alpha$ -naphthyl)-3-methyl-5-pyrazolone, + Cr (red).

C. H.

Azo dyestuffs [containing copper]. IMPERIAL CHEM. INDUSTRIES, LTD., and F. L. ROSE (B.P. 446,520, 30.10.34),-2:5:7-NH<sub>2</sub>·C<sub>10</sub>H<sub>5</sub>(OH)·SO<sub>3</sub>H (I) or an N-substituted derivative thereof, e.g., 2-benzamido-, 5:5'-dihydroxy-2:2'-dinaphthylamine-7:7'-disulphonic acid (II), 5:5'-dihydroxy-2:2'-dinaphthylurea-7:7'disulphonic acid (III), is coupled completely in alkaline media with a diazotised 2-amino-5-aroylaminobenzoic acid (IV) or, where the coupling component contains two residues of (I), as in (II) and (III), with 1 mol. of (IV) and 1 mol. of another diazotised o-amino-carboxylic acid or -phenol, followed by treatment in substance or on the fibre with a Cu salt. Bordeaux to violet shades are obtained. Examples of (IV) are 2-amino-5-benzamido-, m.p. 245-250° (decomp.), 2-amino-5-(4'-toluylamido)-, m.p. 244-245° (decomp.), 2-amino-5-(2'-chlorobenzamido)-, m.p.190-195° (decomp.), 2-amino-5-(4'-anisamido)-, m.p. 246° (decomp.), -benzoic A. W. B. acid.

Manufacture and application of a new coppercontaining azo dyestuff. IMPERIAL CHEM. INDUSTRIES, LTD., and M. MENDOZA (B.P. 446,425, 29.10.34).— Diazotised 2:6:4-NH<sub>2</sub>·C<sub>6</sub>H<sub>3</sub>Cl·SO<sub>3</sub>H (2 mols.) is coupled with 5:5'-dihydroxy-2:2'-dinaphthylamine-7:7'-disulphonic acid (1 mol.) in aq. alkali and the resulting dye treated with Cu or an agent yielding Cu. The product dyes cotton in violet shades of good lightfastness. A. W. B.

Manufacture of azo dyes on the fibre [ice colours]. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 447,611, 14.11.34).—The alkali in the grounding liquor is replaced wholly or in part by an amine, NHRR', in which R = aliphatic group  $\Rightarrow C_4$  other than acyl, and R' = H or R, or by an alkaline-reacting salt of such amine. Examples are : dicyano-diamide, guanidine carbonate, NHEt<sub>2</sub>. Such grounding solutions are suitable for ice colours on acetate silk, or for developing arylamines diazotised on acetate silk. C. H.

Manufacture of azo dyes [for acetate silk]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 447,911, 26.11.34).—Diphenylhydroxyalkylamines are coupled with diazotised arylamines, especially those containing NO<sub>2</sub> and/or halogen, both components being free from  $CO_2H$  and  $SO_3H$ . Examples are: p-NH<sub>2</sub>: $C_6H_4$ :NO<sub>2</sub> (I)  $\rightarrow$  diphenyl- $\beta$ -hydroxyethylamine (II) (orange); 2-chloro-p-nitroaniline  $\rightarrow$  (II) (red); 2:4-dinitroaniline  $\rightarrow$  (II) (reddish-violet); (I)  $\rightarrow$ 4-methyldiphenyl -  $\beta$  - hydroxyethylamine (reddishorange); (I)  $\rightarrow$  4 - ethoxydiphenyl -  $\beta$  - hydroxyethylamine (brown). C. H.

Manufacture of azo dyes [for acetate silk]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 446,745, 31.10. and 11.12.34).—An aromatic nitrodiazo compound is coupled with 5- or 6-membered heterocyclic compounds derived from 1:5-aminonaphthol (I). Examples are : 3:7-dihydroxy-1:2:3:4tetrahydro- $\alpha$ -naphthoquinoline [from (I) with epichlorohydrin]  $\leftarrow p$ -NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·NO<sub>2</sub> (blue), 5-nitroo-anisidine (blue), 5-nitro-o-aminophenol (greenish-blue) or 6-bromo-2:4-dinitroaniline (bluish-green); 7-hydroxy-1:2:3:4-tetrahydro -  $\alpha$  - naphthoquinoline (from Laurent acid by Skraup reaction, hydrogenation, and alkaline fusion)  $\leftarrow p$ -NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·NO<sub>2</sub> (greenish-blue).

C. H.

Manufacture of azo dyes [for leather]. I. G. FARBENIND. A.-G. (B.P. 447,775 and 447,814, 24.11.34. Ger., 25.11.33).—A monoazo dye, NH<sub>o</sub>Ar  $\rightarrow$  Ar'OH, in which Ar = aryl of the  $C_6H_6$  or  $C_{10}H_8$  series carrying in ortho position OH, alkoxyl, halogen, or CO2H, and Ar' is Ph carrying in positions 2 and 4 or 6 OH, NH2, or substituted NH, and in position 6 or 4 H or a substituent, is coupled with a diazo compound, and, if desired, coupled again with a diazo compound, the dye being (A) coppered, (B) converted into other heavy-metal complex, before or after the last coupling. Examples are: (A) o-anisidine-4-sulphonic acid (I)  $\rightarrow$  resorcinol (II),  $+ Cu, \leftarrow m \cdot NH_2 \cdot C_6 H_4 \cdot NO_2$  (brown); (I)  $\rightarrow$  (II)  $\leftarrow$ (I), + Cu (brownish-red); 6-chloroaniline-3-sulphonic  $acid \rightarrow (II), + Cu, \leftarrow dehydrothiotoluidinesulphonic acid$ (III) (brown); (I)  $\rightarrow$  (II), + Cu,  $\leftarrow$  4-nitro-*m*-phenylenediamine  $\leftarrow$  (I) (yellowish-brown); 2(I)  $\rightarrow$  (II)  $\leftarrow$ p-NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·NO<sub>2</sub>, + Cu (reddish-brown); (B) 6-chloroo-aminophenol-4-sulphonic acid  $\rightarrow$  (II)  $\leftarrow$  (III), + Cr (reddish-brown); anthranilic acid  $\rightarrow$  (acid) *m*-aminophenol, + Cr,  $\leftarrow$  (alk.) *p*-nitroaniline-3-sulphonic acid (yellowish-red); nitro-1:2:4-aminonaphtholsulphonic acid  $\rightarrow$  (II), + Cr,  $\leftarrow$  naphthionic acid (grey to greyishblack). C. H.

Manufacture of coloured products [cellulose derivatives pigmented with vat dyes]. BRIT. CELANESE, LTD. (B.P. 447,862, 25.7.35. U.S., 25.7.34).— A leuco-vat dye is oxidised in solution together with a cellulose derivative; e.g., a 55—65% solution of cellulose acetate in COMe<sub>2</sub> is mixed with leuco-compound and air-oxidised to give a finely-divided vat-dye prep. for colouring spinning solutions, or the COMe<sub>2</sub> may be removed, leaving a solid prep. The leuco-compound may be prepared in COMe<sub>2</sub> from vat dye and a Zn-Cu or Al-Cu couple. C. H.

Manufacture of dye-sulphonic acids [sulphonated diarylaminobenzoquinones]. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 447,899, 21.9.34).— 2 : 5-Diarylamino-p-benzoquinones, in which the aryl group is sulphonated and contains > 2 nuclei and alkyl, aryl, or halogen may occupy positions 3 and 6,

are treated with  $H_2SO_4$ ,  $ClSO_3H$ , oleum, or an alkaline condensing agent, preferably in presence of an oxidant. Suitable starting materials are products from chloranil and : 3-amino-9-ethyl- or -methyl-carbazole-6-sulphonic acid (blue on wool, cotton, or viscose) ; 2-aminochrysene-8-sulphonic acid (greenish-blue) ; 2-aminofluorenedisulphonic acid (violet) ; 6-sulpho-2-aminodiphenylene oxide (bluish-red). C. H.

Manufacture of [vat] dyes [of the pyrenequinone series]. Soc. CHEM. IND. IN BASLE (B.P. 447,890, 8.7.35. Switz., 7.7.34 and 8.6.35).-A tetrahalogenated pyrenequinone is condensed with a primary or sec. amine. Examples are : tetrabromopyrenequinone, m.p. 350-355°, with 0.36 pt. of NH<sub>2</sub>Ph (grey-green vat dye, m.p. 325°) or 21 pts. of  $NH_2Ph$  (greenish-olive); tetra-chloropyrenequinone, m.p. 338°, with  $NH_2Ph$  (1.6 pts., blue, m.p. 305°; 11 pts., olive-grey, m.p. 250°); tetrachloropyrenequinone, m.p. 355°, with NH,Ph (greygreen, m.p. 295-300°); tetrachloropyrenequinone, m.p. 350°, with  $\beta$ -C<sub>10</sub>H<sub>7</sub>·NH<sub>2</sub> (brownish-violet, m.p. > 380°; or olive, m.p. 283-285°), or NH2Ph (green, m.p. 285-290°), or 2 : 5-dichloroaniline (olive-black, decomp. 330°); tetrachloropyrenequinone, m.p. 360°, with dianisidine (olive-green, m.p.  $> 400^{\circ}$ ); tetrachloropyrenequinone, m.p. 362°, with 3-chloro-p-toluidine (yellowish-olive, m.p. 285-290°). C. H.

Manufacture of (A, B) N-substitution products of 1:4-diaminoanthraquinones, (C, D) anthraquinone derivatives. W. W. GROVES, COUTTS & Co., and F. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 447,037, 447,088, and 447,107-8, [A, C, D] 3.8.34, [B] 3.5.35).-NN'-Substituted 1: 4-diaminoanthraquinones are prepared by usual methods, the substituents being (A) alkyl hydrocarbon, R, and hydroxyalkyl, R', R + R'being  $\Rightarrow C_6$ , (B) two different alkyl hydrocarbon groups, R and R", R + R" being  $\Rightarrow C_6$ , (c and D) two different alkyl, cycloalkyl, or aralkyl groups, R" and R", the starting material being (C) a leucoquinizarin, which may be produced in situ, or (D) a leuco-1-hydroxy- or 1-aminoanthraquinone, carrying NHR" in position 4, which also may be produced in situ. Examples are anthraquinones having the following substituents: (A) 1-methylamino-4-β-hydroxyethylamino- (I), m.p. 190°, from 4-bromo-1-methylaminoanthraquinone (II) and NH2 [CH2]2 OH (blue on acetate silk); 1-ethylamino-4- $\beta$ -hydroxyethylamino-, m.p. 280° (blue); (B) 1-methylamino-4-n-butylamino- (blue), from (II) and NH2Bua; (c) (I), from quinizarin and Zn (or leucoquinizarin) with NH2Me and NH2 [CH2]2 OH; 1-methylamino-4-β-phenylethylamino- (blue), from leucoquinizarin, NH2Me, and C2H4Ph·NH2; 1-cyclohexylamino-4β-hydroxyethylamino- (blue) and 1-methylamino-4-βhydroxyethylamino-5: 8-dihydroxy- (greenish-blue) similarly; (D) (I), from leuco-1-methylamino-4hydroxyanthraquinone. [(A) Stat. ref.] C. H.

[Preparation of] (A) anthraquinone derivatives [ $\beta$ -phenylbenzanthrones], (B) [derived] dyes of the dibenzanthrone series. O. M. BISHOP and A. J. WUERTZ, ASSTS. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 2,005,529 and 2,005,574, 18.6.35. Appl., [A] 2.5.30, [B] 3.8.34).—(A) 2-Phenyl-, -chloro- or -hydroxy-phenyl-anthraquinone is submitted to the benzanthrone

condensation. E.g., 2-p-chlorophenylanthraquinone with glycerol, Cu powder, and 80—83%  $H_2SO_4$  at 112—120° gives three isomeric p-chlorophenylbenzanthrones, m.p. 131·8—134·4°, 102·4—103·6°, and 160—162°, respectively. Similar products are obtained from 2-o- and 2-m-chlorophenylanthraquinone. (B) The above products are converted into dibenzanthrone derivatives by fusion with KOH-EtOH (at 140—160°); the resulting dichlorodiphenyldibenzanthrones dye cotton greenishblack. H. A. P.

Manufacture of anthraquinone vat dyes. IMPERIAL CHEM. INDUSTRIES, LTD., F. IRVING, and C. SHAW (B.P. 446,910 and 447,545, [A] 7.11.34 and 4.9.35, [B] 20.11.34). —(A) The carbazole from 4-amino-4'-benzamido-1:1'dianthraquinonylamine is acylated with a thiazoloanthrone-3-carboxylic halide; or the diacylated diamino-1:1'-dianthraquinonylamine is carbazolated. The product gives olive-green shades. (B) Caledon Olive R is treated with 96%  $H_2SO_4$  or other agent to remove 1 Bz group, then acetylated, and cyclised to the pyridone (brown vat dye). C. H.

Production of [acid] anthraquinone colouring matters. J. H. CROWELL, J. OGILVIE, and D. G. ROGERS, ASSTS. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,960,233, 29.5.34. Appl., 22.6.28).—The 2-chloroquinizarin from phthalic anhydride and dichlorinated phenol is condensed with 2 mols. of arylamine (NH<sub>2</sub>Ph) to give the 2:4-diarylamino-1-hydroxyanthraquinone, which is disulphonated (grey to black on chromed wool). C. H.

Manufacture of [acid] anthraquinone dyes. IMPERIAL CHEM. INDUSTRIES, LTD., N. H. HADDOCK, F. LODGE, and C. H. LUMSDEN (B.P. 446,819, 31.10.34).-The  $\alpha$ -halogen in a 1-chloro- or -bromo-anthraquinone carrying in position 4 NH<sub>2</sub>, NHMe, or OH is replaced by NHAr in which Ar = Ph carrying an alkyl side-chain C4-C7, and SO3H is introduced into the product if necessary. Examples are: 1:3-dibromo-4-aminoanthraquinone with p-NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>Bu<sup>\*</sup> or p-NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·C<sub>5</sub>H<sub>11</sub>-(iso), and then with Na<sub>2</sub>SO<sub>3</sub> (blue); 1-chloro-4-hydroxyanthraquinone with p-NH2.C6H4.C5H11(iso) (product, m.p. 95°), sulphonated with oleum (violet); 1-bromo-4-aminoanthraquinone-3-sulphonic acid with p-C<sub>6</sub>H<sub>4</sub>Bu<sup>a</sup>·NH<sub>2</sub> (blue). C. H.

[Manufacture of acid] anthraquinone dyes. IMPERIAL CHEM. INDUSTRIES, LTD., N. H. HADDOCK, F. LODGE, and C. H. LUMSDEN (B.P. 449,010-2, 14.11.34. [c] Addn. to [A]. [B] Addn. to B.P. 446,819; above).—Anthraquinone acid dyes for animal fibres, especially fast to severe washing and milling, are prepared by interaction of (A) a primary alkylaniline  $(C_4 - C_7)$  with a leucoquinizarin (or a mixture of this and the corresponding quinizarin) (I), or a 1:4-dihalogenoanthraquinone (II), or leuco-1-hydroxy- (or a mixture of this and the corresponding 1-hydroxy-), or 1-halogeno-4-arylamino-anthraquinone, (B) a primary alkoxy-(OPr. to C20Ha1.O) or alkylthiol- (SPr to C7H15S) -aniline with a 1-chloro- or 1-bromo-4-amino-, -4-methylamino-, or -4-hydroxy-anthraquinone, (c) a primary alkoxyaniline (OPr to C<sub>9</sub>H<sub>19</sub>·O) with (I) or (II) and (A-c) if necessary sulphonating or, in suitable cases,

replacing a 3-halogen by SO<sub>2</sub>H by treatment with a sol. sulphite. Examples are : condensation of (A) leucoquinizarin and p-butyl- (III), -isoamyl -(IV), or -heptyl- (V) -aniline gives 1: 4-di-p-butyl-, -isoamyl-, or -heptyl-anilinoanthraquinones, leuco-1:4:5-trihydroxyanthraquinone and (III), (IV), or (V) give 1-hydroxy-5:8-di-p-butyl-, -isoamyl-, or -heptyl-anilinoanthraquinones, 1:4:5:8-tetrachloroanthraquinone and (III) give 1: 4-dichloro-5: 8-di-p-butylanilinoanthraquinone, 1-p-toluidino-4-hydroxyanthraquinone and its leucocompound and (III) give a compound ; (B) 1 : 3-dibromo-4-aminoanthraquinone and p-dodec-, (VI), -but-, (VII) or -oct- (VIII) -oxyaniline or 4-NH2 C6H4 SBu give 2-bromo-1-amino-4-p-dodec-, -but-, or -oct-oxy-, or butylthiol--anilinoanthraquinone; 1-chloro-4-hydroxyanthraquinone and (VI), (VII), or (VIII) give 1-p-dodec-,-but-, or -oct-oxyanilino-4-hydroxyanthraquinone. (c) Leucoquinizarin and p-but-(IX), -isoamyl-(X), or -oct-oxyaniline give 1:4-dip-butyl-, -isoamyl-, or -oct-oxyanilinoanthraquinone; leuco-1:4:5-trihydroxyanthraquinone and (IX), (X), or p-heptoxyaniline give 1-hydroxy-5:8-di-p-but-, -isoamyl-, or -hept-oxyanilinoanthraquinone; 1:4:5:8-tetrachloroanthraquinone and (IX) give a compound. These are converted into sulphonic acids to give violet to green dyestuffs. N. H. H.

Manufacture of acid wool dyes. W. W. GROVES, and I. G. FARBENIND. A.-G. (B.P. 447,608, 16.10.34. Addn. to B.P. 299,721; B., 1930, 455).-An aminonaphthalic acid or anhydride is condensed with a polynuclear aromatic or hydroaromatic compound having in different nuclei an NH2-group and a chelate grouping, and the product is sulphonated if it does not already contain SO<sub>3</sub>H; or a nitronaphthalic acid or anhydride is so condensed and the NO, subsequently reduced (with or without simultaneous introduction of SO<sub>2</sub>H). The products may be chromed in substance. Examples are : 4-aminosulphonyl-1: 8-naphthalic acid with N-maminobenzenesulphonylanthranilic acid (yellow on wool; + Cr), 5-(p-toluidine-2'-sulphonylamino)salicylic acid (yellow; + Cr, redder), 4'-amino-4-hydroxy-3-methyldiphenylmethane-5-carboxylic acid (+ Cr, yellow), or 5-m-aminobenzamidosalicylic acid (+ Cr, yellow); 4nitro-1: 8-naphthalic anhydride with 4-hydroxy-3carboxybenzenesulphon-p-phenylenediamide, boiled with aq. NaHSO<sub>3</sub> (+ Cr, yellow). C. H.

Decanting indigo slurries. D. W. VAN DONGEN, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,987,879, 15.1.35. Appl., 13.10.30).—The dil. aq. alkaline slurry containing about 2% of indigo separates into layers most rapidly at about 90°. C. H.

Manufacture of compounds of the cœroxene and cœrbioxene series [oil colours]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 448,180, 22.11.34).— A cœroxonol or cœroxenol is reduced in an alkaline medium until insol. in alkali. Thus, 4:14-dimethylcœroxonol (from phthalic anhydride and *p*-cresol in H<sub>2</sub>SO<sub>4</sub>, anhydrised with oleum) is reduced with Zn dust and NaOH, or electrolytically in alkaline medium. 4:14-Ditert.-butyl-, 3:13(or 15)-dimethyl-, 3:4:13:14tetramethyl-, 2:16-dichloro-3:13-dimethyl-, and 4:14-dichloro-cœroxonols are similarly reduced.

Manufacture of vat dyes [of the naphthoylenebenziminazole series]. I. G. FARBENIND. A.-G. (B.P. 447,286, 19.11.34. Addn. to B.P. 237,294; B., 1925, 840).—Naphthalene-1: 4:5:8-tetracarboxylic acid or anhydride is condensed with an o-aminoazo compound, and the product is reduced, e.g., with Zn dust and AcOH. Suitable aminoazo compounds are :  $p-C_6H_4Me\cdotNH_2$  or  $-C_6H_4Cl\cdotNH_2 \rightarrow 2$ -methoxy-p-toluidine (product before reduction melts above 300°; final product, reddish-blue vat dye);  $p-C_6H_4Me\cdotNH_2 \rightarrow o-4$ -xylidine (red-brown). C. H.

Dye-producing composition [mixtures of diazoimino-compounds and coupling components]. E. A. MARKUSH, ASST. to PHARMA CHEM. CORP. (U.S.P. 1,986,276, 1.11.35. Appl., 25.11.32).—Diazotised arylamines are coupled with partly or completely hydrogenated heterocyclic imines carrying  $CO_2H$  or  $SO_3H$ . The colour is developed on the fibre by an acid treatment. Examples are : 5-chloro-o-anisidine  $\rightarrow$  proline, with bisacetoacetylbenzidine (yellow); o-aminodiphenyl ether  $\rightarrow$  piperidine-mono- or -di-carboxylic acid, with 2 : 3hydroxynaphthoic 4-chloro-o-anisidide (scarlet).

C. H. Manufacture of a yellow substantive dyestuff for cotton. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 448,909, 12.3.35).—A yellow dye for cotton is obtained by oxidising 6-carboxy-2-*p*-aminophenylbenzthiazolesulphonic acid by means of alkaline oxidising agents, *e.g.*, NaOCl. N. H. H.

[Manufacture of orange] sulphur colours. J. T. LINSTER, H. B. PRAY, and H. A. LUBS, ASSTS. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,981,055, 20.11.34. Appl., 7.6.33).—A mixture of *m*-tolylenediamine, a diamine of the Ph<sub>2</sub> series (benzidine), and a dibasic acid (phthalic, oxalic, succinic, hexahydrophthalic) is thionated. C. H.

Fluorescent liquid hydrocarbons.—See II. Dyeing leather. Azo dyes. Dyeing or printing.—See VI. Lakes.—See XIII.

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

Oriented adsorption and capillary-active surfaces. Their rôle in the textile industry. J. VALLÉE (Rev. Gén. Mat. Col., 1936, 40, 313-322).—A lecture. E. S. H.

Developments in real silk processing. ANON. (Silk & Rayon, 1936, 10, 618, 620).—The correlation of differences in cross-section axis-ratios of the fibres with two-tone faults in silk hose dyeing, the val. of  $\eta$  measurements of cuprammonium solutions of silk for detection of silk degradation, and the relative efficiencies of various degumming agents are discussed. A. J. H.

Lanital [casein] wool. ANON. (Text. Merc. & Argus, 1936, 95, 143).—The physical and chemical properties of Italian artificial wool are described. Lanital (I) consists of cut-up fibres spun from casein solution; it is warm, soft, and burns similarly to wool. (I) loses < 1% in wt. when boiled in aq. soap solution for 3 hr., and has a greater resistance than wool to aq. NaOH. Fibres of (I) have a smooth surface and are cylindrical; they do not felt, but a 50–50 wool–Lanital mixture felts satisfactorily. (I) can be Cr-mordanted and dyed with

mordant dyes as in wool dyeing. In producing 1 kg. of (I) 1 kg. of casein (obtained from skimmed cows' milk) is used. A. J. H.

Chlorine content of feathers. H. F. KNIGHT (Analyst, 1936, 61, 473; cf. B., 1928, 443).—Polemical against Dodd (A., 1936, 748). The Cl content of feathers washed with cold H<sub>2</sub>O is 60—120 p.p.m. J. W. S.

[Chlorine content of feathers.] F. R. DODD (Analyst, 1936, 61, 473).—A reply to Knight (cf. preceding abstract). J. W. S.

Hemicellulose and its determination in the alkali-cellulose of rayon manufacture. F. CASTELLANI (Boll. Staz. Sperim. Ind. Carta Fibre Tess., 1936, 31, 284–286).—The alkali-cellulose is washed on a porous porcelain filter (the residual  $\alpha$ -cellulose may be dried and weighed), and the filtrate heated for 1—5 hr. (according to the vol. of the liquid) with K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>-H<sub>2</sub>SO<sub>4</sub>, followed by KI and Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> titration in the usual way.

E. W. W.

Chemical examination of groundwood pulp. T. OKADA (J. Cellulose Inst., Tokyo, 1936, 12, 194—196). —The fraction (44%) of a groundwood pulp of todomatsu (*Abies sachalinensis*) passing through a 120-mesh screen contained more lignin and less cellulose than the bulk, but the ratio  $\alpha$ -cellulose/total cellulose was higher. A. G.

Use of bitumen emulsions in the paper industry. F. HOYER (Papier-Fabr., 1936, 34, 275-279).—The prep. of emulsions and their uses both as impregnants and adhesives are described. D. A. C.

Utilising cellulose materials.—See XVIII. Photographic papers.—See XXI.

#### PATENTS.

Refining cellulose fibre for conversion into cellulose derivatives. BROWN Co. (B.P. 448,582, 3.5.35. U.S., 25.7.34).—After pretreatment with dilalkali of < mercerising concn., cellulose pulp is refined in alkali of mercerising concn. ( $\leq 10\%$ ) at a sufficiently high temp. ( $\leq 50^{\circ}$ ) to avoid mercerisation and is finally washed with hot  $H_2O$ . F. R. E.

Production of cellulose esters [secondary propionates]. E. I. DU PONT DE NEMOURS & Co. (B.P. 449,183, 21.12.34. U.S., 21.12.33).—A low-substituted cellulose derivative (cellulose formate containing 3—13%  $HCO_2H$ ) or cellulose, which may be pretreated with  $\gg$  its wt. of  $EtCO_2H$  and 1—5% of  $H_2SO_4$ , is treated with  $(EtCO)_2O$  and  $H_2SO_4$  as catalyst to form a primary tripropionate sol. in  $COMe_2$  and  $C_6H_6$ , which is then hydrolysed with aq.  $EtCO_2H$  and  $H_2SO_4$  until a product sol. in  $COMe_2$  but insol. in  $C_6H_6$  and possibly insol. in BuOAc is obtained. F. R. E.

Cellulose ester compositions and articles produced therefrom. H. P. STAUDINGER (B.P. 449,489, 7.2.35. Addn. to B.P. 431,565; B., 1935, 799).—An aliphatic or aromatic alcohol, e.g., BuOH (> 30 wt.-% of the total solution), is incorporated with the solution of cellulose and citric esters before forming into the required shape. F. R. E.

Preparation of cellulose fibres. J. P. FOSTER (U.S.P. 2,013,042, 3.9.35. Appl., 5.2.34).—Bagasse is

shredded and screened, and digested with 0.5% aq. NaOH at 71° for 1—3 hr. The NaOH is drained and the bagasse washed and then steeped for 2—4 hr. at 71° in 2% HNO<sub>3</sub> solution. After draining and washing, the bagasse is steeped at 49° in 5% aq. NaOH or Na<sub>2</sub>CO<sub>3</sub>, washed, and finally bleached at room temp. in 1—3% CaOCl<sub>2</sub> solution. The treatment takes place in a conical vessel containing an inner concentric perforated cone (containing the pulp) which is fitted at its apex with a propeller-agitator effecting a mild disintegrating action. Steam-coils are placed in the space between the cones. D. A. C.

Production of artificial filaments and like materials. BRIT. CELANESE, LTD. (B.P. 448,570, 15.2.35. U.S., 20.2.34).—A solution containing an org. derivative of cellulose is spun into an evaporative atm., and a current of moist steam is directed obliquely on to the filaments immediately after extrusion. Suitable apparatus is claimed. F. R. E.

Manufacture of artificial textile materials. BRIT. CELANESE, LTD. (B.P. 449,443, 27.11.35. U.S., 4.12.34). —Warps containing org. esters of cellulose are hydrolysed in the required patterns, woven into fabric, and treated with dyes having a different affinity for the hydrolysed and unhydrolysed portions. F. R. E.

Manufacture of artificial filaments, threads, and the like [of varying thickness]. COURTAULDS, LTD., and F. J. COLLINGS (B.P. 449,181, 21.12.34).—During spinning, the nozzle is moved backwards and forwards in the direction of the thread motion to vary its distance from the drawing-off means (winding or guiding device). F. R. E.

Fibre liberation. L. D. SMILEY, ASST. to C. D. ALTICK and E. T. TURNER (U.S.P. 2,011,595, 20.8.35. Appl., 7.12.33).—The material being digested (e.g., by the sulphite process) is kept, throughout the cook, under a const. hydrostatic head by filling in the chips to the topmost level of the digester (D) cone. The liquor circulates from the top of D to a recovery compartment where the liberated gases and vapours are cooled, and collected to be reintroduced together with the liquor into the bottom of D. D. A. C.

Refining of [cellulose] pulp. C. B. THORNE (U.S.P. 2,013,115, 3.9.35. Appl., 21.9.31) .- Pulp is shredded and introduced into a vertical cylindrical tower, in which it is passed by means of a vertical screw conveyor (A) on to rotating combing arms (B) which in turn discharge it into a gas-filled chamber. Thence it is conveyed by a vertical screw (C) into a larger vertical reaction tower which encloses the lower portion of the top tower. The gas (or liquid bleach) is injected into the pulp through perforated rotating arms which, together with A, B, and C, are fixed to a common shaft. Excess gas is withdrawn from the lower tower by a fan, and recovered by absorption (e.g., in H<sub>2</sub>O). The gas supply is automatically stopped if the pulp flow into A D. A. C. ceases.

Protein mixtures.—See III. Coloured cellulose derivatives.—See IV. Cellulose acetate composition.—See VI. Pickling metals.—See X.

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

Absorption of dyes by cellulose. S. M. NEALE (J. Soc. Dyers & Col., 1936, 52, 252-260).—Chiefly an account of work on the absorption of highly purified dyes already published (cf. B., 1935, 59, 302). A table is given showing the absorption and diffusion coeff. of 47 representative dyestuffs on cotton and viscose and the significance of these data, particularly as regards level dyeing, is discussed. S. C.

Dyeing of spun viscose fabrics containing acetate rayon. ANON. (Silk & Rayon, 1936, 10, 606). —The difficulty of dyeing the acetate rayon (I) without staining the cellulose rayon (II) is discussed. Staining is attributed to mechanical adherence or occlusion of the (I) dye, and this increases in the order viscose rayon, mercerised cotton, and cotton. Yarn consisting of 80% of (II) and 20% of non-mercerised cotton stained less than 100% (II) yarn with 5 (I) dyes; this result is attributed to the dominating influence of occlusion as favoured by the highly sponge-like character of the spun rayon yarn. A. J. H.

Moisture in textile processing [dyeing and finishing]. A. J. HALL (Text. Merc. & Argus, 1936, 95, 145).—The influence of  $H_2O$  content of cotton in calendering, "Simili"-mercerisation, and anti-crease finishing, and of wool in hot-pressing is described. The higher  $H_2O$  content of mercerised cotton is ascribed to the more highly dispersed state of the cellulose, whereby it becomes more susceptible to attack by deteriorating influences. A. J. H.

Colour discharge printing on silk. ANON. (Silk & Rayon, 1936, 10, 608-609).—Prep. of the silk fabric, the selection of thickening agents, composition of the discharge printing paste (coloured discharges are chiefly obtained with basic and Cr-mordant dyes), and steaming methods are described. Gum arabic must not be used as a thickener with Cr-dyes since it makes the silk harsh in handle. "Scum" and "streak" difficulties are reduced by first padding the fabric with a 1% aq. solution of Resist Salt L (I.C.I.). A. J. H.

Finishing silk and rayon mixture hose. Anon. (Hosiery Times, Aug., 1936, 10, 25).—Dyeing faults and dulling methods, using  $BaSO_4$  and  $TiO_2$ , are described from a practical viewpoint. A. J. H.

Absorption of tannin by acetate silk. F. JEANMOT (Rev. univ. Soie, 1935, 10, 163—165; Chem. Zentr., 1935, ii, 925—926).—The absorption is not chemical in character; tannin absorption (promoted by soaking the silk and by low temp.) is favourable for even dyeing with basic dyes. H. N. R.

Anti-gas clothing. A. P. J. HOOGEVEEN (Chem. Weekblad, 1936, 33, 474—475).—The following means may be employed for producing clothing for protection against mustard gas (I): (1) impregnation of cloth with substances evolving HOCh, such as bleaching powder or, preferably, chloramine-T, dichloramine-T, or Halzone (p-CO<sub>2</sub>H·C<sub>6</sub>H<sub>4</sub>·SO<sub>2</sub>·NCl<sub>2</sub>); (2) the use of thin metal foil; (3) soaking ordinary clothing in glycerol, in which (I) is insol. : the effect is only temporary owing to the deliquescence of glycerol, even when used in conjunction with starch, gelatin, pipeclay, etc.; (4)

materials which absorb (I), such as rubber, synthetic resins, and prepared leathers: these are only temporarily protective and are difficult to clean after exposure. The presence of (I) in clothing may be detected by leaving it over a vessel containing conductivity-H<sub>2</sub>O, the conductivity of which is raised owing to the reaction  $S(C_2H_4Cl)_2 + 2H_2O = S(C_2H_4\cdotOH)_2$ + 2HCl. D. R. D.

Determining  $CS_2$ .—See III. Silk processing.— See V.

PATENTS.

Manufacture of dyed [rayon] filaments and films. I. G. FARBENIND. A.-G. (B.P. 448,447, 6.12.34. Ger., 6.12.33).—Unreduced vat dyes are incorporated in viscose or cuprammonium solutions and the rayon products spun from these in the usual manner are treated with reducing agents, *e.g.*, Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>, to develop the colour. A. J. H.

Dyeing of leather. J. R. GEIGY A.-G. (B.P. 443,859, 7.12.34. Ger., 7.12.33) .- The use of N-hydroxyalkylated aminoazobenzenesulphonates is claimed to give thorough penetration and through-dyeing of leather. Examples are the dyes:  $m-NH_2 \cdot C_6H_4 \cdot SO_3H \rightarrow NPh([CH_2]_2 \cdot OH)_2$ and  $\rightarrow$  bis- $\beta\gamma$ -dihydroxypropylaniline (both yellow). Other examples are derived from o-C6H4Cl·NH2, 1:2:5and  $1:3:4-\mathrm{NH}_2\cdot\mathrm{C}_6\mathrm{H}_3\mathrm{Cl}_2$ , o-, m-, and  $p-\mathrm{NH}_2\cdot\mathrm{C}_6\mathrm{H}_4\cdot\mathrm{NO}_2$ ,  $\begin{array}{l} 1:4:2\text{-}\mathrm{NH}_2\cdot\mathrm{C}_6\mathrm{H}_3(\mathrm{Cl}\cdot\mathrm{NO}_2,\ 1:2:6:4\text{-}\mathrm{NH}_2\cdot\mathrm{C}_6^{-}\mathrm{H}_2^{-}\mathrm{Cl}_2\cdot\mathrm{NO}_2,\\ 1:2:4\text{-}\mathrm{NH}_2\cdot\mathrm{C}_6\mathrm{H}_3(\mathrm{NO}_2)_2, \qquad 4:1:3\text{-}\mathrm{NH}_2\cdot\mathrm{C}_6\mathrm{H}_3\mathrm{Me}\cdot\mathrm{NO}_2,\\ 2:1:4\text{-}\mathrm{NH}_2\cdot\mathrm{C}_6\mathrm{H}_3(\mathrm{OMe})\cdot\mathrm{NO}_2, \quad 4:1:3\text{-}\mathrm{NH}_2\cdot\mathrm{C}_6\mathrm{H}_3(\mathrm{OE})\cdot\mathrm{NO}_2,\\ \end{array}$  $\begin{array}{l} 3:1:4:6^{-}\mathrm{N}\overset{\circ}{\mathrm{H}}_{2}\overset{\circ}{\mathrm{C}}_{6}\mathrm{H}_{2}\overset{\circ}{\mathrm{M}}_{6}(\mathrm{O}\overset{\circ}{\mathrm{M}}_{6})^{\cdot}\mathrm{NO}_{2}, \quad p^{-}\mathrm{N}\overset{\circ}{\mathrm{H}}_{2}\overset{\circ}{\mathrm{C}}_{6}\mathrm{H}_{4}\overset{\circ}{\mathrm{SO}}_{3}\overset{\circ}{\mathrm{H}}, \\ 1:5:2^{-}\mathrm{and}\ 1:4:3^{-}\mathrm{N}\mathrm{H}_{2}\overset{\circ}{\mathrm{C}}_{6}^{-}\mathrm{H}_{3}^{-}\mathrm{Cl}\overset{\circ}{\mathrm{SO}}_{3}\mathrm{H},\ 1:2:5\overset{\circ}{:}4^{-}\mathrm{and} \end{array}$  $1:2:5:3-NH_2 \cdot C_6H_2Cl_2 \cdot SO_3H$ , 1:2:4-, 1:5:2-, and 1:4:2-NH2 C6H3(NO2) SO3H, and 4-nitro-2-aminophenyl B-hydroxyethyl ether as first components, and β-hydroxyethyl derivatives of NH2Ph (I), o- and  $m\text{-}\mathrm{NH}_2\text{-}\mathrm{C}_6\mathrm{H}_4\text{-}\mathrm{SO}_3\mathrm{H}, m\text{-}\mathrm{NHEt}\text{-}\mathrm{C}_6\mathrm{H}_4\text{-}\mathrm{SO}_3\mathrm{H}, m\text{-}\mathrm{C}_6\mathrm{H}_4\mathrm{Me}\text{-}\mathrm{NH}_2$ (II),  $\tilde{2}: \tilde{4}: 1-\tilde{\mathrm{NH}}_2 \cdot C_6 H_3 \mathrm{Cl} \cdot \mathrm{OMe}$ ,  $m-\tilde{\mathrm{C}}_6 H_4 \mathrm{Cl} \cdot \tilde{\mathrm{NH}}_2$  (III), 3:1:4-NH2.C6H3Me.OMe (IV), and 1:4:2-C6H3Me2.NH2, and  $\beta\gamma$ -dihydroxypropyl derivatives of (I)-(IV) as coupling components, the colours obtained ranging from yellow to red, violet, brown, and grey. H. A. P.

Production on the fibre of insoluble azo dyes. E. I. DU PONT DE NEMOURS & Co., M. A. DAHLEN, and E. ETZELMILLER (B.P. 447,985, 28.11.34).—Blue and violet shades are produced by coupling a diazotised 4-hexahydrobenzamido-2:5-dialkoxyaniline with an arylamide of 2:3-hydroxynaphthoic acid free from H<sub>2</sub>Osolubilising SO<sub>3</sub>H or CO<sub>2</sub>H (e.g., the o-toluidide, 5-chloro-2-toluidide,  $\beta$ -naphthylamide, 4-bromo-2-anisidide, and 5-methoxy-2-toluidide) padded on the fibre.

A. J. H.

[Cellulose acetate] composition of enhanced affinity for dyes. C. J. MALM and C. R. FORDYCE, Assrs. to EASTMAN KODAK CO. (U.S.P. 2,011,345, 13.8.35. Appl., 17.12.32).—Cellulose acetate containing 10—20% of cellulose acetate-phthalate or -succinate (etc.) is claimed as having enhanced affinity for basic dyes as well as the usual dispersed dyestuffs used for acetate silk. S. C.

Dyeing or printing of textile materials. HALL LABS., INC., Assees. of E. B. BELL (B.P. 445,466, 10.7.34. U.S., 12.7.33).—Improved penetration and fastness are obtained by dyeing in presence of  $NH_4$  or alkali hexametaphosphates. A. W. B.

Dyeing or printing of fibrous material [with azoic dyes]. Soc. CHEM. IND. IN BASLE (B.P. 447,349 and 447,351, [A] 15.1.35, [B] 28.1.35. Switz., [A] 23.1.34, [B] 30.1.34).-(A) Bluish-red to violet dyeings fast to light and kier-boiling are obtained by coupling on the fibre an arylide of 2:3-hydroxynaphthoic acid derived from alkyl- or alkoxy-anilines (cf. B.P. 383,064; B., 1933, 144) with a base of formula 1:3:4:6-NH2·C6H2Hal(NHAcyl)·OAryl obtained by reducing and subsequently acylating the condensation products of 2:5-dihalogeno-4-nitroanilines with a phenol. The examples include 4-chloro-2-amino-5-acetamidodiphenyl ether (I)  $\rightarrow$  2 : 3-hydroxynaphthoic 5'-chloro-2'-methoxy-, 4'-ethoxy-, 2'-methoxy-5-methyl-(bordeaux), 5'-chloro-2'-methyl- (garnet) -anilides ; 4'-Me derivative of (I)  $\rightarrow$  2'-methoxy-, 2'-methyl-, 4-methoxy-2'-methyl-, 2': 5'-dimethoxy- (bordeaux), 5-chloro-2methyl- (garnet) -anilides; 4'-Cl-derivative of  $(I) \rightarrow$ 2': 5'-dimethoxy-, 5'-chloro-2': 4'-dimethoxy-, 4'-chloro-2-methoxy-anilides ; 4'-OMe-derivative of (I)  $\rightarrow$  2- or 4'-methoxy-, 2'- or 4'-methyl-, 4'-ethoxy-, 2': 4'- or 2'-5'-dimethyl-, 2'-methoxy-5-methyl-, 2': 5'dimethoxy-anilides. Violet shades are obtained when the corresponding 5-benzamido-derivatives are used instead of the NHAc-compounds. (B) Orange dyeings fast to kier-boiling and Cl<sub>2</sub> are obtained by coupling a 4-alkyl carboxylate of a 2-aminodiarylsulphone (prepared by condensing a 4-halogeno-3-nitrobenzoic acid with an arylsulphinic acid, esterifying, and reducing) with a 2: 3-hydroxynaphthoic arylamide. The examples include Et 2-amino-1: 1'-diphenylsulphone-4-carboxylate and its 4'-Me and 4'-Cl-derivatives  $\rightarrow 2:3$ -hydroxynaphthoic anilide and various substitution products thereof. S. C.

Printing textile products. Soc. CHEM. IND. IN BASLE (B.P. 446,381, 26.6.35. Switz., 27.6.34).— Prints of vat, indigoid, anthraquinonoid, or S dyes of improved depth and fastness are obtained from printing pastes containing monoalkylaminobenzene-3-sulphonic acids or salts thereof, e.g., 1:3-NHEt·C<sub>6</sub>H<sub>4</sub>·SO<sub>3</sub>Na. A. W. B.

Delustring of lustrous textile materials and articles made therefrom. J. CRAIK, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 445,571, 12.10.34).— Fabrics, e.g., rayon, are treated with dulling agents, e.g., ZnO, TiO<sub>2</sub>, china clay, in a medium of a dil. aq. caustic alkali solution of an incompletely sol. cellulose derivative, *i.e.*, one which can be solubilised by temporary cooling or by colloid milling, e.g., methyl- or glycol-cellulose. Impregnation is followed by treatment with dil. aq. or mild gaseous acids, e.g., HCl, AcOH, CO<sub>2</sub>. A. W. B.

Production of effects on fabrics, foils, films, fibres, and the like containing cellulose esters or ethers. BRIT. CELANESE, LTD. (B.P. 445,706, 16.10.34. U.S., 6.1.34).—Effect materials, e.g., bronze or Al powder, or inorg. pigments, are applied to the fabric incorporated in a cellulose ester or ether thickening agent, e.g., nitrate or acetate, and a plurality of solvents for the cellulose derivative in the fabric, e.g., dioxan, COMe<sub>2</sub>,  $CH_2Ph$ ·OH. Non-flaking prints which are fast to rubbing are obtained. A. W. B.

Treatment of textile fabrics. RADUNER & Co. A.-G. (B.P. 445,774, 12.10.34. Switz., 14.10.33).— Calender finishes which are fast to washing are obtained by impregnating the fabric with solutions of carbamides and aldehydes, *e.g.*, urea and CH<sub>2</sub>O, and calendering, whereupon a resin is formed on the fibres and hardened. A. W. B.

Showerproof fabrics and the like materials. INTERNAT. LATEX PROCESSES, LTD., E. A. MURPHY, and E. W. MADGE (B.P. 448,711, 22.12.34).—" Non-rubbery," showerproof, air-permeable materials are produced by impregnating fabric with an aq. dispersion containing  $\geq 10\%$  of rubber (e.g., latex, reclaimed vulcanised rubber, gutta-percha, balata) and 25% (on the dry rubber) of a solid H<sub>2</sub>O-repellent org. substance (e.g., paraffin wax, fatty acids, chloronaphthalenes, hard waxes), followed by heating or immersing the fabric in a coagulant (e.g., 10% aq. CaCl<sub>2</sub>); vulcanising agents may be added to the impregnating liquor. A, J. H.

Sulphonation products. Wetting etc. agents. Diazoamino-compounds. Sulphonated terpenes. —See III. Artificial textiles.—See V. Washing etc. agents.—See XII.

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

Contact or lead-chamber process? W. Siecke (Angew. Chem., 1936, 49, 475–478).—The histories, present positions, and relative merits for various purposes of the two  $H_2SO_4$  processes are discussed.

J. W. S.

Bismuth in Cottrell dust of sulphuric acid factories. V. A. NAZARENKO and A. M. GRANOVSKAJA (J. Chem. Ind. Russ., 1936, 13, 483–484).—The dust contains 0.004—0.014% of Bi. R. T.

Rare gases in ammonia synthesis residues. N. P. KAPUSTIN (J. Chem. Ind. Russ., 1936, 13, 464–466).—The content of rare gases in the gases taken from various stages of the synthetic  $NH_3$  process is in most cases considerably > in air; the possibilities of their recovery are discussed. R. T.

Manufacturing costs of gases for ammonia synthesis. F. G. LAUPICHLER (Gas World, 1936, 105, 71-75).—The production of  $NH_3$ -synthesis gas from water-gas and producer gas by conversion of the CO by the catalytic water-gas process at atm. pressure, the absorption of the CO<sub>2</sub> so produced by scrubbing with  $H_2O$  at 25 atm., and the removal of the unconverted CO by washing with Cu solutions at 200 atm., is described. The economics of the various stages in the above process are discussed in detail, and it is shown that there is a certain % of CO in the converted gas for which the total costs of the synthesis become a min.

H. C. M. Industrial applications of liquid ammonia. B. B. VASILIEV (Coll, Trans. 1st U.S.S.R. Conf. Non-aq. Solutions, Kiev, 1935, 105—113).—A review. R. T.

Washing of "milk of magnesia" through a permeable membrane. E. Moness, W. A. LOTT, and

W. G. CHRISTIANSEN (J. Amer. Pharm. Assoc., 1936, 25, 524–529).—Experimental and theoretical data are given for the washing of milk of magnesia by  $H_2O$ separated by a permeable membrane (canvas filtercloth). F. O. H.

Potassium content of Chinese common salt. C. N. TSAO (J. Chem. Eng. China, 1936, **3**, 132—140).— The K was determined by the cobaltinitrite method, the  $K_2NaCo(NO_2)_6$  being decomposed with HCl containing urea and the excess acid titrated. The majority of the samples contained 0.05—0.20% K, but one sample contained 7.9% K. C. I.

Purity and composition of common salt consumed in China. CENTRAL FIELD HEALTH STATION, NANKING (J. Chem. Eng. China, 1936, 3, 141—168).— NaCl consumed in China is either derived from sea- or lake-H<sub>2</sub>O or from brine wells, or is prepared by washing surface soil with H<sub>2</sub>O. Analyses of a large no. of samples are reported. Many are coloured and most are alkaline to litmus. They usually contain varying amounts of MgCl<sub>2</sub>, CaCl<sub>2</sub>, CaSO<sub>4</sub>, etc., and in a few cases considerable amounts of nitrates. Extension of refining of NaCl for domestic use is very desirable. C. I.

Fatigue of cyanide solutions. I. N. PAKSIN and N. A. SUVOROVSKAJA (Sovet. Zolotoprom., 1935, No. 3-4, 52-59).—The most active causes of fatigue are Cu and Zn, which, at concess of 0.3 and 0.4%, reduce the extraction ability of the solution by 10 and 15%, respectively. Fe, Sb (in alkaline solution), Hg, and Na salts, up to 1, 0.3, 0.4, and 10%, respectively, are not harmful. The fatigue effect of a mixture of impurities is not additive. Fatigue is most pronounced in the percolation process and least in the process of cyaniding simultaneously with grinding. CH. ABS, (e)

Preparation of water-glass without the use of alkalis. S. K. TSCHIRKOV (J. Chem. Ind. Russ., 1936, 13, 469-474).—SiO<sub>2</sub> and 9:1 NaCl-KCl are heated at 800-900° in a stream of air containing 0.2 g. of H<sub>2</sub>O per litre; HCl and a mixture of Na and K silicates are formed. The fugacity of NaCl-KCl mixtures remains approx. const. over the range 25-75% KCl at 940-980°, the amount of salts volatilising being 2-4 times as great as that reacting with SiO<sub>2</sub>; the fugacity rises rapidly with increasing KCl content above 80% KCl. Max. fugacity shifts with rising temp. from 800° to 1000° from the region of mixtures of low to that of high KCl content. R. T.

Preparation of Ammophos from phosphoric acid from Viatka flotational phosphorite. N. I. KRIUTSCHKOV (J. Chem. Ind. Russ., 1936, 13, 466– 469).—NH<sub>3</sub> is passed into the phosphorite extract, containing 37% of  $P_2O_5$ , until the ratio  $NH_3: P_2O_5 =$ 0.35; the solution is conc. to yield a product containing total  $P_2O_5$  49.5, assimilable  $P_2O_5$  46.6,  $H_2O$ -sol.  $P_2O_5$ 40.9, total  $NH_3$  15.6,  $H_2O$ -sol.  $NH_3$  13.9, SO<sub>3</sub> 4.7,  $Fe_2O_3 + Al_2O_3$  6.8, and  $H_2O$  2.4%. R. T.

\_ Relation between specific gravity and lead content of basic lead acetate solution. I. NÉMEDY (Magyar gyóg. Társ. Ért., 1935, 11, 351-361; Chem. Zentr., 1935, ii, 883).—The variation of sp. gr. with Pb content is tabulated. H. N. R.

Po-Shan bauxite as a possible raw material for production of aluminium in China. S. T. Leo and W. C. WEI (J. Chem. Eng. China, 1936, 3, 113—123).— This bauxite (I) contains  $Al_2O_3$  50—54,  $Fe_2O_3$  13—14, and  $SiO_2$  15—21%.  $Al_2O_3$  prepared from (I) was electrolysed in the laboratory for production of Al and compared with Al produced from Merck-quality  $Al_2O_3$ . Allowing for the fact that Si is deposited at the commencement of electrolysis and for the various divergences between laboratory- and large-scale practice, it is deduced that it should be possible to produce Al of 99% purity on a commercial scale from (I). C. I.

Causes of decomposition of hydrogen peroxide, and the stabilisation of perhydrol. S. N. LURE and T. Z. KOPELEVITSCH (J. Chem. Ind. Russ., 1936, 13, 475-482).—The decomp. of  $H_2O_2$  is promoted by  $H_2SO_4$ ,  $K_2SO_4$ , FeSO<sub>4</sub>, and HNO<sub>3</sub>; (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, H<sub>3</sub>BO<sub>3</sub>, and urea have a slight, and  $H_3PO_4$ ,  $H_4P_2O_7$ , NHPhAc, and salicylic acid a powerful, stabilising action. In absence of stabilisers the rate of decomp. in glass containers is > in paraffined ones, and is lower for purified than for technical paraffin. Stabilisation is best effected by removing  $H_2SO_4$  by means of Ba(OH)<sub>2</sub>, and adding NHPhAc. R. T.

Determination of sulphide-sulphur in pyrites. G. ORTNER (Z. anal. Chem., 1936, 106, 28—30).—About 0.2 g. of pyrites is introduced into a stoppered flask with capillary side-tube dipping into a mixture of Br and HNO<sub>3</sub>. By heating and then cooling the flask this solution is drawn over and reacts with the pyrites without danger of loss of S. The excess of Br is removed by heating, SiO<sub>2</sub> is filtered off, and the Fe pptd. with NH<sub>3</sub> before acidifying and pptg. with BaCl<sub>2</sub>. A double pptn. of the Fe eliminates the possibility of SO<sub>4</sub>" occlusion. J. W. S.

Selenium and tellurium content of pyrites and waste products of U.S.S.R. and Caucasian sulphuric acid factories. R. M. BAUMAN (J. Chem. Ind. Russ., 1936, 13, 482–483).—U.S.S.R. carboniferous pyrites contains 0-0.016% of Se + Te. Cottrell dust contains 0.03% of Se and 0.1-0.25% of Te, Glover tower sludge contains up to 2.23% of Se, and chamber sludge 0.003-0.47% of Se. R. T.

Acid handling. Solubility of  $Na_2SO_4$ .—See I. H<sub>2</sub> from coal gas.—See II. Potash for ceramic industry.—See VIII. NaCl in road stabilisation.— See IX. CN' in Cd-plating solutions.—See X. Hypophosphites.—See XX.

#### PATENTS.

Manufacture of sodium carbonate monohydrate. R. B. MacMULLIN, Assr. to MATHIESON ALKALI WORKS, INC. (U.S.P. 2,005,868, 25.6.35. Appl., 25.3.32).—Aq. NaHCO<sub>3</sub> is decomposed by steam, and Na<sub>2</sub>CO<sub>3</sub>, H<sub>2</sub>O is pptd. from the liquor by adding NH<sub>3</sub>; NaCl is added to the mother-liquor, which is then carbonated, the NaHCO<sub>3</sub> pptd. is returned to the commencement of the cycle, and NH<sub>3</sub> and CO<sub>2</sub> are recovered. L. C. M.

Purification of natural heavy spar. J. and M. MÜLLER, ASSTS. to SACHTLEBEN A.-G. F. BERGBAU U. CHEM. IND. (U.S.P. 2,005,296, 18.6.35. Appl., 17.2.34. Ger., 6.3.33).—A white product is obtained by calcining at 1300—1350° under reducing conditions and quenching in dil. mineral acid. L. C. M.

Blanc fixe. H. S. McQUAID, Assr. to GRASSELLI CHEM. Co. (U.S.P. 2,006,219, 25.6.35. Appl., 17.2.34).— Equimol. proportions of  $(NH_4)_2SO_4$  and BaS are caused to react in aq. solution at 50—84°; the BaSO<sub>4</sub> is washed, and dried at 48° to remove occluded  $(NH_4)_2S$ .

L. C. M.

Recovery of salts and other compounds [from pickling solutions]. J. C. WHETZEL and R. E. ZIMMER-MAN, Assrs. to AMER. SHEET & TIN PLATE CO. (U.S.P. 2,005,120, 18.6.35. Appl., 3.2.32).—FeSO<sub>4</sub>,H<sub>2</sub>O is recovered from pickling liquor containing approx. 0.5%of free H<sub>2</sub>SO<sub>4</sub> by autoclaving at 150—210°. L. C. M.

Stabilised phosphorus pentoxide. B. MALISHEV, Assr. to SHELL DEVELOPMENT Co. (U.S.P. 2,005,944, 25.6.35. Appl., 14.3.32).—Claim is made for a drying agent containing  $P_2O_5$  (2 pts.) with heavy fuel oil, pitch, asphalt, bitumen, or other inert binder (1 pt.).

L. C. M.

Graphite.-See II. Fertilisers.-See XVI.

## VIII.—GLASS; CERAMICS.

Thermal endurance of different types of flat glass in relation to thickness. R. W. WAMPLER and G. B. WATKINS (Bull. Amer. Ceram. Soc., 1936, 15, 246— 247).—Test-pieces 3 in. square were quenched in  $H_2O$ at 0°. A smooth hyperbolic type of curve was obtained by plotting the coeff. of thermal expansion against the product of the "temp. diff. required to produce fracture" and the thickness. The curves for sheet, plate, heatabsorbing, and structural glass were coincident.

J. A. S.

Strength of flat glass under uniform Ioad. G. B. WATKINS and R. W. WAMPLER (Bull. Amer. Ceram. Soc., 1936, 15, 243–245).—Breaking tests were made with H<sub>2</sub>O pressure on square and rectangular panels  $(\frac{1}{10} - \frac{1}{4}$ in. thick) rigidly clamped between rubber gaskets. Structural design data are recorded. J. A. S.

Determination of magnesium in glass by means of 8-hydroxyquinoline. M. LOBUNETZ (Bull. Sci. Univ. Kiev, 1935, 1, 167—174).—1 g. of glass is fused with Na<sub>2</sub>CO<sub>3</sub>, the melt is extracted with 50% HCl, the extract is filtered, and Ca is pptd. as CaC<sub>2</sub>O<sub>4</sub>. The filtrate is made alkaline with NaOH, Mg is pptd. with 5 c.c. of 4% 8-hydroxyquinoline in EtOH, the ppt. is dissolved in 2N-HCl, KBr and KBrO<sub>3</sub> are added, and the solution is titrated with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. The results, for glass containing 0·19% of Mg, are about 10% high. R. T.

Cleansing of bottles. G. KRAUSS (Woch. Brau., 1936, 53, 153-158, 161-166, 169-174, 177-182, 186-190, 193-197).—The requirements of bottle-washing plant and processes are critically discussed, and the literature is reviewed, with illustrative experiments.

I. A. P. **Opacifiers in wet and dry enamels.** L. STUCKERT (Internat. Tin Res. Dev. Counc., Tech. Publ. A42, 31 pp.).—The effect of various opacifying agents (pure  $SnO_2$  and mixtures,  $Sb_2O_3$  and compounds,  $CeO_2$ , gas opacifiers) on the coating power, bending strength, heatresistance, solubility in acids, and lustre of wet and dry

enamels was determined. The opacifier causes fundamental changes in the properties of an enamel which cannot be explained by the simple enveloping of the opacifying particles. The phenomenon of increased opacity with repeated firing, due to the slow attainment of equilibrium, is discussed. The coating power of a slip is increased by addition of  $SnO_2$ , but decreased by  $CeO_2$ ,  $Sb_2O_3$ , etc. The bending strength of an enamel is increased by  $SnO_2$  and reduced by all the other solid opacifiers and by gas opacifiers if overfired. The thermal resistance is increased by addition of an optimum of 6% of  $SnO_2$  or  $Sb_2O_3$  and decreased by all other additions.  $CeO_2$  gives a greater opacity than  $SnO_2$ , but the latter is the more stable to over-firing and reduction.  $SnO_2$  is the most satisfactory of all opacifiers.

#### J. A. S.

Applications of potash in the ceramic industry. N. W. TAYLOR (Amer. Inst. Min. Met. Eng., 1936, Contr. 101, 6 pp.).-Present and possible future uses for K compounds in ceramic products, glass, enamels, and glazes are discussed. With glass the use of K rather than of Na compounds secures greater brilliance, "ring" when struck, and chemical durability. Na2O-CaO-SiO2 and Na<sub>2</sub>O-SiO<sub>2</sub> ultra-violet-transmitting glasses are unstable photochemically, whereas K2O-CaO-SiO2 glass is not appreciably affected by ultra-violet radiation. In certain glasses K<sub>0</sub>O appears to show an insulating advantage over Na2O, and the efflorescence of glass used for the "double-glazing" method of insulation (two glass sheets spaced parallel) is eliminated by using a K<sub>2</sub>Obearing (e.g., 6.5-10 wt.-% of K<sub>2</sub>O) glass. K<sub>2</sub>O may find application as a non-poisonous substitute for PbO in brilliant glazes. A. L. R.

Constitution of ceramic products. B. TAVASCI (Chim. e l'Ind., 1936, 18, 338-348).—The results of a chemical and microscopical examination of various clays and ceramics made from them are reported. An examination of the system  $Al_2O_8$ -SiO<sub>2</sub> indicates the existence of  $3Al_2O_3$ , 2SiO<sub>2</sub> (mullite) and the non-existence of  $Al_2O_3$ , SiO<sub>2</sub> (artificial sillimanite). D. R. D.

Pottery plaster. A. HEATH (Trans. Ceram. Soc., 1936, 35, 286–299).—An account of the prep. and properties of plaster of Paris with special reference to its use for pottery moulds. W. O. K.

Semi-dry pressing of chamotte stones. R. KLESPER (Tonind.-Ztg., 1935, 59, 265-267, 381-383; Chem. Zentr., 1935, ii, 903). J. S. A.

Ferriferous quartzite, a mineraliser for silica bricks. P. P. BUDNIKOV and W. M. PARIS (Tonind.-Ztg., 1935, 59, 165—166, 191; Chem. Zentr., 1935, ii, 903).— Up to 5% of ferriferous quartzite (equiv. to 2.75% of Fe<sub>3</sub>O<sub>3</sub> in all) may be incorporated. J. S. A.

Slag-resistance of steelworks refractories. J. H. CHESTERS and L. LEE (Trans. Ceram. Soc., 1936, 35, 271— 283).—A review of methods of determining the probable slag-resistance of various refractories and discussion of the factors involved. W. O. K.

Testing slag attack [of refractories]. S. SACHS (Tonind.-Ztg., 1934, 58, 1195—1197; Chem. Zentr., 1935, ii, 903).—Procedure for testing the resistance to molten slag is described. J. S. A.

#### PATENTS.

Manufacture of laminated [glass] articles. E. I. DU PONT DE NEMOURS & Co. (B.P. 448,740, 22.7.35. U.S., 21.7.34).—The interlayer consists of an org. ester of cellulose, e.g., the acetate (I) (100 pts.), plasticised with an ester (80—100 pts.) of a polyhydric ether alcohol and a saturated fatty acid ( $C_{3-5}$ ), e.g., diethylene glycol dipropionate. A second plasticiser which has greater solvent action on (I), e.g., o-C<sub>6</sub>H<sub>4</sub>(CO<sub>2</sub>Me)<sub>2</sub>, may also be incorporated. S. M.

Protein mixtures.-See III.

#### IX.—BUILDING MATERIALS.

Problem of clinker burning without after-oxidation. R. SELZER (Tonind.-Ztg., 1935, 59, 92-93; Chem. Zentr., 1935, ii, 903).—The suppression of oxidation of tiles burned in a reducing atm. is discussed.

J. S. A.

Determining the resistance of Portland cement to sulphate waters. Accelerated test. R. W. STENZEL (Ind. Eng. Chem. [Anal.], 1936, 8, 263—266).—A slab of neat cement is cast. After setting, the top surface is scraped, and the sides and bottom are painted with a waterproof composition. The slab is then immersed in 10% aq. Na<sub>2</sub>SO<sub>4</sub>. Attack at only one face causes the slab to warp, the extent of which is determined by a spherometer. E. S. H.

Puzzuolanas. I. Zeolitic nature of puzzuolanas. II. Flocculation. III. Behaviour of cement-puzzuolana mixtures in excess of water. Q. SESTINI and L. SANTARELLI (Annali Chim. Appl., 1936, 26, 193-196, 197-202, 202-206).—I. The zeolitic activity of various puzzuolanas (I), examined by the method used by Biehl and Wittekind and by Stoepoe for trasses, is small, and of secondary importance in the absorption of CaO.

II. Flocculation experiments are more a measure of the velocity of action of (I) than of their val. and quality.

III. Methods for determining puzzuolanic action in the presence of excess of  $H_2O$  are shown to be insufficient and a series of tests on the paste has been started, in the cold and in the hot. L. A. O'N.

Puzzuolanic cements. C. VITTORI (Chim. e l'Ind., 1936, 18, 349–353).—Polemical against Zamboni (B., 1936, 545). D. R. D.

Constitution of aluminous cements. A. TRAVERS (Ciment, 40, 11–13; Chem. Zentr., 1935, ii, 573; cf. B., 1936, 193).—A review and discussion of previous work. Aluminous cements are resistant to MgSO<sub>4</sub> and  $(NH_4)_2SO_4$ , but not to conc. alkali salt solutions. H. J. E.

Quality, design, and economy of concrete. I. Lyse (J. Franklin Inst., 1936, 221, 653-672, 745-768; 222, 83-98; cf. B., 1936, 499).—Various desirable qualities in concrete are discussed. The factor influencing compressive strength is the cement concn. in the  $H_2O$  in the concrete at the time of hardening. High modulus of elasticity, low plastic flow, low leakage, high durability, and high fire-resistance are associated with high strength. Vol. changes increase with increase in cement content. For a given type and gradation of aggregates the net amount of mixing  $H_2O$  in the concrete remains practically const. for a given placeability,

and is independent of the richness of the mix. The economy of plain concrete increases markedly with increase in strength; variables such as aggregate grading and strength quality of cement are less important. The economics of reinforced concrete under various conditions are considered. T. W. P.

Road dust. T. B. LOFTHEIM (Medd. Veidirektøren, Oslo, 1936, 65—68; Road Abs., 1936, **3**, No. 303).—In dust from different roads > 50% of the particles had diam. too small for ultramicroscopical measurement; the remainder had a mean size of  $2 \cdot 3 \mu$  and were composed of pear-shaped or spherical forms of pyroxene, felspar, quartz, and hornblende. The content of org. matter was  $7 \cdot 67$ — $10 \cdot 75\%$ . T. W. P.

Use of sodium chloride in road stabilisation. H. RES (Amer. Inst. Min. Met. Eng., Tech. Publ., 1936, No. 721, 8 pp.).—Tests (e.g., vol. shrinkage,  $H_2O$  content after various drying treatments, rate of  $H_2O$  percolation through clay-sand-NaCl mixtures) were made on a representative series of clays used for stabilising sandgravel road mixtures (I), both alone and in admixture with 1—6% of NaCl. Addition of NaCl assists in compacting the (I) and in retaining the moisture whether it is conc. on the surface or in the underlying soil. About 2 lb. of NaCl per sq. yd. is usually added to a 3-in. course of (I). A. L. R.

Capillary action of tars and bitumens in road surfaces. A. LIEUTE (Gas World, 1936, 105, 52) .--Various types of road tars and bitumens have been examined by the capillary-rise method of measuring surface tension. The vals. of the surface tensions were similar even with materials of widely different viscosities; this is attributed to the structure of the tars, the surface tension measured being that of the oil forming the continuous phase. The sweating of the binder from road surfaces is due partly to capillary rise in the interstices between the aggregates, but is dependent also on the viscosity of the material and its ageing by evaporation. All these characteristics can be assessed by the capillarytube test. To prevent sweating and so maintain the initial roughness of the surface, coal may be incorporated with the tar or bitumen. A. B. M.

Correlation of low-temperature tests with resistance to cracking of sheet asphalt pavements. L. F. RADER (Assoc. Asphalt Paving Tech., Proc. Tech. Sessions, Cleveland, Ohio, Jan. 23, 1936, 29-43; Road Abs., 1936, 3, No. 309).-Results of an investigation on two road surfaces having different performances in practice, due to differences in the bitumens used, are described. It is concluded that the moduli of rupture and elasticity are measures of the resistance to cracking of sand-carpet mixtures and may be used as controls in design. Toughness at low temp. is a measure of the resistance to impact and may be used as a control test of the ability of the carpet to withstand the action of traffic at low temp. Other features for low-temp. conditions are : asphalt surfacing mixtures should not be designed to have great resistance to displacement (as measured by stability tests) without considering resistance to cracking; mixtures containing the highestpenetration bitumen consistent with stability should prove most resistant to cracking. The necessity for proper control of plant and constructional operations is emphasised. T. W. P.

Wood in chemical apparatus construction. I. J. KLINOV and V. P. SCHISCHKOV (Chim. Mash., 1934, No. 3, 32—36).—Wood impregnated with liquid bakelite (Amodification) was resistant to 25—36% aq. NaCl (60°), 50% H<sub>3</sub>PO<sub>4</sub> (60°), 40% H<sub>2</sub>SO<sub>4</sub> (24°), 60% AcOH (60°), 8% H<sub>2</sub>C<sub>2</sub>O<sub>4</sub> (20°), 40% HF (25°), 25% aq. NH<sub>3</sub> (20°), 60% AlCl<sub>3</sub> (60°), molten MgCl<sub>2</sub> (170°), 20% NH<sub>4</sub>Cl or (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> (20°), 10% Na<sub>2</sub>CO<sub>3</sub> (60°), 3·5—4% NO<sub>2</sub> (60°), 7% Cl<sub>2</sub> (70°), and 14·5% HCl (35°). The resistance to 10% aq. NaOH (20°) was poor. CH. ABS. (e)

Susceptibility of asphalts to temp. change.— See II. Wood primers.—See XIII. "Soil acid."— See XVI. Concrete fermenting vessels.—See XVIII.

## PATENT.

**Treatment of marble surfaces.** R. W. H. and C. R. Fox (B.P. 448,568, 12.2.35).—To render the colour and grain prominent the marble is cleaned with, *e.g.*, NaOH and  $CCl_4$ , pickled with bleaching powder, washed, and any hollows are filled with an oil-bound composition of, *e.g.*, china clay and a synthetic resin ; the surface is then ground and coated with a transparent varnish (a glyptal resin in oil) which is flatted down and a second coat applied and polished with a suitable abrasive. S. M.

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

Progress in metallurgical chemistry. Y. Sürü (Tech. Kurir, 1936, 7, No. 7, 1-2; No. 8, 1-6).-A lecture. E. P.

Volatilisation process in metallurgical work. W. LEITGEBEL (Metall u. Erz, 1935, 32, 205-211; Chem. Zentr., 1935, ii, 907-908).—The volatilisation of metals during extraction and working is discussed. J. S. A.

Solubility of high-sulphur iron in cupola slag. B. P. SELIVANOV, A. S. GINZBERG, and M. M. VOROVICH (Repts. Inst. Met. Leningrad, 1933, No. 15, 171—177; cf. B., 1936, 410).—With slag containing SiO<sub>2</sub> 50, Al<sub>2</sub>O<sub>3</sub> 5, Ca 30, and FeO 15%, FeS dissolves up to 5% at 1300°. Desulphurisation of the metal depends both on the ability of the slag to absorb S and on the distribution coeff. of S between the slag and metal. Replacement of CaO by MnO ( $\gg$  10%) increases the S retained by the slag even at lower temp. When FeS is > 5% the addition of MnO has no appreciable effect on desulphurisation.

Сн. Авз. (е)

Use of cast iron in the [American] chemical industry. H. L. MAXWELL (Chem. Eng. Cong. World Power Conf., 1936, A8, 15 pp.).—The service behaviour of plain and alloy cast irons is discussed and attention drawn to the improvements in corrosion-resistance etc. effected by additions of small proportions of Ni, Cr, Mo, and Si to alloy cast Fe for wear-resistance, e.g., Ni-Hard and Diamite, and to austenitic cast Fe. In scaling tests at 750° and 950° for 100 hr. on ordinary cast Fe (A), Ni-Cr cast Fe (B), and Niresist (C) when either protected from the outside air, or subjected to a 2-litre/hr. stream of dry air, air saturated with H<sub>2</sub>O, or illuminating gas, C was less affected than A or B. A thin, dark, adherent scale formed on C at both temp., but the scale was loosened by moist air at 950°. E. H. B.

Modern cast irons in chemical plant equipment. J. G. PEARCE (Chem. Eng. Cong. World Power Conf., 1936, A5, 11 pp.).—Cast Fe is defined and recent developments are outlined. Growth of cast Fe on heating is due to internal oxidation. The austenitic alloys Niresist and Nicrosilal are 100 times more resistant to N-HCl and  $N-H_2SO_4$  than is ordinary grey Fe or hæmatite Fe, but little better in N-HNO<sub>2</sub>. E. H. B.

Behaviour of grey cast iron between  $650^{\circ}$  and  $800^{\circ}$ , and the influence of silicon. E. JIMENO and A. MODOLELL (Anal. Fís. Quím., 1935, 33, 279–296).— The relation between combined C, hardness, and microstructure has been investigated. Fe<sub>3</sub>C is destroyed by prolonged heating and by the presence of Si. Ni and Cr increase the hardness. The change from pearlite to ferrite depends on the amount of Si present. F. R. G.

Corrosion and protection of cast-iron and steel pipe-lines in the soil. C. A. H. VON WOLZOGEN KÜHR and J. P. PFEIFFER (Chem. Eng. Cong. World Power Conf., 1936, A7, 34 pp.).—Anaërobic corrosion of Fe by the formation of FeS is frequently encountered and is regarded as an electro-biochemical process. With alternate anaëroby and aëroby, the FeS is converted into acid Fe<sup>III</sup> sulphate and thus in absence of Ca<sub>2</sub>CO<sub>3</sub> in the soil the corrosion is accentuated. A protective coating must be proof against aggressive soil influences, waterproof, and mechanically strong. Blown asphaltic bitumens are preferred to coal-tar pitches, but fillers are often necessary and must be selected with care.

E. H. B.

Tests on cast iron with a cylindrical test-piece of 5.64 mm. diameter. P. NICOLAU (Rev. Mét., 1936, 33, 393—399).—Details of impact, alternating and repeated bending, magnetic, and thermoelectric testing of cast Fe, using in each case a test-piece of 5.64 mm. diam., are given. The methods are intended to be used for routine testing. W. P. R.

Iodine method of determining sulphur in cast iron and steels by combustion in oxygen. F. WALD (Rev. Mét., 1936, 33, 390–392).—The method is based on the direct titration by I of the SO<sub>2</sub> formed by combustion of steel in a current of very dry O<sub>2</sub> free from H<sub>2</sub>. Details of the apparatus, including a carborundum resistor furnace working at 1350°, are given. W. P. R.

Silicon cast irons. H. THYSSEN (Rev. Mét., 1936, 33, 379-389).—In order to attain high resistance to oxidation at high temp. the C and Si contents of cast Fe must be such as to produce a ferritic hypoeutectic structure which has no transformation point up to the temp. at which it is intended to be used. W. P. R.

Desulphurisation of pig iron by manganese. H. WENTRUP (Arch. Eisenhüttenw., 1935-6, 9, 535-542).—The presence of C, Si, P, and Mn in pig Fe reduces the solubility of FeS and MnS in the metal at 1250-1350° almost to zero; the FeS-MnS separates from the metal simply by a segregation process and dissolves to a limited extent in the slag. The higher is the Mn content of the metal the lower is the solubility of the sulphides. The addition of Na<sub>2</sub>CO<sub>3</sub> or excess of CaO to the slag increases the rate at which the sulphides are absorbed by promoting their oxidation. A. R. P.

Chemistry of the acid open-hearth [steel] process. H. SCHENCK and E. O. BRÜGGEMANN (Arch. Eisenhüttenw., 1935—6, 9, 543—553).—From temp. measurements and analyses of the metal and slag at various stages in the open-hearth process the equilibria of the Si and Mn reactions and the distribution of FeO between the metal and slag have been determined. The results are expressed by equations and illustrated graphically, and the causes of their deviation from the results of earlier workers are discussed. A. R. P.

Direct method of stainless-steel production. R. P. FORSYTH (J. Chem. Met. Soc. S. Africa, 1936, 36, 319– 337).—The chemistry and economics of the production of 12% Cr steel by the reduction of  $Cr_2O_3$  by Fe–Si above a prepared bath of low-C steel are discussed. High-MgO slags are employed. E. H. B.

Behaviour of carbon and molybdenum steels at high temperatures. C. H. M. JENKINS, H. J. TAPSELL, G. A. MELLOR, and A. E. JOHNSON (Chem. Eng. Cong. World Power Conf., 1936, A6, 37 pp.).-13 C steels tested at 450° under a load of 8 tons/sq. in. are found to fall into two divisions, one exhibiting min. creep rates of 0.3-2.1 in./in./hr. and the other 10.8-107 in./in./hr. In the first group increase in C content from 0.15 to 0.40% lowers creep-resistance. Steels of similar C content and containing 0.5 or 1.0% of Mo are markedly affected by heat-treatment. In a 0.011, Mo 0.5% steel (M) the oil-hardened and tempered condition is the most resistant to creep. A coarsened structure is more resistant than that produced by air-cooling from just above the normalising temp., whilst spheroidising (S)greatly reduces the resistance; at 550° and under a load of 9 tons per sq. in. S gives 80 times the creep rate of oil-quenching and tempering in steel M. With a steel containing 0.4% C, however, oil-quenching and tempering reduces the creep-resistance below that of the air-cooled condition. Failure at high temp. after long periods of creep is generally the result of intercryst. cracking. All the steels tested have similar rates of corrosion, both in synthetic flue gas and in superheated steam. E. H. B.

Deep-drawing capacity of the harder carbon steels. F. EISENKOLB (Arch. Eisenhüttenw., 1935-6, 9, 575-578).—Expressions are derived and nomograms constructed for calculating the tensile strength, elongation, and Erichsen val. of hard C steels, one from the other. A. R. P.

Tensile properties of steels at temperatures below and above normal. D. A. R. CLARK (Engineering, 1936, 142, 104—105).—Elastic limit, yield point, and tensile strength of 0.4% C steels are little affected by temp. in the range 200° to  $-40^\circ$ , but below  $-40^\circ$  the vals. rise steeply, to 63, 57, and 31 tons/sq. in., respectively, at  $-180^\circ$ . The elongation is a max. at room temp. and decreases to zero at  $-180^\circ$ . At 120—200° Lüders lines are produced extensively and the fractures of tubes are helical. Theoretical consideration of the results indicates that the best materials for low-temp. service are those in which at. heat does not decrease rapidly at a moderate temp., e.g., Cu, and those with small or negative coeffs. of expansion, e.g., Fe–Ni alloys. E. H. B. Dilatometric and magnetic peculiarities of quenched eutectoid steels, especially if quenched in molten tin at 300–325°. J. SEIGLE (Rev. Mét., 1936, 33, 353–361).—Although entirely different properties can be obtained in a eutectoid steel by quenching in molten Sn at temp.  $> 300^{\circ}$  compared with quenching in H<sub>2</sub>O and tempering at  $> 300^{\circ}$ , it is probable that the dilatometric, micrographic, and magnetic characteristics are caused by the state of dispersion of  $\alpha$ -Fe and Fe<sub>3</sub>C, which varies continuously from a state of solid solution (max. dispersion) to lamellar pearlite. W. P. R.

Small plastic deformations in strip steel. N. P. Goss (Met. & Alloys, 1936, 7, 131–134).—Preferred orientation of grain fragments produced by small plastic deformations in steels can be detected by X-ray diffraction methods. A similar orientation is produced by hot-rolling steel strip. The preferred orientation in hotand cold-drawn wires is developed in different directions. The diffuseness of the Laue spots in diffraction patterns of steels which have been slightly cold-worked is ascribed to grain fragmentation and not to lattice strain, since the lattice parameters are not changed by this treatment. A. R. P.

Wear of metals in sliding friction. B. KEHL and E. SIEBEL (Arch. Eisenhüttenw., 1935—6, 9, 563—570). —The effect of structure on the rate of wear of dry and lubricated sliding surfaces of cast Fe and steel and of cast Fe and steel in bronze and white-metal bearings has been determined. A. R. P.

Hard-facing [of metals] with boron. M. E. SMITH (Amer. Machinist, 1935, 79, 548–550).—" Sweat-on" and "cast-on" B crystals are used to hard-face ferrous metals. The properties of such coatings are described. The crystals of sweat-on are mixed with Na silicate and  $H_2O$  and brushed or spread on the surface. The metal is then brought to a sweating heat. Previous case-hardening is advantageous. CH. ABS. (e)

Case-hardening steels. C. C. HODGSON (Met. Treatment, 1935, 1, 82-87).—A discussion of heat-treatment and failure of case-hardened parts. Suitable alloys for various types of service are described.

#### Сн. Авз. (е)

Alteration of steel during hardening, tempering, and annealing. A. VON VEGESACK (Z. anorg. Chem., 1936, 227, 145–178).—The results of microscopical examination of steels of different composition submitted to hardening, tempering, and annealing under varied conditions, and measurements of hardness, length, and sp. vol., are recorded and discussed. F. L. U.

Local surface-hardening of crankshafts. H. Voss (Z. Ver. deut. Ing., 1935, 79, 743-749); Chem. Zentr., 1935, ii, 909).—Local surface-hardening of steel bars (0.35% C, 1.0% Cr, 0.2% Mo) by treatment with the  $C_2H_2-O_2$  flame is a rapid process which produces a hard skin to a sufficient depth without adversely affecting the body of the material. J. S. A.

Origin and avoidance of hardening and abrasion fissures, and of flaking of the hardened layer in case-hardened materials. R. Koch (Automobiltech. Z., 1935, 38, 276—279; Chem. Zentr., 1935, ii, 909).— A review. J. S. A. Transformation temperatures of martensite and austenite in hardened steels. B. KJERRMAN (Met. & Alloys, 1936, 7, 127—129).—Measurement of the electrical resistance of quenched steel containing C 1, Mn 1, Cr 1, and Si 0.5% during prolonged annealing at  $40-240^{\circ}$  shows that untempered martensite is labile at room temp. and slowly reverts from the tetragonal to the cubic form at as low as  $40^{\circ}$ . Retained austenite does not decompose until the martensite change is completed, and then only at  $240^{\circ}$  A. R. P.

Annealing twins in ferrite. A. L. TARE (Met. & Alloys, 1936, 7, 130).—After annealing Armco Fe in moist  $H_2$  at 1490° for 18 hr., holding it at 890° for 12 hr., and cooling slowly to room temp., large, well-defined ferrite twins are produced on the surface of the sheet. The effect is ascribed to practically complete removal of the C, S, P, O, and N from the surface layers.

A. R. P.

Steels for autoclaves. R. J. SARJANT and T. H. MIDDLEHAM (Chem. Eng. Cong. World Power Conf., 1936, A3, 45 pp.).—The operating conditions of autoclaves are reviewed and it is indicated that the most important properties in the material are resistance to creep and to embrittlement during extended heating. The attack of steels by H<sub>2</sub> is specially considered; this leads to decarburisation and intergranular fissuring, with a consequent impairment of mechanical strength. Mild steel is severely attacked at 325° by H<sub>2</sub> at 250 atm. Addition of Cr or Cr-V lessened the degree of penetration of  $H_2$ , but severe embrittlement occurs at 550° with long exposure. Ni-Cr-Mo steel was superior, but not entirely immune from embrittlement. Cu-Cr-Mo steel was fully resistant to  $H_2$  at 550°/250 atm. More highly alloyed steels showed good resistance to attack, but Cr steels were slightly embrittled. Austenitic Ni-Cr steels were not attacked or embrittled under the conditions of experiment. E. H. B.

Action of molten zinc on iron, steels, and special steels. I. MUSATTI and A. LA FALCE (Rev. Mét., 1936, 33, 362—370).—The corrosive action of molten Zn on Fe and steel increases with the C content and above 0.5% C the attack is very rapid. Si is harmful, Cr has little effect, but Ni appears to be beneficial. S and P in the proportion usually found in steels have no appreciable effect. Armco Fe and rimming steels resist molten Zn because of their low C and Si contents. The presence of a layer of Fe–Zn compound of high m.p. formed on the surface of these two materials accounts for their increased resistance. W. P. R.

Corrosion protection of iron by arsenic. G. TAMMANN and H. WARRENTRUP (Z. anorg. Chem., 1936, 228, 92—96).—Adherent amorphous deposits of As on Fe may be formed by electrolysis from a saturated solution of  $As_2O_3$  in HCl ( $\gg 4$  volts, initial c.d. 0.1-0.2amp./sq. cm.). The deposit becomes cryst. at 300°. It contains very little AsCl<sub>3</sub>, in contradistinction to a similar deposit of Sb, which may contain 20% of SbCl<sub>3</sub>. A similar protective film is formed on adding  $As_2O_3$  to  $H_2SO_4$  or HCl in which Fe is being dissolved. Steel (0.93% C) may be similarly protected, but such coatings develop faults in time. Fe may also be heated in  $AsH_3 + H_2$  at 100—200°, when a film of As is deposited. On heating Fe in As vapour at  $600-700^{\circ}$  the outer film formed consists of FeAs. Below it is a layer of mixed crystals (6% As). This film is also corrosionresistant. Cu and Ni form similar films when heated in As vapour at 600°. H. J. E.

Physico-chemical interpretation of the action of [steel pickling] inhibitors. E. JIMENO and I. GRIFOLL (Anal. Fís. Quím., 1935, 33, 947-951).—The action of the inhibitors in the "pickling" of steel (cf. B., 1934, 581) is regarded as due to preferential adsorption in the cathodic regions producing an increase in c.d. and large H overvoltage. The surface tension of the acid bath is also diminished, loss of H being thus prevented both by electrochemical and mechanical means. F. R. G.

Mechanism of chemical reactions in weld metal. J. H. PATERSON (Welding J., 1935, 32, 134–135).— A standard electrode is proposed consisting of mild steel wire (C 0.11-0.13, Mn 0.35-0.45, S 0.4, P 0.4%, Si trace). The coating consisted of Fe<sub>3</sub>O<sub>4</sub> 70, SiO<sub>2</sub> 30%. The N content of weld metal is discussed. CH. Abs. (e)

Determination of gases in steel and ferro-alloys by vacuum melting. R. CASTRO and A. PORTEVIN (Arch. Eisenhüttenw., 1935-6, 9, 555-562).—A modified vac. furnace and the ancillary gas-analysis apparatus are described and some results obtained on various steels are tabulated and discussed. A. R. P.

Spectral-analytical determination of antimony in copper. B. A. LOMAKIN and A. L. OSTACHEVSKAJA (Z. anorg. Chem., 1936, 228, 44—48).—By using controlled arc conditions, Sb may be determined in Cu by comparing the intensity of the Sb line at 2311.50 A. with Cu lines at 2303.13, 2319.56 A. The method was tested with alloys containing 0.0005—0.1% of Sb. H. J. E.

Magnesium silicide in 85:15 brass. H. S. VAN KLOOSTER and J. E. HARRIS, JUN. (Met. & Alloys, 1936, 7, 135—139).—The hardness of chill-cast 85:15 brass increases linearly with addition of Mg<sub>2</sub>Si and is about doubled with 4.7% Mg<sub>2</sub>Si. The *d* simultaneously decreases from 8.75 to 8.05. No age-hardening occurs at room temp. but the hardness of cold-worked metal increases on annealing to a max. at 450—550°. Addition of 4.7% Mg<sub>2</sub>Si lowers the f.p. from 1020° to 980°. Characteristic microstructures after various heat-treatments are illustrated. A. R. P.

Rapid photometric determination of copper in brass and related alloys. K. DIETRICH and K. SCHMITT (Z. anal. Chem., 1936, 106, 23–27, 80).— 1 g. of the metal is dissolved in 10 c.c. of 33% HNO<sub>3</sub>, evaporated with 2–3 c.c. of H<sub>2</sub>SO<sub>4</sub>, converted into the CuSO<sub>4</sub>-NH<sub>3</sub> complex, and the extinction coeff. of the solution determined in a special form of photometer (described). Alternatively, 1 g. of metal is dissolved in 15 c.c. of 50% HNO<sub>3</sub> and converted into the Cu(NO<sub>3</sub>)<sub>2</sub>-NH<sub>3</sub> complex, the extinction coeff. of which is measured. J. W. S.

Self-hardening bronzes on a copper-nickel-tin basis. IV. Influence of heat-treatment on corrosion-resistance. E. FETZ (Korros. u. Metallschutz, 1935, 11, 100-107; Chem. Zentr., 1935, ii, 910).--The highly disperse phase produced from the supersaturated  $\alpha$ -solid solution by heat-treatment promotes corrosion by HNO<sub>3</sub> by intensified local-element formation. Tempered bronzes form a compact protective coating in HCl. Variations in resistance with heattreatment, ageing, and composition are discussed.

J. S. A.

Lead bronzes. W. CLAUS (Z. Metallk., 1936, 28, 84—91).—Recent literature and patents are reviewed. A. R. P.

K-Monel metal and its technical properties. R. MÜLLER (Z. Metallk., 1936, 28, 97–99).—The alloy contains Ni 63·2, Cu 30·8, Al 3·5, Fe 1·5, Mn 0·5, C, Si, etc. > 0.5% and has lower d and m.p. and a much higher electrical resistance than ordinary monel metal; in addition, the Curie point is at  $-40^{\circ}$  and the impact strength and endurance limit are very high even at high temp. A. R. P.

Production of zinc from blast-furnace dust. O. JOHANNSEN (Angew. Chem., 1936, 49, 478—480).— Blast-furnace dust contains 0.3-0.8% of Zn, mainly as ZnO, the upper dust being rich enough to work direct in a Zn furnace, but not the lower. The Zn is best extracted by ammoniacal aq. (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub>, the extract on heating depositing the Zn as basic carbonate. The economics of the process are discussed. J. W. S.

Reducibility of zinc ferrites. M. REV and G. BAIWIR (Rev. univ. Mines, 1935, [viii], 11, 226-228; Chem. Zentr., 1935, ii, 909-910).—The reduction of Zn proceeds less readily than from ZnO, but precedes that of Fe (cf. Lindt, B., 1914, 697). J. S. A.

Welding of zinc sheet. H. A. HORN (Autog. Metallbearb., 1935, 28, 169—171; Chem. Zentr., 1935, ii, 914). —The process is described. The use of  $C_2H_2$  with 10% excess of  $O_2$  is satisfactory. J. S. A.

Zinc die-casting alloys containing aluminium. W. CLAUS [with R. WEIDNER] (Metallwirts., 1935, 14, 67—68; Chem. Zentr., 1935, ii, 910).—Mechanical properties of the alloys are improved by the use of highgrade electrolytic Zn. The presence of much Cu is unfavourable. J. S. A.

Melting and casting metals in vacuo. W. J. P. ROHN (Trans. Electrochem. Soc., 1936, 70, Preprint 4, 49-51).—The notched-bar impact val. of an alloy containing Ni 65, Cr 15, Fe 20% is enhanced by vac.melting, in furnaces up to 4 tons capacity, according to the method described. E. H. B.

Non-ferrous casting alloys of high strength. A. J. MURPHY (Trans. Amer. Foundry. Assoc., 1935, Preprint No. 35-14, 32 pp.).—A review. CH. ABS. (e)

Flotation of cassiterite and wolframite. M. WINKLER (Metall u. Erz, 1935, 32, 181–183; Chem. Zentr., 1935, ii, 911).—Twofold flotation followed by table washing is claimed to give a recovery of 90—97% of Sn or WO<sub>3</sub>. J. S. A.

White alloys in printing. A. BARGILLIAT (Chim. et Ind., 1936, 36, 3-15).—A review is given of the properties, uses, and analysis of binary and ternary alloys of Sn, Sb, and Pb. J. L.

Action of sodium bisulphate on arsenical ores. V. TAFEL and G. LAMPE (Metall u. Erz, 1935, 32, 183—187; Chem. Zentr., 1935, ii, 910).—Ore is fused with NaHSO<sub>4</sub> at 200—300°, the mass being finally roasted at 400°. As is oxidised to  $As_2O_3$  and volatilised, and Ni, Co, and Cu are obtained as sulphates.

J. S. A.

Recent developments in gold milling practice. W. B. TIMM (Trans. Canad. Inst. Min. Met., 1935, 330– 340).—A review of Canadian developments.

Сн. Авз. (е)

Measurement of pressure in aluminium dust explosions. W. GLIWITZKY (Z. Ver. deut. Ing., 1936, 80, 687—692).—Bomb experiments showed that the pressures resulting from explosion increased with increasing fineness of the Al powder. Pressures up to 12.6 atm. were recorded. R. B. C.

Recommended practice for [casting] common aluminium-copper alloys. T. D. STAY *et al.* (Trans. Amer. Foundry. Assoc., 1935, Preprint No. 35–18, 26 pp.).—Details of casting are given. CH. Abs. (e).

Light alloys of aluminium, zinc, and cadmium. J. IBARZ (Anal. Fís. Quím., 1935, 33, 140–165).— Photomicrographs have been prepared and the coeff. of expansion, resilience, hardness, and resistance to corrosion by N-NaOH and N-HCl with and without added NaCl have been determined for 99.54% Al (containing a little Fe and Si) and for several alloys of Al containing 5–20% Zn and 0·1–2% Cd. The Cd appears to stabilise Al<sub>2</sub>Zn<sub>3</sub> but its inclusion in the alloys is concluded to have no industrial val. F. R. G.

Forgeability of various types of light and ultralight alloys. A. PORTEVIN and P. BASTIEN (Compt. rend., 1936, 202, 2072–2074).—Mg can be spun when alloyed with  $\gg 15\%$  Cu or 6—9% Al. Alloys with  $\gg 15\%$ Cu can be forged. Al + 12% Cu can be spun or forged. The limit for forging Al-Mg alloys is 5—10% Mg. (Cf. B., 1936, 599.) H. J. E.

Corrosion of ultra-light alloys. P. BASTEIN (Aciers spéc. Mét., Alliages, 1934, 9, 408–414; Chem. Zentr., 1935, ii, 916).—A discussion of corrosion phenomena in Mg alloys, and their prevention. J. S. A.

Corrosion experiments with bolts in light-metal constructional parts. H. BAUERMEISTER and R. KERSTEN (Z. Ver. deut. Ing., 1935, 79, 753-756; Chem. Zentr., 1935, ii, 915-916).—Measures preventing the corrosion of screw threads in light-metal structures exposed to sea-H<sub>2</sub>O and -air are discussed. J. S. A.

Hardening of solid solutions (age-hardening). C. BENEDICKS (Jernkont. Ann., 1935, 118, 241-248). A discussion. CH. ABS. (e)

Submerged corrosion test. Y. YAMAMOTO (Bull. Inst. Phys. Chem. Res., Japan, 1936, 15, 451—580).— The effect of the testing conditions on the amount of corrosion of the test-piece immersed in the corrosive solution was observed. This amount is reported as the average penetration corr. for the vol. change of the test-piece. N. M. B.

Preparation of metal specimens for the microscope. R. C. STOCKTON (Metallurgia, 1936, 14, 57–58).— Methods of selecting, mounting, polishing, and buffing specimens are briefly described. A. R. P. Electrochemical protection of iron and steel against corrosion. C. G. FINK (Helv. Chim. Acta, 1936, 19, Spec. No., 59-68).—A lecture.

Electrolytic refining, and its place in the production of aluminium. E. I. CHAZANOV (Coll. Trans. 1st U.S.S.R. Conf. Non-aq. Solutions, Kiev, 1935, 174—190).—Electrolysis is conducted in a molten mixture of BaCl<sub>2</sub> 60, Na<sub>3</sub>AlF<sub>6</sub> 34, NaCl 3, and Al<sub>2</sub>O<sub>3</sub> 0-2% at 750°, with periodic addition of NaCl and AlF<sub>3</sub> to replace losses due to volatilisation (c.d. 0.5 and 1.5 amp./sq. cm. at the anode and cathode respectively; 3 volts). Using different Al alloys as the anode, 99.9% pure Al is deposited at the cathode in 90% yield.

R. T. Electric deposits on aluminium. G. ELSSNER (Aluminium, 1935, 17, 376—378).—The Al is degreased with  $C_2H_3Cl_3$ ,  $C_6H_6$ , or petroleum and washed with dil. HNO<sub>3</sub>, with or without addition of HF. The surface should be roughened, preferably by pickling in dil. HCl or HF. Sandblasting is objectionable. Alkaline pickling solutions are made from Na zincate with excess of NaOH and are used at 30—40°. Al which has been treated with zincate can be Ni-plated only in a citrate Ni bath with stirring. Plating Al with Cd, brass, or Cr requires an intermediate Ni layer.

Сн. Авз. (е)

Determination of free cyanide in cadmiumplating solutions. (A) J. DAWSON. (B) E. E. HALLS (Metallurgia, 1936, 14, 75, 75–76).—Difficulties encountered in the direct titration of the free KCN in  $K_2Cd(CN)_4$ baths are discussed. Fairly accurate results are obtained only when the NaOH content is 1—4 oz. per gal.; excess of NaOH produces an unsatisfactory end-point since the ppt. formed is granular, and a deficiency of NaOH produces high results since Cd(CN)<sub>2</sub> is appreciably sol. in  $H_2O$ . A. R. P.

Electroplating with metals and alloys in molten salts. N. N. GRATZIANSKI (Coll. Trans. 1st U.S.S.R. Conf. Non-aq. Solutions, Kiev, 1935, 213—216).—A review. R. T.

Characteristics of electrolytic metals. L. JENICEK (Rev. Mét., 1936, 33, 371–378).—Electrolytic Cr and Fe contract on annealing > Ni but no contraction has been observed in electrolytic Cu. The contraction is accompanied by a decrease in hardness but higher annealing temp. are required to attain max. softening. W. P. R.

X-Ray diffraction and the fatigue of metals. C. S. BARRETT (Physical Rev., 1935, [ii], 47, 333).—The radial widening of Debye lines is relatively insensitive to changes in the metal due to fatigue, but the peripheral widening of spots in Debye rings or the equiv. asterism is a sensitive detector of changes during fatigue.

L. S. T.

Welding of wires with condenser discharges. O. ZDRALEK and J. WRANA (Elektrotech. Z., 1935, 56, 579—580; Chem. Zentr., 1935, ii, 913).—A two-stage process for welding Cu, Al, constantan, and nickelin wires is described. J. S. A.

Pressure vessels.—See I. Ni and its alloys in petroleum refining.—See II. CN' solutions. Al from bauxite.—See VII. Steelworks refractories.— See VIII. Melting furnaces. Annealing furnaces. —See XI. Corrosion of milk tins.—See XIX.

#### PATENTS.

Pickling of ferrous metals. W. P. TER HORST, Assr. to INDUSTRIAL RAYON CORP. (U.S.P. 2,005,601, 18.6.35. Appl., 17.11.34).—The use of spinning bath solution from the manufacture of artificial silk and regenerated cellulose paper as an inhibitor is claimed.

L. C. M.

Determination of copper in copper-coated wire and the like. W. F. MUEHLBERG (U.S.P. 2,005,156, 18.6.35. Appl., 14.3.33).—The wire is immersed in aq. AgNO<sub>3</sub> (30 g. per litre) for 30 sec., and Cu determined in the solution either colorimetrically or by difference in wt. L. C. M.

[Nickel] alloys. J. M. LOHR, Assr. to DRIVER-HARRIS CO. (U.S.P. 2,005,430-3, 18.6.35. Appl., [A, B] 12.10.34, [c, D] 4.1.35).—Ni alloys for electricalresistance elements are claimed, containing (A) Cr 15—25 (20), Mo 1—20 (5), Zr 0.1—1 (0.2), and Ca 0.01—0.2(0.02)%; (B) Cr 15—25, Al 0.01—1 (0.07—0.38), Zr 0.01—0.5 (0.2), and Ca 0.01—0.2 (0.03)%; (c) Cr 10—15 (15), Fe 17—30 (25)%, and Mo, Zr, and Ca as in (A); (D) Cr 10—18 (10—15), Fe 17—30 (25—30)%, and Al, Zr, and Ca as in (B). L. C. M.

(A) Treatment of, (B) smelting, scrap storagebattery plates and lead-bearing materials of similar composition. (A, B) J. O. BETTERTON and (A) C. W. HANSON, ASSTS. to AMER. SMELTING & REFINING Co. (U.S.P. 2,006,256—7, 25.6.35. Appl., [A] 13.9.32, [B] 12.1.33. Renewed [A, B] 28.12.34).—(A) The material is heated in a reverberatory furnace at 900—980° with 1.6 wt.-% of C; petroleum coke is added to reduce the Pb in the slag, and the latter and matte are removed; the remaining Pb is agitated under oxidising conditions until the Sb content is < 0.02%. (B) The matte is broken down by adding Na<sub>2</sub>CO<sub>3</sub>, and a slag rich in Sb is obtained. L. C. M.

Spot-welding of refractory metals. W. W. EITEL and J. McCullough, Assrs. to HEINTZ & KAUFMAN, LTD. (U.S.P. 2,005,256, 18.6.35. Appl., 2.1.34).—Before welding, thermionic valve electrodes etc. of Mo, W, Ni, or Ta are coated very thinly with Ni; after welding the excess of Ni is removed by a dil. acid bath.

L. C. M.

Concentration of ores by flotation. C. H. KELLER, Assr. to MINERALS SEPARATION NORTH AMER. CORP. (U.S.P. 2,006,049, 25.6.35. Appl., 27.7.32).—Xanthic acid and its amyl, Bu, furfuryl, and other derivatives are claimed as flotation agents; 0.05 lb. is used per ton of ore. L. C. M.

Gold recovering device and method. J. B. GIRAND (U.S.P. 2,004,939, 18.6.35. Appl., 9.2.32).— The construction and use of a conical rotating-drum separator are claimed. L. C. M.

Pickling of metals. V. VOORHEES, Assr. to STAND-ARD OIL CO. (U.S.P. 2,005,605, 18.6.35. Appl., 28.11.31). —Crude shale oil distillate is agitated with 50% H<sub>2</sub>SO<sub>4</sub>, the acid is neutralised, and the org. bases set free are employed as an inhibitor. L. C. M. Salts from pickling solutions.—See VII. MetaI coatings.—See XIII.

## XI.-ELECTROTECHNICS.

Present state of electric furnace industries. G. FLUSIN (Helv. Chim. Acta, 1936, 19, Spec. No., 69-78).—A lecture.

Electric annealing furnaces and their heating elements. W. ROHN and J. E. W. GINGER (Metallurgia, 1936, 14, 7—9).—From both technical and economic viewpoints Ni–Cr heating elements are preferable to other types, but when an extended temp.range (> 1100°) is essential Cr–Al–Fe alloys and ceramic SiC rods are employed. Cr–Al–Fe alloys form a protective oxide film only above 1100°. E. H. B.

Repairs to electric melting furnaces. W. LISTER (Metallurgia, 1936, 14, 21–23).—The construction of non-conducting and conducting hearths is described. E. H. B.

Position and prospects of technical electrolysis. J. BILLITER (Helv. Chim. Acta, 1936, 19, Spec. No., 5-32).—A lecture.

Chemical spectral analysis of technical illuminating tubes, especially those with rare gas-mercury filling. H. SCHOBER (Z. tech. Physik, 1935, 16, 67–73; Chem. Zentr., 1935, ii, 893–894).—Cooling to --60° diminishes the intensity of the readily excited Hg lines in presence of the rare gases (I), without favouring the excitation of (I) sufficiently for spectral analysis. A sharp change in the colour of the discharge in Hg-Ne, Hg-Ne-A, or Hg-A mixtures occurs on cooling the walls to a definite temp. J. S. A.

Tantalum capacitators. B. H. PORTER (Transit J., 1935, 15, 14—15).—Ta is superior to Al for electrolytic condensers of very low resistance since its oxide is insol. in the electrolyte, and it resists corrosion by all materials except conc. alkalis and HF. The cathode of the Ta cell may be any material insol. in the electrolyte. Mo and Mo-W are satisfactory for use in H<sub>2</sub>SO<sub>4</sub> solutions. CH. ABS. (e)

Electro-graphite. Determining acid ityof insulating oil.—See II. Al from bauxite.—See VII. Corrosion protection of Fe. Welding of wires. Electrolytic refining. Electroplating. Cd-plating solutions. Plating A1.—See X. Eliminating salts from H<sub>2</sub>O.—See XXIII.

#### PATENTS.

Electric radiation furnace. Soc. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNY & CIREY (B.P. 449,745, 15.1.36. Fr., 16.1.35). —A monophase electric furnace comprising a resistor formed of one or more conducting rods and a metallic non-magnetic envelope, *e.g.*, of Cu, arranged near the periphery of the furnace for return of the current, is claimed. J. S. G. T.

**Primary batteries.** NAT. CARBON Co., INC. (B.P. 449,587, 17.6.35. U.S., 17.8.34).—A battery comprising a regenerating material (R), e.g., CaO, in contact with the electrolyte to remove deleterious substances from

solution, and an electrolyte-permeable extender, e.g., asbestos distributed throughout R, is claimed.

J. S. G. T.

Electrolytic condensers. PLESSEY Co., LTD. From P. R. MALLORY & Co., INC. (B.P. [A] 449,645, 2.7.35, and [B] 449,839, 13.6.35).—(A) At least one electrode is made of an alloy of Ta or Nb with  $\bigstar$  98% Al. (B) A conducting spacer-member comprising a thin, flexible, plasticised sheet of albuminous material, *e.g.*, gelatin, impregnated with an electrolyte which may be a plasticiser, *e.g.*, a glycol or glycerol compound, is claimed. J. S. G. T.

Electric-discharge tubes and screens for use therein. N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 449,727, 4.9.35. Ger., 9.10.34).—The electrodes are separated by electrostatic screens of C or graphite.

J. S. G. T.

Dielectric composition. J. E. MOOSE and J. E. MALOWAN, ASSTS. to SWANN RES., INC. (U.S.P. 2,010,495, 6.8.35. Appl., 24.8.32).—The fraction, b.p.  $211-245^{\circ}$ , obtained by chlorinating  $C_6H_6$  until the product has  $d^{25}$  1.37 is frozen or treated with small amounts (usually 5-8%) of  $C_6HCl_5$  or  $C_6H_2MeCl_3$ . The f.p. of the product, which has a dielectric const. of 4, may be thus regulated between 4° and 11°. R. S. C.

Electric accumulators. CHLORIDE ELECTRICAL STORAGE CO., LTD., and A. W. BROWNE (B.P. 449,744, 10.1.36).

Halogenated benzophenone and diphenylmethane.—See III. Ni alloys. Storage-battery plates. Spot-welding.—See X. Rubber compositions.— See XIV.

#### XII.—FATS; OILS; WAXES.

Tests for butter and margarine fats. J. GROSS-FELD (Fette u. Seifen, 1936, 43, 100–103).—Improvements in the authors' analytical methods are discussed. Butter and coconut fats may be estimated by determination of the  $PrCO_2H$  val. and the total lower fatty acid content : the lauric acid val. serves as confirmation. The *iso*oleic val. is used for determining the presence of hardened oils in foodstuffs. Analysis may be made on 1.5 g. of fat. F. C. B. M.

Refractometric determination of fat in copra. J. P. GROENHOF (Pharm. Weekblad, 1936, 73, 1002— 1016).—The fat content of copra can be determined rapidly and accurately, by grinding 5 g. with 5 g. of  $CH_2Ph$ ·OH, tetralin, or  $C_{10}H_7Cl$ , and 5 g. of sand, and measuring *n* of the resultant solution. Measurements are conveniently carried out with a Zeiss immersion refractometer having a double prism mounted on the tube and reading to 5 decimals. S. C.

Surface tension of fatty acid condensation products and sulphonated fatty alcohols. W. WELTZIEN and H. OTTENSMEYER (Fette u. Seifen, 1936, 43, 91–93). —Purified Gardinol (an aliphatic alcohol sulphonate) and Igepon A and T (fatty acid condensation products) decrease surface tension ( $\gamma$ ), measured with a Traube stalagmometer at 20° and 80°, < unpurified samples. Addition of electrolytes to purified specimens produces marked lowering of  $\gamma$  in dil. solutions, and their presence in commercial samples is assumed to be, in part, the cause of their activity. F. C. B. M. Analysis of ester mixtures containing two saturated and two unsaturated fatty acids. T. P. HILDITCH (Fette u. Seifen, 1936, 43, 97—100).—The author's method of analysis is illustrated with data obtained on a partially hydrogenated S. Georgia whale oil. The Me esters of the solid and liquid acids, obtained by a modified Twitchell method, are fractionated, and the sap. equivs. and I vals. determined for each fraction. Unsaturated esters are then destroyed by KMnO<sub>4</sub> oxidation of each fraction and the sap. val. of the residual saturated material is determined. From the above figures the composition of the ester mixture may be calc. F. C. B. M.

"Wecker" fatty acid distillation process. E. M. SCHÖNBERGER (Fette u. Seifen, 1936, 43, 109–112).— The finely-divided preheated fat or fatty acid is treated with  $H_2O$  vapour, the temp. difference being of the order 240—270°. The  $H_2O$  particles expand explosively and remove the volatile constituents from the reaction chamber. The process is continuous and is conducted at 15—25 mm. pressure; it is characterised by no increase in saponifiable matter, lack of distillation odour, good colour of distillate, no change in I val., avoidance of tarry residue, and independence of the degree of saponification. F. C. B. M.

Preparation of milled toilet soap with modern drying and milling equipment. ANON. (Seifensieder-Ztg., 1935, 62, 362-363, 381-382; Chem. Zentr., 1935, ii, 776).—Technical details are given. The importance of adjustment of free acid or alkali and NaCl content is noted. A. G. P.

**Stand-oil formation.** J. SCHEIBER (Fette u. Seifen, 1936, 43, 103—105, and Farbe u. Lack, 1936, 315—316, 329—330, 341, 351—352).—Mainly a reply to Kurz (B., 1936, 749). Stand-oil formation is assumed to be due, primarily, to ethylenic polymerisation, preceded in the case of linseed oil, perilla oil, etc. by a shifting of the double linkings to form a conjugated unsaturated system. The formation of free fatty acids and the disappearance of glycerol observed during linseed stand-oil production is ascribed to the pyrolytic fission of triglyceride mols. into free acids and vinyl compounds, and not to condensation of a glyceride mol. with a reactive  $CH_2$  of a second glyceride mols. F. C. B. M.

Oil formation in groundnuts. J. S. PATEL and C. R. SESHADRI (Agric. Sci. [India], 5, No. 2; Bull. Mat. Grasses, 1936, 20, 169—172).—The free fatty acid content of the oil is high in the early stages of development and declines rapidly as the oil content of the groundnut increases. A. G. P.

Polymerisation of some [Italian] vegetable oils. I. Grape-seed oil. M. BRAMBILLA and G. BALBI (Chim. e l'Ind., 1936, 18, 353-355).—By heating the oil (e.g., for 2 hr.) at 330° in a stream of CO<sub>2</sub>, a stand oil of pale colour and good drying properties may be prepared. The acid val. is, however, high, rising from 8.97 to 27 during 2 hr. cooking.  $\eta$ , d, n, and I, thermosulphuric, and acid vals. are given for the raw oil and for the oil cooked for various periods up to 150 min.

D. R. D.

Nomograph for iodine value of tung oil. C. S. WAN and K. Ho (Ind. Eng. Chem. [Anal.], 1936, 8, 282-283).—The nomograph gives the corrections to be applied for various excesses of Wijs reagent and working temp. from 16° to 40°. S. C.

Analysis of " EL boiled oil." E. FONROBERT and A. MÜNCHMEYER (Farben-Ztg., 1936, 41, 747-748).-Solvent is determined by heating the EL boiled oil for 4 hr. at 105°.  $o-C_6H_4(CO_2H)_2$  (I) is separated from resin acids (II), fatty acids (III), and unsaponifiable matter (IV) by saponifying with excess of KOH-EtOH (for  $\lt$  6 hr.), distilling off most of the EtOH, dissolving the soaps in H<sub>2</sub>O, acidifying with HCl, and shaking with  $C_6H_6$  in which (II) and (III) are sol., the insol. (IV) floating in the C6H6 layer, and the (I) remaining in the aq.-EtOH later. After separating and washing the layers, (I) is pptd. as o-C<sub>6</sub>H<sub>4</sub>(CO<sub>2</sub>)<sub>2</sub>Pb, and converted into PbSO<sub>4</sub>, from the wt. of which the (I) content is calc. (II), (III), and (IV) are separated by known methods. The accuracy of the process is shown by examples. The advantages of determining (I) as Pb salt (rather than as K salt according to Kappelmeier) are discussed.

S. S. W. Determination of saponification value of paprika oil in filtered ultra-violet light. E. A. Kocsis and P. Vass (Z. Unters. Lebensm., 1936, 71, 442–445).— 4-Methylumbelliferone was used as a fluorescent indicator. The sap. val. of 6 samples of oil was 186.8—187.5. E. C. S.

**Report of American Chemical Society Committee** on analysis of commercial fats and oils. W. H. IRWIN, R. W. BAILEY, T. C. LAW, C. P. LONG, H. J. MORRISON, M. L. SHEELY, L. M. TOLMAN, H. P. TRE-VITHICK, and J. J. VOLLERTSEN (Ind. Eng. Chem. [Anal.], 1936, 8, 233-237).-Detailed official methods are described for (a) a modified Wiley m.-p. method in EtOH-H<sub>2</sub>O, (b) determination of the SCN val. using Pb(SCN)<sub>2</sub> and Br in anhyd. AcOH, (c) separation of liquid and solid fatty acids by a modified Twitchell Pb-salt method, suitably corr. by the I and SCN vals. of the solid acids, and (d) detection of tristearin in unhydrogenated pork fats by crystallisation from COMe<sub>2</sub> and determination of the m.p. of the separated triglycerides (I) and of the fatty acids (II) obtained from them on hydrolysis. Adulteration has occurred if the sum of the m.p. of (I) plus twice the difference between the m.p. of (I) and (II) is  $< 73^{\circ}$ . S. C.

Schiebler filter.—See I. Vegetable oils and Diesel motors.—See II. Mustard.—See XVI. Soya bean. Storage of ghee.—See XIX. Margarine effluents. —See XXIII.

#### PATENTS.

Manufacture of shaped washing and cleansing agents. I. G. FARBENIND. A.-G. (B.P. 443,795, 9.10.34. Ger., 10.10.33).—H<sub>2</sub>O-sol. salts of amines or quaternary NH<sub>4</sub> bases having at least one alkyl of  $\measuredangle C_6$ , or the NH<sub>4</sub> bases themselves, are mixed with at least one representative of the following classes and pressed into bars etc.: H<sub>2</sub>O-sol. polymerisation or condensation products of high mol. wt. derived from oxy-genated substances of low mol. wt., e.g., (CH<sub>2</sub>)<sub>2</sub>O condensation products; superfatting agents; org. fat

solvents; carbohydrates or their  $H_2O$ -sol. derivatives; softening agents, e.g.,  $PO(O \cdot C_6H_4Me)_3$ , glycerol; inorg. filling agents, e.g., bentonite, talc, Na silicate, and  $H_2O$ -sol. salts. H. A. P.

Breaking petroleum emulsions.—See II. Thiosulphate esters.—See III. Oil colours.—See IV.

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

Wood primers. W. LUDWIG (Farben-Chem., 1936, 7, 252—254, 292—294, 301).—The structure and swelling power of soft and hard woods are discussed with particular reference to spring and summer growths and the penetration, elasticity, and adhesion of ground coats containing various vehicles and pigments. The paint film should not be completely gelled, but should retain some of the dispersion medium either as solvent or, in the case of drying oils, as saturated acids; such a primer will not dry rapidly. Al powder is superior to other pigments in promoting  $H_2O$ -resistance and durability. S. M.

Problems in the paint industry. H. C. F. RANDALL (J. Oil & Col. Chem. Assoc., 1936, 19, 203-219).— Factors which influence the grinding of paints and their consistency are discussed. An instrument for measuring the latter is described. S. M.

Panel-test evaluation of exterior house paints. G. W. ASHMAN (Ind. Eng. Chem., 1936, 28, 934-939).-Panel-exposure tests show that durability is generally decreased when (a) the amount of oil used is sufficient to form only a stiff instead of a soft paste, (b) there is a long drying period between application of each coat, (c) the paint is applied and dried indoors, and (d) the back of the panel is not painted. The reported durability varies also with ageing of the paint in the can and of the fresh film before exposure, the type of wood, paints applied on weathered films, the colour, the skill of the painter, the season, weather, climatic extremes, industrial and other atm. conditions, the size and inclination of the panel and the accelerated nature of panel tests, and the personal factors. S. M.

Paint in the brewery. M. DE LA CROIX (Bull. Assoc. anc. Étud. Louvain, 1935, 35, 59-74; Chem. Zentr., 1935, ii, 937).—The suitability of various paints for use in the humid atm. of a brewery is discussed.

H. N. R.

Apparatus for determining non-inflammability and fireproof properties of paints and varnishes. G. BALBI and G. MARCHESINI (Chim. e l'Ind., 1936, 18, 348-349; cf. B., 1935, 1151).—Means for measuring the temp. of the film more accurately are described.

D. R. D.

Pigments for primers. F. Kolke (Farben-Chem., 1936, 7, 250-251).—A discussion.

Hydrated chromium oxide green [pigments]. H. WAGNER and A. RENE (Farben-Ztg., 1936, 41, 821– 823).—The structure and properties of coarse and fine samples of genuine Guignet's green (prepared by calcination) are described and tabulated alongside those of a pptd. hydrated  $CrO_3$ . X-Ray examination shows the former to be amorphous, whilst the latter has definite crystal structure. This is correlated with differences in flowing properties, oil absorption, sedimentation, etc. of these pigments. S. S. W.

Morphology of pigments. IV. Microscopy of soot. A. V. PAMFILOV and O. S. FEDOROVA (J. Gen. Chem. Russ., 1936, 6, 645—649).—Lampblack can be distinguished microscopically from other forms of soot. R. T.

Analysis of colours from pre-colonial Peru. J. A. PARERA and O. BACA (Anal. Fis. Quim., 1935, 33, 244—246).—Coloured earths found in excavations at Saccsayhuaman have the compositions of azurite, orpiment, realgar, cinnabar, and SnAs<sub>4</sub>S<sub>12</sub>. F. R. G.

Manufacture of Lake Red D for printing inks and paints. H. BOSSEL (Farben-Chem., 1936, 7, 245— 246).—The method is outlined. For printing inks  $Al(OH)_3$ -blanc fixe is added; for paints heavier substrates, *e.g.*, barytes, are used. Treatment with BaCl<sub>2</sub> gives a bluish-red product. S. M.

Drying of printing inks. R. BEUERLE (Farben-Chem., 1936, 7, 246—250).—Exposure tests (tabulated) show the accelerating effect of light (particularly ultraviolet) and the decelerating effect of moisture on the drying time of C-black stand-oil inks with and without Co driers. These inks dried also in a  $CO_2$  atm. free from  $O_2$ . It is inferred that stand oils dry mainly by condensations which take place at the CH<sub>2</sub> groups, followed by coagulation. The reactions involved in the setting of linseed oil are reviewed. S. M.

Relation of thinners in overlapping varnish coatings. J. K. STEWART and H. L. BEWICK (Ind. Eng. Chem., 1936, 28, 940-943).-The wet-edge time (tw) is the max. allowable interval between the application of two varnish strips so that a permanently visible junction is not formed.  $t_w$  (method of determination described) of a 100% phenolic resin-tung oil varnish diluted with kerosene and mineral spirits in various proportions varied from 18 to 27 min., and reference to evaporation curves shows that at that stage 70-80% of the thinners are retained.  $t_w$  was affected by the type of kerosene and was reduced by addition of BuOH.  $\eta$ -thinner curves show that the  $\eta$  of the film is at a crit. stage during the  $t_w$  interval, and immediately afterwards increases rapidly. S. M.

Evaporation of solvents from varnishes. H. WOLFF and G. ZEIDLER (Farben-Chem., 1936, 7, 285-292).— $\eta$  changes in a film during initial formation are obtained by measuring the times taken for a steel ball to roll down the supported inclined film (apparatus described) at intervals during the evaporation of the solvents. With most varnishes equations for rate of increase of  $\eta$  resembled closely those developed by Blom (B., 1931, 308) for rate of evaporation of the solvents. The  $\eta$  thus obtained of a 16% nitrocellulose solution in C<sub>6</sub>H<sub>6</sub>-BuOAc rapidly overtook that of a 19% solution; with PhMe in place of C6H6 the rate of increase of  $\eta$  was the same for both concns.; with xylene in place of  $C_6H_6$  the 16% solution showed a smaller rate of  $\eta$  increase. These variations are explained by differences in (a) the initial  $\eta$  and (b) the behaviour of the mixed solvents during evaporation and the consequent effects on the conces. Some data obtained with

 $o-C_6H_4(CO_2Bu)_2$  and  $(C_6H_4Me)_3PO_4$  as plasticisers are also discussed. S. M.

Topical structure of oil molecules and the filming process. B. F. H. SCHEIFELE (Kolloid-Z., 1936, 76, 111).—A criticism (cf. B., 1936, 558). E. S. H.

Effect of chemicals on phenol-resin bonded laminated [products]. H. E. RILEY (Ind. Eng. Chem., 1936, 28, 919–922).—Laminated canvas panels bonded with a bakelite resin were exposed to H<sub>2</sub>O, various acids, NaOH (1 and 20%), oxidising agents, and alkali salts for 180 days. The wt. increases (graphed) in nearly all cases varied from 2.5 to 4%; at higher temp. the initial absorptions were more rapid, but the ultimate increases were unaffected. Increases in thickness after 10 days generally followed the wt. increases and were greater with dil. than with conc. reagents. Physical, mechanical, and electrical properties of laminated products are listed and some uses discussed. S. M.

Viscosity in solution and condensation velocity of phenol-formaldehyde resins. K. H. KLAASSENS and R. HOUWINK (Kolloid-Z., 1936, 76, 217–223).— The relative viscosity of the solutions,  $\eta_r$ , varies with concn. (up to 40%) in accordance with log  $\eta_r = x c_s^a$ , where  $c_v$  is the vol.-concn. of the disperse phase, and x and a are consts. a varies with the shape of the disperse particles and dx/dt (where t is the condensation time) can be used as a measure of condensation velocity. Results are given for four resins. E. S. H.

Measuring plasticity.—See I. White alloys in printing.—See X. Stand oil. Grape-seed oil.— See XII. Soya bean.—See XIX. Synthetic camphor. —See XX.

#### PATENTS.

Decoration of hard surfaces.  $O_{XVAR}$ , LTD. From OXFORD VARNISH CORP. (B.P. 448,003, 10.4.35).—In simulating wood grain etc. by the half-tone or photogravure process the design is printed on the ground coat with a translucent ink which contains a non-sol. flaky material, *e.g.*, powdered bronze, Al, homogeneously dispersed in an oil, nitrocellulose, or other medium, the reflecting power of which is < that of the pigment particles. The whole is protected with a finishing coat of varnish or lacquer. S. M.

Coating materials [for sealing cans]. A. L. KRONQUEST and S. C. ROBISON, ASSTS. to CONTINENTAL CAN CO., INC. (U.S.P. 2,009,776-8, 30.7.35. Appl., [A] 5.3.31, [B, c] 4.4.31. Renewed [A, B] 7.5.35, [C] 27.10.34).--(A) A dough prepared from rubber (30 pts.) dissolved in naphtha or other solvent (70), ZnO (100), adhesive ester gum (15), liquid paraffin (3), and an antioxidant ( $\frac{1}{3}$ ) is thinned before use with benzol etc. (B) S is also incorporated with the dough and a vulcanisation accelerator is added with the thinner. (c) A composition which remains tacky for several months is obtained by milling ZnO (80) with rubber (30) before adding the solvent and other components. S. M.

[Plasticisers for] cellulose derivative compositions. E. I. DU PONT DE NEMOURS & Co., and M. M. BRUBAKER (B.P. 447,764, 21.11.34).—The use of esters of hydrogenated castor oil with a polycarboxylic acid, e.g., o-C<sub>6</sub>H<sub>4</sub>(CO)<sub>2</sub>O, succinic, citric, is claimed. S. M.

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Production of metal coatings on celluloid substitutes. M. Ow-ESCHINGEN (U.S.P. 2,010,805, 13.8.35. Appl., 22.11.32. Austr., 3.12.28).—Articles made from casein or synthetic resins are silvered by immersion first in a solution of a reducing agent  $[p-C_6H_4(OH)_2, OH \cdot C_6H_4 \cdot NH_2, C_6H_4(NH_2)_2]$  and then, after drying, in AgNO<sub>3</sub> solution. S. C.

Manufacture of colour lakes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 448,508, 5.12.34).— Cyanines, carbocyanines, and styryl compounds containing an indolenine nucleus (which may contain acid groups), when treated with complex compounds of tungstic or molybdic acid, give colour lakes which are especially fast to light, oil, and spirit, and superior to the corresponding lakes from triarylmethane dyes. *E.g.* (1) 1:3:3:1':3':3'-hexamethylindocyanine chloride treated in H<sub>2</sub>O with a complex phosphotungstomolybdic acid gives a yellow ppt.; (2) 6:6'-dimethoxy-1:3:3:1':3':3'-hexamethylindocarbocyanine chloride, treated with Al(OH)<sub>3</sub> paste and a complex silicotungstic acid, gives a violet colour. F. M. H.

Stencil paste. J. W. ILIFF, P. ROBINSON, and W. WHITESCARVER, ASSTS. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,011,898, 20.8.35. Appl., 2.5.30).—The binder for the pigment etc. consists of a drying oil-modified alkyd resin to which is added a dil. aq. or EtOH solution of a soap of a bivalent metal (Ca, Mg); the effectiveness in preventing creeping of the ink beneath the stencil is increased by adding also a soap of a univalent metal, e.g.,  $NH_4$  oleate. S. M.

Manufacture of gasproof [tung oil] varnish. J. HYMAN, ASST. to VELSICOL CORP. (U.S.P. 2,012,279, 27.8.35. Appl., 28.1.33).—Tung oil is bodied at  $\geq 218^{\circ}$ , resins are incorporated, and an aromatic amine, phenol, or thiophenol, e.g., NH<sub>2</sub>Ph,  $\beta$ -C<sub>10</sub>H<sub>7</sub>SH, is added together with the usual driers and thinners. The films do not crack or frost when stoved. S. M.

Synthetic [phenol-aldehyde] varnish composition. J. C. WOODRUFF, Assr. to RESINOX CORP. (U.S.P. 2,010,123, 6.8.35. Appl., 11.5.31).—Neutral esters of  $H_3PO_4$  and aliphatic alcohols, *e.g.*,  $Bu^a_3PO_4$ , are incorporated with thermo-hardening phenol-aldehyde resins; the films are baked at 127—175°. S. M.

Cellulose mixed-ester coatings. C. R. FORDYCE and J. D. COLEMAN, JUN., Assrs. to EASTMAN KODAK CO. (U.S.P. 2,013,825, 10.9.35. Appl., 21.5.32).—Mixed org. esters of cellulose containing an acid radical  $> C_7$ , e.g., cellulose acetate-stearate, are heated with vegetable oils or other non-volatile org. softeners until homogeneous; the products solidify on cooling and are used to form protective coatings on wire etc. S. M.

Manufacture of plastic and coating compositions having a basis of cellulose ester or ether. CELLULOID CORP. (B.P. 450,454, 14.1.35. U.S., 12.1.34).—Cellulose nitrate etc. are plasticised with a tri-ester of  $H_3PO_4$ and a phenol which has a nuclear substituent (>  $C_2$ ), *e.g.*, *p-tert*.-amylphenyl phosphate (prep. described). S. M.

Manufacture of pigmented cellulose-derivative compositions and of artificial filaments, films, lacquers, plastic masses, and other materials containing them. BRIT. CELANESE, LTD. (B.P. 450,285, 10.10.35. U.S., 20.10.34).—Improved dispersion is claimed by working up the pigment with a mixture of cellulose acetate or other ester or ether and  $\geq 10\%$  of its wt. of an oil, *e.g.*, low- $\eta$  castor, tung; a softening agent is not used. S. M.

Sulphonation of rosin and its derivatives. H. J. WEILAND and M. A. PRAHL, Assrs. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 2,015,023, 17.9.35. Appl., 15.2.34).—SO<sub>3</sub> is distilled into a solution of rosin or derivative, *e.g.*, abietene, in an inert solvent, *e.g.*,  $C_2H_2Cl_4$ , maintained at  $< 20^\circ$ .  $H_2O$  is preferably excluded. S. M.

Heterocyclic esters of resin acids. J. N. BORGLIN, Assr. to HERCULES POWDER CO. (U.S.P. 2,012,622, 27.8.35. Appl., 11.6.32. Renewed 24.1.35).—Rosin, abietic acid or a salt thereof is heated with a heterocyclic monohydric alcohol, *e.g.*, furfuryl, preferably in presence of HCl or other acid, as catalyst, and PhMe etc. to facilitate removal of  $H_2O$ . The products may be used in lacquers or as a colloiding agent for nitrocellulose. S. M.

Shellac (A) composition containing dicyanodiamides, (B) -sulphanilic acid composition, (c) composition for moulding and protective coatings, (D) composition containing a polycarboxylic acid or its anhydride. W. H. GARDNER, Assr. to U.S. SHELLAC IMPORTERS' ASSOC., INC. (U.S.P. 2,010,224-7, 6.8.35. Appl., [A-c] 22.10.32, [D] 27.10.32).—The softening point of shellac is increased by heating it with (A) dicyanodiamide, (B) sulphanilic acid, (c)  $\alpha$ -o-tolyldiguanide, (D) o-C<sub>8</sub>H<sub>4</sub>(CO)<sub>2</sub>O, succinic acid, etc. S. M.

Preparation of resin product from cashew nutshell liquid. F. M. DAMITZ, ASST. to HARVEL CORP. (U.S.P. 2,014,370, 17.9.35. Appl., 27.9.32; cf. B., 1932, 900).—The liquid is heated to about 315° with a fossil resin, *e.g.*, kauri, ester gum, rosin, etc., until the cooled product is brittle. It is then used for making air-drying varnishes by cooking with, *e.g.*, linseed oil and a drier. S. M.

Manufacture of stable vinyl resins. C. O. YOUNG and S. D. DOUGLAS, ASSTS. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 2,013,941, 10.9.35. Appl., 28.3.31).—Vinyl polymerides are rendered stable by incorporating < 3% of basic compounds, e.g., NH<sub>3</sub>, arylor alkyl-amines, (CH<sub>2</sub>)<sub>6</sub>N<sub>4</sub>, N(C<sub>2</sub>H<sub>4</sub>·OH)<sub>3</sub>, CaO. S. M.

Manufacture of [thermoplastic] resinous complexes and compositions of synthetic origin containing sulphur. C. ELLIS, ASST. to ELLIS-FOSTER Co. (U.S.P. 2,011,573, 20.8.35. Appl., 15.6.29).—Urea (1 mol.) and NH<sub>4</sub>CNS (1 mol.) are condensed with aq. CH<sub>2</sub>O ( $\lt$  2 mols.); the product is dehydrated at 90—100°. S. M.

(A) Manufacture of urea and/or urea derivative plastic. (B) [Urea-aldehyde] resinous substance, (c) condensation product. C. ELLIS, (B, c) Assr. to ELLIS-FOSTER CO. (U.S.P. 2,009,545, 2,009,986-7, 30.7.35. Appl., [A] 20.4.29, [B] 10.1.29, [c] 28.1.24 and 21.9.32).—(A) A phenol-aldehyde, phenol-S, or natural resin is incorporated with an org. acid-catalysed urea-aldehyde resin (I) which hardens when pressed at 110-150°, and a filler. (B) (I) is incorporated with a S-bound resin, e.g., dimethylolurea with a phenol-S product, (I) with a  $CS(NH_2)_2$ -aldehyde resin. (c) (I) is incorporated with a phenol- $CH_2O$  resin free from uncombined phenol. S. M.

Moulding of urea-formaldehyde condensation products. L. SMIDTH, ASST. to ALDUR CORP. (U.S.P. 2,013,702, 10.9.35. Appl., 25.2.31).—The material used is plastic so that pressures of approx. 2000 lb./sq. in. suffice. The mould is initially partly closed by inserting a spacing means, and is opened for about 30 sec. to allow degassing and stiffening of the contents. S. M.

**Production of synthetic [phenol-aldehyde]** resins. S. P. BURKE and H. M. ENTERLINE, Assrs. to COMBUSTION UTILITIES CORP. (U.S.P. 2,010,773, 6.8.35. Appl., 22.8.29).—Potentially reactive resins suitable for the prep. of laminated products are obtained by heating an alkaline solution of high-b.p. tar acids (from lowtemp. tar) and  $\measuredangle$  an equiv. quantity of CH<sub>2</sub>O with an intermediate PhOH-CH<sub>2</sub>O resin. S. M.

[Modified alkyd] resinous compositions. M. M. BRUBAKER and R. E. THOMAS, ASSTS. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,009,432, 30.7.35. Appl., 3.5.32).—In preparing alkyd resins, 15-40% of the  $o-C_6H_4(CO)_2O$  or other polybasic acid is replaced by the equiv. amount of an aminocarboxylic acid, *e.g.*, glutamic. S. M.

Lævulic esters. Ether acid esters. Protein mixtures.—See III. Treating fabrics.—See VI. Blanc fixe.—See VII. Laminated articles.—See VIII. Marble.—See IX.

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

Bacterial decomposition of the rubber in Hevea latex. D. SPENCE and C. B. VAN NIEL (Ind. Eng. Chem., 1936, 28, 847-850; cf. B., 1935, 1005).-Uncoagulated sterile latex was obtained by dialysing NH<sub>2</sub>-preserved latex against a strongly buffered solution of approx. the same  $p_{\rm H}$  as fresh Hevea latex (6.8-7.2) and then heating for 15 min. at 120°. Such latex creamed on keeping but could still be redispersed by gentle shaking after 6 months in the dark. The N necessary for bacterial growth was added as KNO<sub>3</sub>, NH<sub>4</sub>Cl being unsuitable as it caused coagulation during sterilisation. Actinomyces derived from garden soil caused the disappearance of > 70% of the rubber hydrocarbon of the latex in 4 weeks at 30°; the brown residue (black when dry), in addition to bacteria, contained a soft, almost oily product sol. in  $C_6H_6$  and a  $C_6H_6$ -insol. material, probably "insol." rubber. Fresh latex would probably be even more susceptible to bacteria. The results explain the poor physical character and dark colour of earth- and bark-scrap rubbers. D. F. T.

Measuring plasticity.—See I. Determining CS<sub>2</sub>. —See III.

#### PATENTS.

Treatment of rubber. BELVEDERE CHEM. Co., LTD., Assees. of L. H. HOWLAND (B.P. 447,954, 5.10.35. U.S., 23.1.35).—Rubber is preserved by incorporating a product of the reaction of an alkali metal (Na) on a ketone-amine condensation product (e.g., from COMe<sub>2</sub> and NHPh<sub>2</sub>), from which free and combined alkali metal may have been subsequently removed. D. F. T.

Manufacture of rubber articles. INTERNAT. LATEX PROCESSES, LTD. (B.P. 447,870, 10.10.35. U.S., 27.10.34).—A rubber article of desired varying thickness is obtained by providing a form, which may have been coated with uncoagulated latex, with a film of coagulant (e.g.,  $EtOH-CaCl_2$ ) and reducing the effectiveness of the coagulant (e.g., by aq. KOH) in definite areas before applying latex thereto. D. F. T.

Rubber composition containing asphaltene. P. K. FROLICH, ASST. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 2,009,712, 30.7.35. Appl., 2.9.32).—Rubber is compounded with asphaltene powder or "flowers" of asphalt derived from cracked petroleum tar and substantially free from hydrocarbon oils and resins.

D. F. T.

[Manufacture of] rubber compositions and articles. LIVERPOOL ELECTRIC CABLE Co., LTD., and A. E. HUGHES (B.P. 447,926, 22.12.34).—A rubber mixture includes a fire-resistant filler comprising a chlorinated org. compound other than chlorinated  $C_{10}H_8$ or pitch, e.g., chlorinated mineral rubber (containing approx. 30% Cl), and Se. A prescribed mixture (for electric cables) is rubber 33, C black 0.5, S 0.5, kaolin 5, MgCO<sub>3</sub> 42, chlorinated mineral rubber 14, chlorinated  $C_{10}H_8$  2, Se 1 $\frac{1}{2}$ , with antioxidant and accelerator if desired. D. F. T.

Vulcanisation of rubber. A. J. NORTHAM, ASST. to E. I. DU PONT DE NEMOURS & Co., INC. (U.S.P. 2,006,057, 25.6.35. Appl., 22.8.33).—The use of mono-, di-, or tetra-methyl- or -ethyl-thiuram mono- or poly-sulphides as accelerators is claimed. L. C. M.

Antioxidants [for rubber]. (A) D. CRAIG, (B) W. L. SEMON, (C) A. W. SLOAN, Assrs. to B. F. GOODRICH Co. (U.S.P. [A] 2,009,480, [B] 2,009,526, and [C] 2,009,530, 30.7.35. Appl., [A] 10.6.32, [B], 2.6.33, [C] 26.5.33).-(A) Deterioration of rubber is retarded by a diarylamine substituted with an aliphatic hydrocarbon radical  $\ll C_2$ , e.g., p-isopropyl-, p-hexadecyl-, or pp'-ditert.-butyldiphenylamine or an alkoxy-derivatives thereof. Rubber is preserved with a s-diarylnaphthylenediamine,  $e.g., 1: 4-C_{10}H_6(NHPh)_2$ . (c) Rubber is preserved with a substance of the formula A·R·X·R'·A', A and A' being (substituted) NH<sub>2</sub>, X an aliphatic nucleus, and R and R' aromatic nuclei attached to different C atoms in X, e.g., pp'-diamino- or pp'-dianilino-dibenzyl. D. F. T.

Showerproof fabrics.—See VI. Coating materials.—See XIII.

#### XV.—LEATHER; GLUE.

Pilot-scale calfskin curing with treated salt. R. W. FREY and L. S. STUART (J. Amer. Leather Chem. Assoc., 1936, 31, 254—265; cf. B., 1935, 114).—Raw calfskins were cured with NaCl, NaCl + Na<sub>2</sub>SiF<sub>6</sub> +  $C_{6}H_{2}Cl_{3}$ ·ONa, and NaCl + p-NO<sub>2</sub>· $C_{6}H_{4}$ ·OH, respectively, and subsequently processed for chrome upper leather. No differences were observed in the finished leathers although the NaCl-cured skins had shown signs of putrefaction. The processing was not affected by the other chemicals added to the NaCl cure. D. W. [Physical and chemical tests on] white leather. Report of a Committee [of the American Leather Chemists Association]. A. C. ORTHMANN (J. Amer. Leather Chem. Assoc., 1936, 31, 250–253).—Proposed tests to be made are listed. D. W.

Electronic theory of tanning. III. Vegetable tanning. J. A. WILSON (J. Amer. Leather Chem. Assoc., 1936, 31, 265–272; cf. B., 1936, 706).—In vegetable tanning, the covalent type of combination takes place over the whole range of  $p_{\rm H}$  vals., but both  $\infty$ - and electro-valent combinations occur in the range where the electrical charges on tannin and protein are of opposite sign, *i.e.*,  $p_{\rm H} 2 \cdot 5 - 5 \cdot 0$ . The Procter-Wilson theory applies only to the electrovalent type of combination. D. W.

Ageing tests with leathers tanned in part with sulphite-cellulose [waste] extract. H. B. MERRILL (J. Amer. Leather Chem. Assoc., 1936, 31, 272-274).— Calfskin pieces tanned with a mixture of the extract and a natural vegetable-tannin extract were practically unaffected by storage for 4 years in dim daylight without temp. or humidity control. D. W.

Halogeno - tannins.—See XX. Photographic gelatin.—See XXI.

#### PATENTS.

**Preparation of adhesive.** A. H. BOWEN, Assr. to I. F. LAUCKS, INC. (U.S.P. 2,014,167, 10.9.35. Appl., 4.4.33).—Proteinous materials, *e.g.*, casein, seed meals, are treated with an equal wt. of  $ZnCl_2$  in presence of  $H_2O$  or an alcohol; drying properties and improved  $H_2O$ -resistance are obtained by treating the product with urea and  $CH_2O$  in described manner. S. M.

Protein mixtures. Sulphonated products.—See III. Azo dyes for leather.—See IV. Dyeing leather. —See VI.

#### XVI.—AGRICULTURE.

Soil types of Tunis. V. AGAFONOV (Compt. rend., 1936, 202, 2000—2002).—Analytical data for soils from various regions are recorded and discussed. H. J. E.

Boring tool for volumetric removal of soil samples from any depth. E. A. MITSCHERLICH and H. BEUTELSPACHER (Z. Pflanz. Düng., 1936, 44, A, 310-315). A. G. P.

Sieve and elutriation analysis of soil. A. VON NOSTITZ (Z. Pflanz. Düng., 1936, 44, A, 256-264).--Round- and square-mesh sieves gave appreciably different results in mechanical analysis. No significant difference was apparent between results of dry and wet sieving. Sieving and elutriation methods gave different vals. only when the soil contained considerable amounts of mica. A. G. P.

Influence of the water content on the  $p_{\rm H}$  value [of soils]. D. FERÉR (Z. Pflanz. Düng., 1936, 44, A, 341-346).—A reply to criticisms of Deines and Kurbis (B., 1935, 1108) of the author's method (*ibid.*, 513) of calculating  $p_{\rm H}$  vals. in relation to H<sub>2</sub>O contents of soil. A. G. P.

Effect of irrigation on soil texture. T. H. MEANS (Science, 1936, 84, 39–40).—A discussion. L. S. T. What are "soil acid" and "moor acid"? R. V. RODT (Chem.-Ztg., 1936, 60, 593).—In low-moor soils containing  $\text{FeS}_2$ , the  $\text{H}_2\text{SO}_4$  produced by atm. oxidation attacks concrete. In high-moor soils, injury to concrete is most marked when the hydrolytic acidity (NaOAc extract) is > 20 c.c. Dissolution of Ca from concrete is indicated. The possibility of injury cannot be ascertained merely by  $p_{\text{H}}$  determinations. A. G. P.

Alkali soils and their reclamation. I. N. R. DHAR and S. K. MUKERJI (Proc. Nat. Acad. Sci. India, 1936, 6, 136—148).—Reclamation of very alkaline soils  $(p_{\rm H} \ 10.8)$  by application of molasses depends on the neutralisation of soil alkali by the org. acids produced, replacement of soil Na by Ca from the molasses, and stimulation of N-fixation processes. A. G. P.

Application of the ter Meulen-Heslinga method to the determination of organic matter and "combined" water in soil. E. MORALES (Anal. Ffs. Qufm., 1935, 33, 942—946).—Employing the ter Meulen-Heslinga method it is possible to determine the H present as combined H<sub>2</sub>O separately from org. H. F. R. G.

Determination of nitrogen in soils. V. SUBRAH-MANYAN (Agric. Live-Stock India, 1936, 6, 284–288).— Recent developments of the  $H_2CrO_4$  oxidation process are discussed. A. G. P.

Physiological-chemical determination of the "plant-soluble" nitrogen of soils ("nitrogen number"). G. von DOBY (Z. Pflanz. Düng., 1936, 44, A, 265—282; cf. A., 1927, 79).—The "N no.," *i.e.*, invertase activity of plants without additional N supply + activity with optimum N supply, is utilised in determining the assimilable N in soil by means of pot cultures. A. G. P.

Determination of iron in humates. Use of iodohydroxyquinolinesulphonic acid. N. A. CLARK and D. H. SIELING (Ind. Eng. Chem. [Anal.], 1936, 8, 256—257).—A modification of Yoe's method (A., 1932, 587) is recommended. E. S. H.

Copper in soils and plants in copper-rich areas. S. PRAT and K. KOMAREK (Sborn. Masaryk. Akad. Práce, 1934, 8, No. 8, 1—16; Chem. Zentr., 1935, ii, 870).—Agrostis alba and Melandrium silvestre growing in Cu-mining areas, in soils containing 1—39% of Cu, have 0.2—3.25% of Cu in the ash. A. G. P.

Mineralogical examination of three Mecklenburg soils. C. W. CORRENS and F. K. SCHLÜNZ (Z. Pflanz. Düng., 1936, 44, A, 316—326).—The distribution of the principal soil minerals in the various mechanical fractions of the soils is examined. The degree of saturation with bases increases with the % of montmorillonite. Surface and sub-soils behave differently in this respect. Neubauer vals. for K increase with the quantity of K-bearing minerals in the finer soil fractions. A. G. P.

Soil extracts as nutrient solutions for higher plants. A. ÅSLANDER (Z. Pflanz. Düng., 1936, 44, A, 282—306).—Plants were able to neutralise acid soil extracts when these were used for H<sub>2</sub>O-cultures. Extracts of mineral and humus soils often exhibited growth-stimulating effects (unrelated to their  $p_{\rm H}$ ) which are associated with deposition of org. matter on the plant roots. There was no evidence of an actual intake of org. matter under these conditions. Extracts of sphagnum moss from peat soil were toxic to barley. The toxicity was somewhat decreased by neutralisation or by storing until moulds developed. The organisms probably decompose toxic substances. A. G. P.

Suitability of Mitscherlich pot-culture tests as a standard for the utility of other methods of soil examination. (A) H. NEUBAUER. (B) E. A. MITSCHER-LICH (Z. Pflanz. Düng., 1936, 44, A, 327-341, 349).--(A) In addition to readily available nutrients soil contains mineral matter which may be decomposed by the action of plant roots during growth. The extent to which nutrients in these minerals may be utilised  $\infty$  the total surface area of the roots. In the restricted condition of pot experiments the amount utilised is more nearly  $\infty$  the total amount of soil present. For this reason Mitscherlich tests are not comparable with field trials.

(B) A reply. The pot-culture method yields results based on the ratio of crop yields with and without added nutrient and is not concerned with the actual amount of nutrient taken up. A. G. P.

Nitrogen fixation and Azotobacter count after application of molasses and sugar to soil. I. N. R. DHAR and E. V. SESHACHARYULU (Proc. Nat. Acad. Sci. India, 1936, 6, 99—109).—Addition of molasses to soil effects a rapid increase in the nos. of Azotobacter. The amount of N fixed is not  $\alpha$  the increase in the nos. of organisms. Periods of rapid fixation of NH<sub>3</sub> are associated with a steady level in bacterial nos. Fixation of N in soil is not entirely a bacterial process. Both available and total N are increased by treatment of soil with molasses. A. G. P.

Peruvian guano. J. M. CANCINO (Bol. Soc. Quim-Peru, 1936, 2, 9—18).—The output of Peruvian guano and its composition and analysis are discussed. Adulteration is detected by determination of uric acid, potash, and  $H_2C_2O_4$ . Analyses of a genuine and a false sample are given. L. A. O'N.

Crude lignite as fertiliser. F. BERKNER (Z. Pflanz. Düng., 1936, 44, A, 346-348).—No evidence was obtained in manurial trials of the presence of plant hormones in lignite or in farmyard manure.

A. G. P.

Mixtures of blast-furnace slag with ammonium salts [as fertilisers]. W. UTSCH (Z. Pflanz. Düng., 1936, 44, A, 198–223).—The mixtures produced crop yields as high as those obtained with  $CaCO_3-NH_4$  salts, and exhibited greater neutralising power in soil. Nitrification was improved but to an extent < that produced by  $CaCO_3$ . A. G. P.

Modern manuring, crop quality, and health. L. SCHMITT (Angew. Chem., 1936, **32**, 601-603).— A lecture.

Relations between plant growth, soil, and the nutrient ratio of the fertiliser. III. K. RACKMANN (Z. Pflanz. Düng., 1936, 44, A, 307–309; cf. B., 1936, 292).—Nitrophoska affects the growth of cocksfoot to the same extent as an equiv. combination of individual fertilisers. A. G. P. Influence of deficiency of potassium on the light intake of plants. G. ROHDE (Z. Pflanz. Düng., 1936, 44, A, 247–255).—A discussion. The blue-green leaves of K-deficient plants utilise only red-yellow light.

the yield and health condition of crops.

A. G. P. Effect of micro-elements on plant growth. K. SCHARRER (Z. Pflanz. Düng., 1936, 44, A, 223–247).— A discussion of the influence of secondary nutrients on

Analysis of plant tissue. Application of a semimicro-Kjeldahl method. W. W. UMBREIT and V. S. BOND (Ind. Eng. Chem. [Anal.], 1936, 8, 276–278).— The sample, containing 0.5—5 mg. of N, is heated with 5 c.c. of a mixture of CuSO<sub>4</sub> 2 g., SeO<sub>2</sub> 2 g., Na<sub>2</sub>SO<sub>4</sub> 100 g., H<sub>2</sub>SO<sub>4</sub> 500 c.c., and H<sub>2</sub>O 500 c.c., basified, and the NH<sub>3</sub> removed into standard acid by aëration. In wet samples containing NO<sub>3</sub>', the latter is first reduced with Devarda's alloy; in dry samples, NO<sub>3</sub>' is "fixed" by H<sub>2</sub>SO<sub>4</sub>-salicylic acid. The method is rapid, accurate, and suitable for routine work on relatively small samples. S. C.

Growing healthy raspberries. Control of pests and diseases. R. V. HARRIS (East Malling Res. Sta. 23rd Ann. Rept., 1935, 232-242).—A summary.

A. G. P.

A. G. P.

Possible culture of mustard in Algeria. P. LAUMONT (Bull. Mat. Grasses, 1936, 20, 173—178).— The cultivation of mustard and its utilisation are discussed. The oils and protein contents of mustard reach max. at full flowering  $(7\cdot8 \text{ and } 20\cdot1\%)$ , respectively), and subsequently decline. The crude fibre content increases steadily throughout growth.

A. G. P.

Tree injection. J. HEARMAN, B. F. G. LEVY, and W. A. ROACH (East Malling Res. Sta. 23rd Ann. Rept., 1935, 137—141).—After injection of fertiliser materials into trees, leaf injury is more marked in mature than in immature leaves. In the latter, visible injury may not appear until 3 days after injection. A. G. P.

Leaf injection. W. A. ROACH (East Malling Res. Sta. 23rd Ann. Rept., 1935, 134—136).—Solutions of metallic salts may be injected into leaves by immersion of the cut tips in the solution. Injection of Fe<sup>III</sup> tartrate into a hole cut in a pear-tree stem effectively cured chlorosis. The use of injection methods in the diagnosis and correction of deficiency diseases is indicated. A. G. P.

Insect destruction in grain by ethylene oxidecarbon dioxide mixtures. Note on methyl bromide. A. LEPIGRE (Bull. Soc. d'Encour., 1936, 135, 385–462).— The technique of fumigation with  $(CH_2)_2O-CO_2$  is examined and effects on the quality of grain are considered. MeBr alters the flavour of fumigated tobacco, modifies the baking quality of cereals, and lowers the germinative capacity of certain seeds.

A. G. P.

Relation between quantity of ethylene chlorohydrin absorbed and growth response in treatments for shortening the rest period of potato tubers. L. P. MILLER and F. E. DENNY (Contr. Boyce Thompson Inst., 1936, 8, 121–136).—Effects of treatment with different amounts of  $CH_2Cl\cdot CH_2 \cdot OH$  (I) under varying conditions are examined. On the basis of the amounts of (I) absorbed by tubers (recovered by distillation) stimulative effects are produced by  $15 \cdot 0 - < 1 \cdot 0$  c.c. of  $0 \cdot 1M$ -(I) per 100 g. of tubers at 30° and  $25 \cdot 0 - < 1 \cdot 0$  c.c. at  $22-25^{\circ}$ . Part of the (I) absorbed by tubers is decomposed during the 5-day treatment. The permeability of tubers to (I) declines rapidly during the first 7-10 days after harvesting, and later much less rapidly. Methods of treating larger quantities of potatoes are indicated. A period of 4 days should elapse between treatment and replanting. A. G. P.

Codling moth (Cydia pomonella, L.). W. STEER (East Malling Res. Sta. 23rd Ann. Rept., 1935, 186— 190).—The timing of sprays is examined in relation to the period of larval activity. July application of Pb arsenate (I) gave good results on pears. Derris and nicotine were not effective. Addition of (I) to the petal-fall scab spray where eye entry is prevalent, and also to the second post-blossom spray (4—5 weeks after petal fall), is recommended. A. G. P.

Impregnation of tree-banding materials. II. Apple-blossom weevil and codling-moth experiments, 1935. R. M. GREENSLADE and A. M. MASSEE (East Malling Res. Sta. 23rd Ann. Rept., 1935, 177— 183).—Previous work (B., 1936, 37) is confirmed. Use of bands treated with  $\beta$ -C<sub>10</sub>H<sub>7</sub>-OH gave promising results against codling moth, but not against the weevil. Org. thiocyanates damaged tree stems.

A. G. P.

Control of flea-beetles by means of seed dressing. C. L. WALTON (Ann. Rept. Agric. Hort. Res. Sta., Long Ashton, 1935, 80—86).—Flea-beetle attack on *Brassicæ* was prevented or greatly reduced by treatment of seed with a solution containing kerosene 1 gal.,  $p-C_6H_4Cl_2$ 4 lb.,  $C_{10}H_8$  1 lb., using 0.5-1.0 fl. oz. per lb. of seed. A. G. P.

Control of woolly aphid (*Eriosoma lanigerum*, Hausm.) on nursery trees. H. G. H. KEARNS and E. UMPLEBY (Ann. Rept. Agric. Hort. Res. Sta., Long Ashton, 1935, 67—75).—Immersion of nursery stock in 10% tar-oil emulsion did not give complete control. Immersion in hot  $H_2O$  (43·3°) for 30 min. was effective. Paraffin oil injured stems and shoots of cider apple trees. Infestation was restricted by summer spraying with white oil (1%)-nicotine at 10-day intervals.

A. G. P. **Treatment of dormant nursery stock against** woolly aphis. R. M. GREENSLADE (East Malling Res. Sta. 23rd Ann. Rept., 1935, 184—185).—No appreciable injury resulted from dipping in tar-oil washes (10—25%) or from fumigation with HCN (4—32 oz. of NaCN per 1000 cu. ft.). A. G. P.

Action of fungus spores on Bordeaux mixture. S. E. A. McCALLAN and F. WILCOXON (Contr. Boyce Thompson Inst., 1936, 8, 151-165).—The sensitivity of various spores to dried Bordeaux mixture is closely related to the amount of dry matter in aq. extracts of spores and to the amount of Cu which these extracts bring into solution from the dried mixture. The active material in the extracts probably consists of malic and  $\mathrm{NH}_2$ -acids. Na malate, aspartic acid, and glycine dissolve considerable amounts of Cu from the mixture, forming Cu complexes which exhibit the same toxicity to spores as does CuSO<sub>4</sub>. A. G. P.

Use of derris root as an insecticide. W. STEER (East Malling Res. Sta. 23rd Ann. Rept., 1935, 225— 227).—A summary of trials against various insect pests. A. G. P.

[Insect] egg-killing washes. Ovicidal properties of lauryl rhodanate. H. G. H. KEARNS and H. MARTIN (Ann. Rept. Agric. Hort. Res. Sta., Long Ashton, 1935, 49–57).—The ovicidal efficiency of lauryl thiocyanate (I) in 0.4% sprays on *Anuraphis pomi* is > that of 2% neutral tar-oil preps. and is unaffected by dissolution in petroleum oils and emulsification with Agral S.R. or Na oleate. Similar concns. of (I) were effective against *Psyllia mali*. A. G. P.

Combined [insecticidal] washes. II. Hydrocarbon oils in combination with lime-sulphur. III. Derris extracts in combination with limesulphur. H. G. H. KEARNS, R. W. MARSH, and H. MARTIN (Ann. Rept. Agric. Hort. Res. Sta., Long Ashton, 1935, 37-42, 42-48; cf. B., 1935, 822).-II. Comparative trials with CaO-S-sulphite lyepetroleum oil preps. on black-currant (oil 5%, S 8%) and apple (oil 5%, S 3%) against capsid bug are recorded. Low-grade oil (74% unsulphonatable) caused considerable injury on black-currant. High-grade (98%) oil had no ill-effect. Promising results were obtained. The importance of correct timing of the application is indicated.

III. Rotenone or derris root was as effective as nicotine (I) in CaO-S combinations for sawfly control. Derris and (I) act as stomach poisons when applied up to 8 days before egg-hatching. (I) had no apparent ovicidal action. A. G. P.

Comparative insecticidal efficiency against camphor scale of spray oils with different unsulphonatable residues. A. W. CRESSMAN and L. H. DAWSEY (J. Agric. Res., 1936, 52, 865—878).— Petroleum oils containing 6, 16, and 33 vol.-% respectively of sulphonatable matter (I) are compared. With Na oleate emulsions giving equal oil deposits no differences in scale mortality could be attributed to differences in (I). The amount of oil deposited was independent of (I) but  $\propto$  the oil content of the spray. In field trials with 2% emulsions the oil containing 33% of (I) caused much the heaviest leaf fall. A. G. P.

Field-spraying trial of combined fungicidecontact insecticide sprays, 1935. M. H. MOORE and H. B. S. MONTGOMERY (East Malling Res. Sta. 23rd Ann. Rept., 1935, 191-197).—Improved control of scab was obtained by inclusion in the spray programme of 2 pre-blossom applications of CaO-S with Pb arsenate and petroleum oil-sulphite lye emulsion. Such combinations cause leaf injury and defoliation if given at greenor pink-bud stages. The fungicidal and acaricidal efficiency of CaO-S was not affected by admixture with oil emulsions or with wetting agents. A. G. P.

Fungicidal and phytocidal properties of certain new chemical preparations. H. B. S. MONTGOMERY,

M. H. MOORE, and H. SHAW (East Malling Res. Sta. 23rd Ann. Rept., 1935, 198—203).—Trials with Shirlan, tetramethylthiuram disulphide, dyestuffs, Cu and Zn preps., 8-hydroxyquinoline, chloronaphthol-dekalin emulsion, and petroleum emulsions are described. None was as effective as Bordeaux mixture or CaO-S against scab. A. G. P.

Ammophos.—See VII. Corrosion of pipe-lines. See X. Groundnuts.—See XII.

#### PATENTS.

Production of [fertiliser containing] available phosphoric acid and less common elements. F. W. GENZ, ASST. to VIRGINIA-CAROLINA CHEM. CORP. (U.S.P. 2,005,617, 18,6.35. Appl., 12.9.32).—Fertilisers containing Cu, Mn, Mg, or Zn are manufactured by treating a mixture of phosphate rock and approx. 3.58 wt.-% of the appropriate carbonate with H<sub>2</sub>SO<sub>4</sub>. L. C. M.

Insecticide composition. W. MOORE, Assr. to AMER. CYANAMID CO. (U.S.P. 2,005,797, 25.6.35. Appl., 15.8.31).—Compounds of the type Ph·R, where R is aryl or aralkyl of formula  $C_nH_{2n-7}$ , e.g., Ph<sub>2</sub>,  $CH_2Ph_2$ , (· $CH_2Ph)_2$ , are used, in volatile org. solvents, as insecticides. A. W. B.

**Contact insecticide.** E. W. BOUSQUET, ASST. to GRASSELLI CHEM. Co. (U.S.P. 2,006,227, 25.6.35. Appl., 19.10.32).—Technical soya-bean lecithin, dispersed, at concns. of 0.1-0.2% in H<sub>2</sub>O, with the aid of BuOH, Na oleate, or sulphonated fish oil, is used. Other insecticides, *e.g.*, nicotine, pyrethrum extracts, may also be added.

A. W. B.

Media for combating insect pests. BRABENDER G.M.B.H., and W. BARTHELS (B.P. 449,210, 26.6.35).— The toxicity of  $C_2H_4Br_2$  to insects is increased by  $C_2HCl_3$ , probably owing to increased volatility.  $CCl_4$  and an irritating agent, *e.g.*,  $ClCO_2Me$ , may be added to such insecticidal mixtures. R. S. C.

Manufacture of insecticide and fungicide. M. T. INMAN, Assr. to C. P. BYRNES (U.S.P. 2,011,765, 20.8.35. Appl., 5.12.29).—Nicotine (I) or pyrethrum is combined with Penetrol to give a liquid or solid insecticide. The efficiency of (I) is increased thereby. R. S. C.

Manufacture of parasiticides and insecticides. W. H. VOLCK, Assr. to CALIFORNIA SPRAY-CHEM. CORP. (U.S.P. 2,012,328, 27.8.35. Appl., 23.9.32).—Oil sprays containing an amount of  $NH_4$  soap sufficient to neutralise the hardness of the  $H_2O$  and give about 0.25 lb. of excess soap per 100 gals. of wash have increased insecticidal and fungicidal power. R. S. C.

## XVII.—SUGARS; STARCHES; GUMS.

Influence of plant spacing on yield and chemical composition of sugar beet. LINDNER (Zentr. Zuckerind., 1935, 43, 214—216; Chem. Zentr., 1935, ii, 935).—Increase of spacing of beets lowers the quality and ease of working up, the sugar content and purity quotient falling and the proportion of melassigenic constituents rising. H. N. R.

Beet juice evaporation. Effect of sodium aluminate. W. L. FAITH and C. H. SARTORIUS (Ind.

Eng. Chem., 1936, 28, 872–874).—Thin beet juices from Danek filters were treated with  $Na_2Al_2O_4$  in varying amounts and were then conc. in an experimental singleeffect evaporator. An approx, linear relationship was indicated between the increase in purity of the juice and the amount of  $Na_2Al_2O_4$  used, whilst the treatment appeared to aid the pptn. of colloidal materials, particularly with Steffens house juice ; such variations as were found were probably dependent on the types of colloid present. Treatment of juice from Steffens house run (hard  $H_2O$ ) caused a decrease in scale formation. I. A. P.

Apparatus for pre-defecation [of sugar juice]. J. EISNER (Deuts. Zuckerind., 1935, 60, 263–264, 279–281; Chem. Zentr., 1935, ii, 935).—An apparatus for adding CaO to the crude juice in such amount as to maintain the optimum  $p_{\rm H}$  is described. H. N. R.

Change of colour of thick-juice in working to molasses. (A) E. TROJE. (B) R. MEHRLE (Deuts. Zuckerind., 1935, 60, 101, 123; Chem. Zentr., 1935, ii, 936).—(A) The factors influencing discoloration are discussed. Heating of slightly alkaline or neutral sucrose solutions causes hydrolysis.

(B) The inverting action of alkali has no effect on the final colour of the syrup. H. N. R.

Lead index of maple and cane sugar. P. RIOU and J. DELORME (Compt. rend., 1936, 202, 1941-1942).-Sucrose (I) usually has a Pb index which falls within the normal limits for maple sugar (II) (cf. B., 1935, 1015); hence this quantity is no criterion of the absence of (I) in a sample of (II). J. L. D.

Detection of sucrose in vegetable material. K. TÄUFEL, H. THALER, and G. KOPP (Z. Unters. Lebensm., 1936, 71, 390–393).—Sucrose (I) is converted by exhaustive acetylation into its octa-acetate, which is insol. in  $H_2O$  and is identified by determination of the Ac val. and by reconversion into (I). The detection of (I) in coffee and malt is described.

E. C. S. [Crystal] development and production of anhydrous glucose. W. B. NEWKIRK (Ind. Eng. Chem., 1936, 28, 760—766).—An illustrated account of the crystal development of anhyd. glucose (I) and the hydrate under various conditions and of the large-scale commercial production of (I) containing < 0.05% H<sub>2</sub>0 and < 0.001% of other impurities. R. S. C.

N fixation.—See XVI. Utilising cellulose materials.—See XVIII. Hortvet thermometers. Wood-sugar for pigs.—See XIX.

## XVIII.—FERMENTATION INDUSTRIES.

Naked barley as brewing material. E. M. HESS (Brauer- u. Hopfen-Ztg., 1935, 62, 42-44; Chem. Zentr., 1935, ii, 772).—With proper working no difficulties arise in malting; naked barley is, however, unsuitable for use with unmalted grain. H. N. R.

Influence of degree of ripeness on the resting period of brewing barleys. K. G. SCHULZ and G. KUNISCH (Woch. Brau., 1936, 53, 233-238).—The resting period (P) (time between cutting and stage attaining 95% germination in tests of 3 days' duration) is largely influenced by the seasonal conditions during the formation and ripening of the grain; conditions accelerating ripening also shorten P, this effect being most marked with varieties with normally long P. Advancing ripeness at cutting diminishes P to different extents for different varieties. For successive cuttings of the same barley the decrease in P is  $\triangleleft$  or > the interval between cuttings. Thus, in 1935, samples of Heines Hanna and Hadostreng reached "germ-ripeness" (end of P) at the same time (in each variety) whether cut at the yellow or dead-ripe stage; early cutting of Isaria, however, retarded maturation.

I. A. P.

Chemistry of hops. K. MAYER (Tages-Ztg. Brau., 1935, 33, 293; Chem. Zentr., 1935, ii, 937).—It is not considered possible quantitatively to determine the  $\beta$ -acidic portion of hop acids. H. N. R.

Antiseptic properties of hops. H. ANTELMANN (Woch. Brau., 1936, 53, 185—186, 198—200, 203—207). —The recognition of the bitter principles (P) of hops as the source of hop antiseptic val. (A) is traced, the literature of the methods of measurement of A comprehensively reviewed, and the effects of varying conces. of P on different organisms are noted. The changes in P and A during ripening and drying and during the brewing process are discussed from the literature, and the relation between beer stability and the A of the hops is shown. I. A. P.

Lining concrete fermenting vessels. E. M. DowNES (J. Inst. Brew., 1936, 42, 376–377).—The method of applying an Ebon lining to such vessels is described; good results have been obtained thereby in brewery practice. I. A. P.

Tests on "Diskus" laminated wort-coolers. G. W. A. BRISCHKE (Woch. Brau., 1936, 53, 209–211, 219–224, 227–232).—The "Diskus" enclosed wortrefrigerator consists of horizontal grooved metal plates separated by thin sheets of metal, and the wort is forced through the apparatus under pressure without aëration. It is especially suited to hot-filtered worts, and with yeasts acclimatised to worts poor in  $O_2$  yields beer of very high quality. I. A. P.

Turbidity in wort and beer. I. Measurement of turbidity. C. ENDERS (Woch. Brau., 1936, 53, 201-203, 211—216).—A method for measuring turbidity (T) in wort and beer has been evolved, using the Lange Electrocolorimeter, the results being expressed in terms of mastic (colloidal T) or SiO<sub>2</sub> gel (suspensions). The colour is first determined (Brand scale) with the aid of the opalisator, after which the total absorption is determined against H<sub>2</sub>O. The concn. of mastic corresponding to the T may then be found from the curves and table presented. It was not possible to decide which factor (e.g., air, light) was of special significance for the clouding of Congress wort on keeping, but the effect of Fe was clearly shown. The development of Tin infected wort was initially associated with a slight decrease in acidity; thereafter acidity and T both increased, but the latter at a greater rate than was produced by the corresponding acidity increases alone. I. A. P.

Objective determination of colour in worts and beers. P. KRUMHOLZ, S. PICKHOLZ, and H. HAMBURG (Brau- u. Malzind., 1936, No. 7; Woch. Brau., 1936, 53, 255).—Using strongly filtered blue light, the interference of weak turbidities in photoelectric measurements can be minimised and the colour-sensitivity increased. Strong turbidities can be largely shut out by measuring the absorption in deep-red light; absorption in red light serves as a measure of wort turbidity. A process and an apparatus for the application of these facts to colour determination in wort and beer are indicated. I. A. P.

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Presence of pectin in first wort, in barley, and in malt. F. JUST (Woch. Brau., 1936, 53, 225-227).--Examination of first wort for pectin gave negative results, and direct examination of barley and malt for pectin likewise failed. Preps. from the husk of barley or malt proved to yield small amounts of galacturonic acid on hydrolysis, but this is probably derived from polyuronides rather than from pectin.' Hence, the first entry of true pectin into brewers' wort takes place during hop-boiling. I. A. P.

Possible final attenuation of beer wort and its determination. E. SCHILD and O. GEIGER (Woch. Brau., 1936, 53, 241—245).—The wort concn. is adjusted where necessary, before fermentation, to between 10% and 4% by dilution or evaporation. 10% worts require 48—72 hr. and 4—10% worts 72 hr. fermentation at 25° in presence of 0.5% of pressed yeast and 0.35% of Biospan. The extract is determined after filtration, vol.-% being used for calculation, though wt.-% is sufficiently accurate for comparative measurements. The reasons for obtaining different final attenuations under different conditions of fermentation are discussed. I. A. P.

Non-biological turbidities in beers. E. HELM (J. Inst. Brew., 1936, 42, 379-393).-The nature and classification of beer hazes are discussed and the measurement of chill and permanent ("oxidation") hazes is described. Chill haze is increased by storage (especially at raised temp.), its formation is accelerated by shaking, and when beer is drawn under air it is > when drawn under  $CO_2$ ; chill haze is decreased by secondary fermentation and by addition of tannin or proteases. The chief cause of permanent protein haze in beer is dissolved O<sub>2</sub>; increase in this oxidation haze is accelerated by shaking and is little influenced by the pasteurisation period. Foaming during bottling decreases this haze, due to expulsion of some  $O_2$ ; the haze is also decreased by addition of tannin, e.g., before filtration, or of pepsin, but the effect of papain is dependent on the amount of the enzyme added. I. A. P.

Preparation of finings [for beer]. A. C. REAVENALL (J. Inst. Brew., 1936, 42, 373–374).— During manufacture or storage, isinglass finings should not be allowed to attain temp. > those considered safe for beer, since decrease in  $\eta$  results at elevated temp.  $(21 \cdot 1-26 \cdot 7^{\circ})$  and normal  $\eta$  and fining power may not be re-attained on returning to, and storage at, e.g., 10°. A method is described for the prep. of finings from shredded isinglass in 18—20 hr. I. A. P.

Constituents of wine. Glycerol and lactic acid. L. ESPIL (Bull. Soc. chim., 1936, [v], 3, 1334–1338).—

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Glycerol is determined by means of  $HIO_4$ , and lactic acid by oxidation with  $KMnO_4$  followed by a determination of MeCHO with dimedon in wine freed from other reducing substances. J. L. D.

Stabilisation of wines in barrels. L. GRANDCHAMP (Rev. Viticult., 1935, 82, 251-255; Chem. Zentr., 1935, ii, 937).—A method using KHSO<sub>3</sub> is described. H. N. R.

Employment of ferricyanides and ferrocyanides in wine clarification. E. Powys (Bol. Soc. Quim. Peru, 1936, 2, 19–24).—Preps. based on ferricyanides and ferrocyanides are effective only when the turbidity is due to excess of Fe salts in the wine. Detection of wines which have been treated with such preps. is discussed. A typical prep. was found to consist of two solutions, one of  $ZnSO_4$  and the other of  $K_3Fe(CN)_6$ , to be added to the wine, one after the other.

L. A. O'N.

Fruit products. V. Concentration of fruit juices by freezing, with special reference to apple juice. VI. Fruit syrups. (A) Production; (B) Use of pure fruit syrups in milk beverages. VII. Production of fruit squashes. VIII. Production of fruit wines. (A) Effects of dilution of juice and addition of press cake. (B) Effects of type of sugar and of yeast used in fermentation. (C) Yeast nutrition in fruit wines. V. L. S. CHARLEY (Ann. Rept. Agric. Hort. Res. Sta., Long Ashton, 1935, 150-161, 162-183, 184-191, 192-216; cf. B., 1935, 824).-V. Apple juice is conc. by freezing and removal of ice, to yield a product containing 45-55% of sugar. The flavour is superior to that of vac.-conc. juice. Similar treatment of cider may double the normal EtOH content of the product.

VI. Methods of prep. of syrups from various fruits are examined. Fruit syrups to which no acid has been added may be mixed with milk in the proportion 1:5 without risk of curdling. The  $p_{\rm H}$  of the mixture is approx. 5.0. Syrups containing 45—55% of sugar are preferred for this purpose.

VII. A progress report.

VIII. The prep. of wine from strawberry, loganberry, and raspberry is described. Analyses are recorded. Use of  $NH_4$  phosphate as a yeast nutrient extended, but Ca and Mg checked, fermentation. A. G. P.

Influence of methyl alcohol in the determination of higher alcohols in spirits. J. M. CLAVERA and F. MORENO MARTÍN (Anal. Fis. Quím., 1936, 34, 507— 512).—A determination is made of the correction given in graphical form which must be applied to the vals. obtained by Rose's method for the determination of amyl alcohol in aq. solution, in presence of known amounts of MeOH. F. R. G.

Vinegar from dates. B. DAS and J. L. SARIN (Ind. Eng. Chem., 1936, 28, 814).—Dates are boiled 3 times with equal wts. of  $H_2O$ , and the combined expressed juices, after inversion with HCl and addition of nutrient salts, are submitted successively to alcoholic and acetic fermentation. The product is clarified by sedimentation and filtration and is sterilised at  $65 \cdot 5^{\circ}$ . 100 lb. of fresh fruit yield 12—15 gals. of standard vinegar. I. A. P. Fermentative utilisation of cellulosic materials, especially pentosans. E. I. FULMER (Ind. Eng. Chem., 1936, 28, 778—782).—The fermentative productions of fuel gas, AcOH, and EtOH from cellulosic wastes and of EtOH from wood sugar and sulphite liquors are briefly discussed. The production of furfuraldehyde from oat hulls or their hydrolysate is outlined, and the results of fermentation of xylose, other carbohydrates, and maize meal are compared, the method of cultivation of the organism to ensure rapid fermentation and improved solvent yield being described. I. A. P.

Source of thermal energy in alcoholic fermentation. M. A. M. Jané (Quim. e Ind., 1934, 11, 164; Chem. Zentr., 1935, ii, 936-937).—The significance of the difference between the heats of combustion and fermentation of sugar is discussed.

H. N. R. Paints for brewery.—See XIII. Yeast as nutriment. Determining  $H_2O$ , and N [in malt]. Kephir.—See XIX. Distillery wastes.—See XXIII.

## XIX.—FOODS.

Irradiation of wheat with ultra-short waves. P. P. TARUTIN (Mühlenlab., 1936, 6, 81-87).-The rise of temp. obtained increases with the current used and is a max. at  $\lambda$  5 m.; this is also the most effective  $\lambda$ for the destruction of pests. High initial moisture content decreases both the temp. rise and the energy absorption. The first few sec. of irradiation are the most important. The germinating power of the grain is stimulated below 65° but reduced at higher temp. The endosperm is dried, swollen, and loosened by the evolution of steam, the effect depending on the nature of the grain; the chemical constitution is unaffected, but the gliadin and leucosin are partly coagulated and the diastatic activity is reduced. The baking quality is unaffected below 65° but reduced at higher temp. Irradiation is useful for destroying pests, especially in the resting stage, for conditioning, and for the manufacture of cornflakes etc. E. A. F.

Biochemical influence on wheat of ultra-short waves. P. P. TARUTIN (Mühlenlab., 1936, 6, 103– 110).—Exposure to waves of  $\lambda 4.5$ —6.5 m. improves milling properties and does not affect germination, gluten quality, or gassing power if the temp. is  $\geq 65^{\circ}$ . Prolonged exposure (as, *e.g.*, in treating grain of high H<sub>2</sub>O content) causes loss of germinating power at lower temp., and should be avoided by using high power. Treatment at high temp. does not appear to affect the composition of the grain, but destroys the amylase activity and coagulates the gluten. Very strong heating by ultra-short waves can be used for H<sub>2</sub>O and ash determinations. E. A. F.

Influence of low temperatures on damp grain. N. P. KOSMIN and M. S. ROMANOV (Mühlenlab., 1936, 6, 97–104).—Storage of damp wheat at various tempfrom  $-10^{\circ}$  to  $-41^{\circ}$  does not affect the gluten content, gluten quality, or diastatic activity. Germinating power is destroyed by freezing if the H<sub>2</sub>O content is > about 12%. E. A. F. Nitrogen content of single barley grains. N. NELSEN (Compt. rend. Trav. Lab. Carlsberg, 1936, 21, 247-269).—The N content of a no. of single grains of 6 varieties of 2-rowed barley was determined. Differences in the % N content were  $\lt$  16% for grains from the same ear, usually 20-40%. Between grains from different ears the variation was much greater. No uniformity in the variations was apparent, although vals. tended to be higher towards the top of the ear. With one exception, no difference was observed between the two sides of the ear. Locality and conditions of growth affected the N content. The abs. N content varied quite as widely as the %. The total dry matter content was substantially const. for grains of the same variety. E. A. F.

Rapid determination of nitrogen with selenium as accelerator. F. M. WIENINGER (Woch. Brau., 1936, 53, 251—252).—In N determinations using Se mixture, identical results and reaction times are obtained whether or not the mixture contains  $HgSO_4$ . Further, the recommended mixture of  $Na_2SO_4$ ,  $CuSO_4$ , and Se is more readily sol., is cheaper and non-poisonous, and permits the use of ordinary aq. NaOH for the NH<sub>3</sub> distillation. The application of the method to the determination of protein in barley and malt is described, the decomp. time for 1.75 g. being approx. 16—18 min. ; worts and beers require a somewhat longer time. I. A. P.

Effect of fermentation on some properties of gluten. G. H. GUEST and R. K. LARMOUR (Canad. J. Res., 1936, 14, C, 222—228).—Fermentation causes a loss of protein of about 17%, except in a high-protein and a low-protein flour, which show respectively a much lower and a much higher loss. These two flours also show a change in  $H_2$ O-holding capacity (increase and decrease respectively). There is practically no differentiation between flours from heavily frosted wheat and average sound flours. It is concluded that these two properties of gluten cannot be used to differentiate most flours. E. A. F.

Effects of humidity and high temperature on the [free] amino-content of rice. N. M. BASU and S. R. MAITRA (J. Indian Chem. Soc., 1936, 13, 254—259). —Three samples of milled rice, kept at 33° in a moist atm., first increased and then decreased in NH<sub>2</sub>-content (determined by a modified Van Slyke method), owing probably to liberation first of NH<sub>2</sub>-acids and then of NH<sub>3</sub>. A fourth sample, somewhat disintegrated, showed a slight initial fall, probably owing to previous formation of NH<sub>3</sub>. Dhenki-milled rice is slightly more resistant both to this change and to growth of organisms than is milled rice. The production of NH<sub>2</sub>-acid may be connected with the causation of dropsy by badly stored rice. R. S. C.

Rapid determination of water in cereals at high temperature in the Ulsch drying oven. F. M. WIENINGER (Woch. Brau., 1936, 53, 249—251).—A temp. of 105° in the Ulsch oven is insufficient to remove the whole of the moisture from barley or malt; at 110° (0.52 atm.) all the moisture is driven off, whilst the loss of barley fat is negligible. The somewhat greater loss in wt. obtained by heating barley or malt for 2 hr. at 120° in the electric oven is largely attributable to changes in the fat. For moisture determination in barley and malt, therefore, use of the Ulsch oven is recommended, 2—3 g. being heated at 110° for 35 min. I. A. P.

Evaluation of German bread cereals. H. KÜHL (Landw. Versuchs-Stat., 1936, 126, 307—318).—A discussion of milling practice in relation to the nutrient val. of the product. A. G. P.

Soya bean. W. L. BURLISON (Ind. Eng. Chem., 1936, 28, 772—777).—The development of the cultivation and commercial utilisation of the soya bean in the United States is noted. The compositions of the bean, oil, and oil-free meal are indicated, and processing methods are described. Commodities in which soya bean or its products are at present utilised are listed and in part discussed, including food and feeding products, soaps, paints and varnishes, linoleums, printing inks, plastics, adhesives, etc. I. A. P.

Soya bean as a food product and industrial raw material. H. W. LOHSE (Canad. Chem. Met., 1936, 20, 224-225).—The nutritive val. of the meal and the industrial uses of the oil, protein, and lecithin are discussed. E. C. S.

Determination of milk solids in bread. C. HOFF-MAN, T. R. SCHWEITZER, and G. DALBY (Ind. Eng. Chem. [Anal.], 1936, 8, 298).—Lactose and butter fat, using the Reichert-Meissl val., are determined by standard methods. E. S. H.

Value of rate of reaction curves for baking powder. R. M. LADD (Canad. Chem. Met., 1936, 20, 226).—Owing to differences in the rate of loss of  $CO_2$ during mixing of the dough, baking powders cannot be rated on their available  $CO_2$  alone, but this loss must be ascertained and allowed for. E. C. S.

Dry beer yeast as nutriment. S. VINOKUROV, M. KUZNETZOVA, and K. KOVTUN (Ukrain. Biochem. J., 1932, 5, 113—124).—The influence of the temp. of drying on the digestibility of the proteins and on the vitamin content is investigated. W. O. K.

Determination of carbon dioxide in biological fluids, more particularly milk and cream. F. H. McDowALL (Analyst, 1936, 61, 472-473).—Hepburn's method (B., 1927, 42) of absorbing the  $CO_2$  liberated at low pressure in Ba(OH)<sub>2</sub> solution is applicable to biological liquids if a protein precipitant (phosphotungstic, silicotungstic, or phosphomolybdic acid; 10 pts. in 100 pts. of 5% H<sub>2</sub>SO<sub>4</sub>) is added to the fluid. An improved arrangement of the apparatus is described. J. W. S.

Vitamin-C content of milk powders. L. RENNER (Z. Kinderheilk., 1935, 57, 414-418; Chem. Zentr., 1935, ii, 3258).—The -C content of milk dried in N<sub>2</sub> or in air is the same as that of milk dried by the cylinder or spray method. Churning destroys about 50% of the -C. Addition of lemon juice or lactic acid does not affect the -C content. -C in milk exposed in open cans diminishes slowly. R. N. C.

Standardisation of Hortvet thermometers. J. R. STUBBS and G. D. ELSDON (Analyst, 1936, 61, 455-458). --Standard f.-p. depressions of sucrose solutions intermediate between the original standards (7% and 10%) are calc. to permit closer calibration over the range required for use with milk. J. W. S.

Corrosion of milk tins in ocean transit. W. F. SEYER and R. J. DONALD (Canad. Chem. Met., 1936, 20, 227—228).—External corrosion was rapidly induced by spraying with 4% aq. NaCl and transferring at frequent intervals from a warm to a cool moist atm. and conversely. Corrosion was prevented by packing the tins in cartons built with an asphalt layer and fully sealed at the corners and ends. E. C. S.

**Preparation of kephir with pure cultures.** S. PARASCHTSCHUK (Milch. Zentr., 1936, 65, 233-236).— In order to produce a max. concn. of desirable constituents (lactic acid, EtOH, and vitamin-B and -C) and a min. of undesirable (AcOH and protein hydrolytic products), the fermentation characteristics of a no. of yeasts and of mixtures of yeasts and *Bac. aceti* were determined. Typical milk-fermenting yeasts in pure culture produced a more satisfactory kephir than the customary mixture of organisms. A yeast from sour cream gave the greatest yield of -B and -C. E. C. S.

Technique of Van Oijen's (modified) test [for bacterial content of milk]. H. BARKWORTH (J. Southeast. Agric. Coll. Wye, 1936, No. 38, 143-149).--Appropriate apparatus and procedure are described.

A. G. P.

Use of ammonium sulphate serum of milk in serological investigations. H. KLUGE (Z. Unters. Lebensm., 1936, 71, 405-410).—By means of the precipitin reaction, using the  $(NH_4)_2SO_4$  serum, it is possible to detect adulteration of human milk with  $\bigstar$  10% of cows' milk, but it is not possible so to distinguish cows' from goats' milk. The reaction is given by milk heated to 85° but not by boiled milk. The agglutinin reaction can be used to detect infection by *Bac. abortus*, Bang, both in fresh milk and in milk preserved with CH<sub>2</sub>O. Typhus and paratyphus agglutinins can also be detected. E. C. S.

Application of the catalase test to butter. G. CRUESS-CALLAGHAN (Sci. Proc. Roy. Dublin Soc., 1936, 21, 253—255).—Correlation coeffs., determined by previous workers, between the catalase (I) figure and the quality of butter, and between (I) figure and its keeping property, are analysed statistically. The val. -0.4 is suggested for either coeff. J. L. D.

Vitamin-A assay of ghee. III. Storage of ghee and vitamins. B. N. BANERJEE (Agric. Live-Stock India, 1936, 6, 274–283).—The vitamin-A content (blue val.) of ghee remains substantially const. for approx. 1 month and subsequently declines. Loss of blue val. with time may occur through formation of colour inhibitors and through the more rapid disappearance of the colour. "Vitameter" vals. are not very trustworthy. A. G. P.

Formation of indole during decomposition of meat. I. A. SMORODINCEV and B. S. DISKINA (Ann. Chim. Analyt., 1936, [iii], 18, 174–176).—Indole is not detectable until meat is definitely spoiled. In the initial stages of decomp. true phenols are not present. Commencing decomp. is indicated by an increase in volatile fatty acids. E. C. S.

Vitamin content of sauerkraut. D. ZUVERKALOV and P. NUSHDIN (Ukrain. Biochem. J., 1932, 5, 125—128). —Men on a diet containing sauerkraut, but otherwise lacking in vitamin-*C*, do not develop scurvy. The highest content of -*C* is in the juice. W. O. K.

Etiology and control of blue-mould decay of apples caused by Penicillium expansum, Link. K. F. BAKER (Res. Stud. State Coll. Washington, 1935, 3, 32—33).—Factors affecting the incidence of decay are examined. Equipment should be sprayed with NaOCl (0.4% available Cl). CuSO<sub>4</sub> is not effective. The proportion of decayed fruit is lowered by rinsing for 1 min. in NaOCl following the usual washing operations. Chemically treated wrappings gave inadequate protection. A. G. P.

(A) Physiological changes in the rind of California oranges during growth and storage. (B) Field and storage studies in the composition of the rind of marsh grapefruit in California. E. M. HARVEY and G. L. RYGG (J. Agric. Res., 1936, 52, 723—746, 747— 787).—(A) Variations in H<sub>2</sub>O, sol. solids, sugar, hydrolysable polysaccharide, N, and hesperidin contents and  $p_{\rm H}$ of fruit with growth and environmental conditions are examined in a no. of species from different localities. With navel oranges late picked fruit is the more susceptible to brown stain. Storage at 0° increases susceptibility > does that at 5.5° or 11°. Pitting and spotting in storage are influenced by weather conditions during the last few days prior to picking.

(B) A similar examination of grapefruit is described. Narengenin (I) is much more toxic to rind tissue than is narengin (II). During storage metabolic disturbances which might retard the synthesis of (II) from (I) probably cause injury to rind. A. G. P.

Vitamin content of Greek fruit. I. Figs. II. Carob beans. III. Sultanas. IV. Grapes. M. SCHIEBLICH and V. VLASSOPOULOS (Z. Unters. Lebensm., 1936, 71, 415—420, 420—422, 423—425, 425—428).— I. Figs contained  $2 \cdot 4$  units per g. of vitamin-A, 0.48unit of  $-B_1$ , and 0.4—0.5 unit of  $-B_2$ . -C and -D were not detected.

II. The beans contained no appreciable amount of any vitamin.

III. Sultanas form a rich source of -B, particularly of  $-B_1$ . -A was present in traces; -C and -D were not detected.

IV. Grapes showed marked antiscorbutic activity. Most of the vitamin was found in the juice after pressing. E. C. S.

Characterisation of pectin extracts. H. ECKART (Z. Unters. Lebensm., 1936, 71, 428-434).—The d, n,  $p_{\rm H}$ , acidity, pectin content (as Ca pectate), tensile strength, and  $\eta$  of a no. of commercial preps. are tabulated. Determination of  $\eta$  by Höppler's method is recommended as the best means of characterising the extracts. E. C. S.

Tea adulteration in India. Tea substitutes. A. STEINMANN (Z. Unters. Lebensm., 1936, 71, 446—448). —The common adulterants of tea are reviewed and the characters of *Stachytarpheta jamaicensis*, which is so used in Java, are described. E. C. S.

Tannin complex in coffee. II. C. TIEDCKE (Z. Unters. Lebensm., 1936, 71, 393-404; cf. B., 1936, 713).—CO<sub>2</sub> is evolved from the K salts of chlorogenic (I), quinic (II), and caffeic acids (III) when treated with  $H_2O$  vapour under pressure as from the acids themselves, but the rate of evolution is not a linear function of time and temp. Dihydroxystyrene (IV) is formed when (I), (III), their K salts, or K caffeine chlorogenate is so treated. (IV) was identified by comparison with a sample prepared by an improved method, which is described, and is shown to exist mainly in a polymerised form. (II) and (III) are formed as intermediates in the decomp. of (I) or of its K salt during the Lendrich process. E. C. S.

Hydrolysis of inulin in the juices of chicory and Jerusalem artichoke in presence of acids. P. K. BOBKOV (Z. Spiritusind., 1936, 59, 239–240, 247–248, 257–258).—The titratable acidity (T) of either juice to which mineral acid has been added is < the sum of that of the juice itself and that of the added acid. The original T is higher, the greater is the proportion of the dissolved solids present. A detailed study has been made of the hydrolysis of the inulin (I) of chicory juice under varying conditions of reaction time, temp., and  $p_{\rm H}$ . Based on the results, a table is presented showing the amounts of HCl or H<sub>2</sub>SO<sub>4</sub> which must be added to chicory or artichoke juices of various concns. (5–25% of dry matter) in order to bring about complete hydrolysis of the (I) in 50 min. at 80°, 90°, or 100°.

I. A. P.

Use of polarographic methods in food analysis. G. PROSKE (Z. Unters. Lebensm., 1936, 71, 385–390).— The principle of the method is outlined and its application to, e.g., the determination of  $NO_2'$ ,  $NO_3'$ , MeCHO, cysteine and cystine,  $Ac_2$ , and nicotine described.

E. C. S.

Changes in odour of mushrooms. M. E. J. GILBERT (Parfum. mod., 1935, 29, 155—159; Chem. Zentr., 1935, ii, 934).—The causes of variations in odour are discussed. H. N. R.

Composition of forage crops. I. Rye grass (western wolths). A. G. NORMAN (Biochem. J., 1936, 30, 1354—1362).—The cellulose and lignin contents of rye grass, cut at fortnightly intervals, increased rapidly as maturity approached, and the xylan content of the cellulose increased with age. The polyuronide hemicelluloses, as judged by furfuraldehyde yield, did not exhibit any regular increase and were lower in mature than in young grass. A H<sub>2</sub>O-sol. fructosan was found in younger samples, reaching at one stage > 37%expressed as fructose, but the amount fell rapidly on maturity. Losses on hay-making were of the order of 10%, mostly accounted for by loss of fructosan.

P. W. C.

Composition and digestibility of mung bean silage; the silica-ratio procedure for studying digestibility. W. D. GALLUP and A. H. KUHLMAN (J. Agric. Res., 1936, 52, 889–894).—Analyses and digestibility trials with pigs are recorded. Changes in the ratio, nutrient: SiO<sub>2</sub>, during passage through the digestive tract were not sufficiently regular to permit their utilisation in digestibility trials of this kind.

A. G. P.

Mineral content of German feeding-stuffs and the influence thereon of manuring and source. Cereal and sweet-lupin straws. F. WASSMANN (Landw. Versuchs-Stat., 1936, 126, 227–305).—Numerous analyses are recorded. Considerable variations exist in the composition of the same material grown under different conditions. Vals. in general show marked differences from the commonly used analyses of Wolff. A. G. P.

Use of wood-sugar for fattening pigs. INST. TIERERNÄHR. UNIV. BERLIN (Landw. Versuchs-Stat., 1936, 126, 1-226).-Numerous feeding-trials are recorded in which wood-sugar syrup (from acid hydrolysis of wood) was fed as a 2:1 mixture with barley meal or with potato flakes. The digestibility of the N-free extract of the syrup averaged 71% for pigs and sheep and 79% for cattle. The dry matter of the syrup showed a starch equiv. of 62.1. The sugars of the syrup were as effective as was sucrose. The syrup-barley meal mixture was inferior in physical condition and less satisfactory in use than the syrup-potato ration. The starch equiv. consumption required per unit live-wt. increase was greater for syrup-barley than for barley alone; that for syrup-potato was the same as for potato alone. The mixed rations produced rather better carcase quality than either potato or barley alone. A. G. P.

Emulsions.—See I. Chinese NaCl.—See VII. Butter and margarine fats.—See XII. Insect destruction in grain. Mustard.—See XVI. Detection of sucrose.—See XVII. Fruit products.—See XVIII. Dairy effluents. Margarine effluents.— See XXIII.

#### PATENT.

Manufacture of dried stock feed. D. D. PEEBLES, Assr. to WESTERN CONDENSING Co. (U.S.P. 2,013,476, 3.9.35. Appl., 21.11.32).—Air-dried lucerne is chopped and fed to a desiccating chamber up which it is carried by drying gases. The dry material is pneumatically separated and delivered to a screen which separates the coarser material for grinding. E. B. H.

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

Anæsthetic ether : effect of impurities. Peroxides. J. H. COSTE and D. C. GARRATT (Analyst, 1936, 61, 459—464).—Peroxides in  $\text{Et}_2\text{O}$  are almost nonvolatile under various conditions analogous to those used in anæsthesia. It is concluded, therefore, that peroxides are not the cause of the after-effects produced by impure  $\text{Et}_2\text{O}$ . J. W. S.

Assay of syrups containing hypophosphites official in the National Formulary. G. L. JENKINS and C. F. BRUENING (J. Amer. Pharm. Assoc., 1936,

25, 491—496).—The U.S.P. method gives low results. Oxidation followed by pptn. of  $PO_4^{\prime\prime\prime}$  as  $MgNH_4PO_4$  or oxidation by Br and determination of Br consumed gives accurate results when applied to syrups containing  $NH_4$ , Ca, or Ca + Na hypophosphites. A modified B.P. method is recommended for determination of Ca in Ca-containing syrups. F. O. H.

General reaction of halogeno-tannins. F. ALLEGRI (Boll. Chim.-Farm., 1936, 75, 353—355; cf. A., 1935, 1244).—The violet colour produced by iodotannins in potable  $H_2O$  is not due to oxidation; it is not produced when tannic acid in potable or distilled  $H_2O$  is treated with oxidising agents. Distilled  $H_2O$  gives a yellow colour, the violet colour being produced only in presence of alkaline carbonates. Solutions of iodotannins cannot replace tincture of I in pharmaceutical use. E. W. W.

Synthetic camphor industry. G. BRUS and J. VÉBRA (Bull. Inst. Pin, 1936, 8-21, 57-66, 73-77, 107-113).-Separation of pure pinene hydrochloride from the crude chlorination product is wasteful. The crude hydrochloride from a turpentine with  $[\alpha]_D - 32 \cdot 25^{\circ}$ with NaOPh-NaOH at 160° gave a crude camphene (I),  $[\alpha]_D$  -50.77°,  $d_4^{15}$  0.873; careful fractionation at 3 mm. yielded (a) acyclic hydrocarbons (2%), (b) an inseparable mixture (20%) of camphene (50%) and  $\alpha$ -fenchene, cyclofenchene, tricyclene, (?) bornylene, and (?) another fenchene, (c) l- and dl-camphene (60%), (d) dipentene (11%) and l-limonene (2%), and (e) a mixture of terpinolene, alcohols [borneol, isoborneol, and (?) camphene hydrate], and oxidation products. Fraction (b) by esterification, hydrolysis, and oxidation gives an inseparable mixture of formates, yielding isofenchone and camphor. Fraction (c) gives nearly pure camphor. Formylation of the crude (I) gives esters which are difficultly separable. The best method is to use crude products up to the borneol-fenchol stage and purify these alcohols by way of the borates, which are then hydrolysed and oxidised; the ketones are readily separated. (I) is best determined by formylation.

R. S. C.

Production of synthetic camphor from Indian turpentine. M. L. BERI and J. L. SARIN (Chem. & Ind., 1936, 605—607).—Turpentine from Indian *Pinus excelsa* is suitable for production of synthetic camphor. Conditions for obtaining a 54% yield are detailed.

R. S. C.

Chemistry of barbituric acid derivatives. F. J. VAN LEENT (Pharm. Weekblad, 1936, 73, 873-885, 898-914).—A review of the methods of identifying and determining the most important barbituric acid derivatives, particularly veronal. When titrating these drugs with AgNO<sub>3</sub> and acid it is an advantage to use dil. EtOH instead of H<sub>2</sub>O. The following vals. for the  $p_{\rm H}$  of saturated solutions at 20° are recorded : veronal 5.56, luminal 5.92, dial 5.88. S. C.

Determination and identification of salicin. M. B. JACOBS and N. T. FARINACCI (Ind. Eng. Chem. [Anal.], 1936, 8, 279–281).—25—50 c.c. of solution containing 1—5 mg. of salicin (I) are slowly evaporated down to 10 c.c. with 25 c.c. of conc. HCl at 80°. The resultant saliretin,  $C_{14}H_{14}O_3$ , is filtered off, washed, and determined either gravimetrically or colorimetrically against standards after dissolving in 5—10% aq. NaOH (I) may be detected by the red colour it gives with alkaline  $PhN_2$ ·SO<sub>3</sub>H at 80°. S. C.

Assay of pepsin and its preparations. H. ESCHENBRENNER (Pharm. Ztg., 1936, 81, 790-792).— The hydrolysis of caseinogen by gastric enzyme preps. is approx.  $\infty$  the concn. of enzyme, whilst with actual pepsin preps. the concn. has practically no influence on the extent of the hydrolysis. The probability of this difference being due to the presence of stabilising substances and proteolytic enzymes other than pepsin in the gastric preps. is discussed. F. O. H.

Spongia fluviatilis seu lacustris, L. F. W. FREISE (Pharm. Zentr., 1936, 77, 488–489).—The drug should be preserved in its moist, fresh condition, without washing or drying. These processes destroy the algæ and their capability of producing  $HCO_2H$ . The sponge skeleton is formed by absorption of SiO<sub>2</sub> by the algæ from the surrounding H<sub>2</sub>O. E. H. S.

Preparation of decoctions and infusions of ipecacuanha root and of the powdered root of equal activity. L. DAVID (Pharm. Ztg., 1936, 81, 806-809).—The drug is treated with 10% HCl for 15 min. at 100°. All the alkaloid is extracted and the coagulating effect of the starch is destroyed. The author's process for the analysis (B., 1935, 1117) is superior to those of the Hungarian, German, and Swiss pharmacopœias. The need for the use of alkali-free glass to prevent the formation of H<sub>2</sub>O-sol. cephæline salts is emphasised. E. H. S.

Composition of Bulgarian tobacco. I. VLĂDESCU and N. DIMOFTE (Z. Unters. Lebensm., 1936, 71, 448– 451; cf. B., 1936, 813).—14 samples of Bulgarian tobacco had total N 1.6—3.7, protein 4.6—7.6, nicotine 0.26—2.42, total reducing substances (as glucose) 12.0—26.0, sol. carbohydrate 8.0—22.0, ash 10.0— 19.0%. E. C. S.

"Free" nicotine in tobacco. A. WENUSCH and R. SCHÖLLER (Pharm. Zentr., 1936, 77, 485–487).— The aq. extracts of all the samples of tobacco examined had acid reactions. "Free" nicotine (I) was not present. Its reported isolation is due to the facts that it can be extracted from light petroleum solutions of its salts with tobacco resin acids by water and also that certain (I) salts yield free (I) on steam-distillation. The belief that the higher is the petrol extract of tobacco the poorer is the quality is incorrect. E. H. S.

Determination of nicotine, ammonia, pyridine bases, and tar in tobacco smoke and the efficiency of some filter-tips for the absorption of nicotine. W. PREISS (Pharm. Zentr., 1936, 77, 437-442, 453-458).—Determinations in the indrawn smoke, the "free" smoke, the cigarette stump, and various "nicotine filters" are given. Normally the velocity of the smoke is too high for any considerable absorption to occur ( $\geq 20\%$ of each constituent is removed) and also when a filter is used the resultant stump is generally shorter and the total nicotine indrawn thus approximates to that from a cigarette without filter. E. H. S.

Nature of cigarette smoke. Technique of experimental smoking. J. A. BRADFORD, W. R. HARLAN, and H. R. HANMER (Ind. Eng. Chem., 1936, 28, 836— 839).—The desiderata of experimental smoking are discussed. A suitable machine is described. Cigarette smoke is best collected by gravity, followed by washing first with EtOH and then with  $H_2O$ . Divergence of results of different workers is largely due to variations in technique. R. S. C.

Quinine solutions for injection. E. H. VOGELENZANG (Pharm. Weekblad, 1936, 73, 1030—1031).—Disadvantages due to the strongly acid character of conc. quinine hydrochloride (I)-ethylurethane or -antipyrine (II) solutions can be overcome by adding N-NaOH (III) to  $p_{\rm H}$  7·3—7·4 but the resultant mixtures ppt. when diluted. The most practicable solution contains (I) 30 g., (II) 20 g., (III) 7·5 c.c., and H<sub>2</sub>O 100 c.c. and can be diluted to 3 vols. S. C.

Alkaloid content of Oregon-grown Cytisus scoparius. F. A. GILFILLAN and F. P. LOGAN (J. Amer. Pharm. Assoc., 1936, 25, 505-506).—Me-red is a suitable indicator for titration of sparteine, the content of which (as total alkaloid) in C. scoparius is approx. 1%. F. O. H.

[Alkaloid] determination in Semen sabadillæ and Acetum sabadillæ. F. KÜRSCHNER and W. IMMENKAMP (Pharm. Zentr., 1936, 77, 458—461).—A modified Swiss Pharmacopœia V method is used for Semen sabadillæ, and for Acetum sabadillæ the followslightly modified Saxholm process : EtOH and AcOH are removed from the vinegar (60 g.) by evaporation and the residue is made alkaline with aq. NH<sub>3</sub> and shaken with Et<sub>2</sub>O (60 g.). 50 g. of the clear extract are evaporated, the residue is dissolved in EtOH (10 c.c.) and H<sub>2</sub>O. (10 c.c.), 0.1N-HCl (5 c.c.) is added, and the mixture titrated with 0.1N-NaOH. E. H. S.

Otto of rose. E. J. PARRY (Perf. Essent. Oil Rec., 1936, 27, 278—279).—Adulteration with citronellol (I) is discussed. It is considered that samples which have a (I)/geraniol ratio of > 3 should be regarded with suspicion. The average ratio is about 2. E. H. S.

Presence of methyl alcohol and formaldehyde in essential oils and in solutions of essential oils in ethyl alcohol. Y. R. NAVES (Parfums de France, 1935, 13, 60-73; Chem. Zentr., 1935, ii, 934-935).—Methods of determining MeOH and CH<sub>2</sub>O in essential oils are discussed; the presence of MeOH and CH<sub>2</sub>O is not necessarily due to adulteration with MeOH. H. N. R.

Emulsions.—See I. Insect destruction [on tobacco].—See XVI.

#### PATENTS.

Preparation of remedies for gonorrhœa and dental suppurating diseases, more especially discharge of alveolar pus. H. KIMURA (B.P. 447,949, 23.5.35. Jap., 25.10. and 6.11.34).—A prep. having powerful germicidal properties is made by compounding 0.03 pt. of oleo-resin from pyrethrum extract, purified by cooling a solution in light petroleum (b.p. 20-60°) to -20° and filtering from impurities, 0.02 pt. of perillaldehyde (80-85%) from oil of *Perilla nankinensis*, and 99.95 pts. of paraffin. S. C.

Preparation of choline esters and their salts. J. K. CLINE, ASST. to MERCK & Co. (U.S.P. 2,012,268, 27.8.35. Appl., 8.2.34. Cf. U.S.P. 1,957,443; B., 1935, 287).—The choline salt is heated with excess of the acid anhydride and the ester pptd. with anhyd.  $Et_2O$ . R. S. C.

Manufacture of aliphatic-aromatic amines. K. W. ROSENMUND and F. KÜLZ (U.S.P. 2,006,114, 25.6.35. Appl., 8.2.33. Ger., 27.4.31).-The following compounds (substitutes for papaverine) are prepared by known methods : bis-(\$-phenylethyl)-, b.p. 188-190°/8 mm. (B,HCl, m.p. 137°), bis-(y-phenylpropyl)- (non-cryst. B,HCl; B,HClO<sub>4</sub>, m.p. 70°), β-phenylethyl-β-o-methoxyphenylethyl- (B,HCl, m.p. 155°), -ethylamine, bis-(Bphenylethyl)allylamine, b.p. 182°/4 mm. (B,HCl, m.p. 119—120°),  $\alpha\beta$ -diphenylethyl- $\beta$ -phenylethylpropylamine (B,HCl, m.p. 263°), β-phenylethyl-β-phenylisopropyl-(B,HCl, m.p. 124°), bis-(3: 4-dimethoxy-β-phenylethyl)-(B,HCl, m.p. 242°), β-phenylethyl-β-methoxy-β-phenylethyl- (B,HCl, m.p. 100°), benzyl-y-phenylpropyl-(B,HCl, m.p. 146°), benzyl-δ-phenylbutyl- (B,HCl, m.p. 113°) -methylamine. The following intermediate products are described : β-phenylethylallylamine, b.p. 109°/7 mm.,  $\beta$ -phenlyethyl- $\beta$ -phenylisopropylamine (B,HCl, m.p. 160°),  $\alpha\beta$ -diphenylethyl- $\beta$ -phenylethylamine (B,HCl, m.p.  $\beta$ -phenylethyl- $\beta$ -o-methoxyphenylethylamine 261°). (B,HCl, m.p. 142°), bis-(3:4-dimethoxy-β-phenylethyl)amine (B,HCl, m.p. 199°), β-phenylethyl-β-methoxy-βphenylethylamine, b.p. 213-215°/15 mm. A. W. B.

Manufacture of antiseptic germicidal substances [isobutylchlorophenols]. Boot's PURE DRUG Co., LTD., and J. MARSHALL (B.P. 447,618, 22.11.34).— A mixture of o- and p-C<sub>6</sub>H<sub>4</sub>Bu<sup>g.</sup>OH, b.p. 95—132°/12 mm., obtained by heating equal pts. of PhOH and n- or sec.-BuOH with 3 pts. of ZnCl<sub>2</sub> at 180° for  $\frac{1}{2}$  hr., is treated with 0.95 pt. of SO<sub>2</sub>Cl<sub>2</sub> at room temp. and finally at 100°. The resultant mixture of isobutylchlorophenols, b.p. 105—140°/12 mm., has a PhOH coeff. of 130—140. p-Chloro-o-isobutylphenol, m.p. 57—58°, isolated from the mixture, and the residual mixture of isomerides from the isolated product, have coeffs. of 100. S. C.

Antiseptic. J. EBERT, Assr. to FARASTAN Co. (U.S.P. 2,010,512, 6.8.35. Appl., 5.12.32).—Admixture with a colloidal substance, e.g., 4'-amino-3'-ethoxybenzeneazop-diphenyl-p'-azo-4''-amino-3''-carboxybenzoic acid, greatly increases the bacteriostatic effect of 2-hydroxyquinoline salts and the amount thereof excreted unchanged in the urine, thereby enhancing its val. as urinary and general antiseptic. Thirteen other bisazo compounds, having a similar effect, are enumerated. R. S. C.

Manufacture of stable aqueous solutions of anæsthetic substances [alkamine aminobenzoates]. I. G. FARBENIND. A.-G. (B.P. 447,679, 12.11.34. Ger., 11.11.33).—The addition of a small amount of urea to aq. solutions of alkamine esters of amino- and alkylamino-benzoic acids prevents alteration in  $p_{\rm H}$  6—7 during sterilisation with steam. S. C.

[Preparation of] alkoxyphenyliminazoles [glyoxalines; local anæsthetics]. M. ENGELMANN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 2,005,538, 18.6.35. Appl., 27.8.32).—Alkoxyphenylthiocarbimides are condensed with NH<sub>2</sub>·CH<sub>2</sub>·CH(OEt)<sub>2</sub> (I), and the resulting thiocarbamides are cyclised by heating with aq. inorg. acids to 2-thiolglyoxalines, the SH group of which is removed by oxidation. E.g., p-anisylacetalylthiocarbamide, m.p. 81°, from p-OMe<sup>•</sup>C<sub>6</sub>H<sub>4</sub><sup>•</sup>NCS and (I), is converted by 30% H<sub>2</sub>SO<sub>4</sub> at the b.p. into 2-thiol-1-panisylglyoxaline, m.p. 216—217°, which is oxidised by 20% aq. NH<sub>3</sub> to 1-p-anisylglyoxaline (nitrate, m.p. 133— 134°). p-Phenetyl-acetalylthiocarbamide, m.p. 66—67°, and -glyoxaline, m.p. 50° (nitrate, m.p. 135°; 2-SH derivative, m.p. 205°), are similarly prepared.

H. A. P.

Production of (A) 1-phenyl-2 : 3-dimethyl-5-pyr-[antipyrine], (B) 4-dimethylamino-1azolone phenyl-2:3-dimethyl-5-pyrazolone [pyramidone]. B. REUTER (U.S.P. 2,005,505-6, 18.6.35. Appl. [A, B] 28.11.32. Ger., [A] 12.4.28, [B] 10.3.27).-(A) Methylation of phenylmethylpyrazolone, e.g., with MeCl, Me<sub>2</sub>SO<sub>4</sub>, p-C<sub>6</sub>H<sub>4</sub>Me·SO<sub>3</sub>Me (I), gives, in addition to the 2: 3-Me, compound, some 5-OMe-derivative. This is decomposed by boiling with aq. alkalis or alkali carbonates with regeneration of antipyrine (II). When (I) is used the cryst. p-toluenesulphonate of (II) may first be filtered off. (B) The cryst. p-toluenesulphonate of (II), or the (acidified) solution obtained by boiling the above methylation mixture with alkalis etc., is treated with HNO2, and the resulting suspension of NO-compound is reduced with Zn dust and NaHSO3 at 0-30°; the solution of 4-amino-1-phenyl-2: 3-dimethylpyrazolone thus obtained is filtered from suspended ZnSO<sub>3</sub> and further methylated by heating with CH<sub>2</sub>O and HCO<sub>2</sub>H. The resulting pyramidone is isolated by evaporation of the solution and pptn. with a base or by extraction with a solvent  $(C_6H_6)$  after basification. H. A. P.

Manufacture of sterol derivatives. A. CARPMAEL. From SCHERING-KAHLBAUM A.-G. (B.P. 449,379, 22.11.34, 17.1. and 26.3.35).-The side-chain of sterols is entirely or partly removed by oxidation with dil. CrO3-AcOH, usually at 40-45°, after protection of the OH and cyclic ethylenic linkings. Ketonic products are isolated after removal of acids by NH2 CO.NH. NH2, benzoylation, or distillation/high vac. Stigmasteryl acetate dibromide is oxidised by CrO3 at 45° or CaMnO4-AcOH and then treated with Zn dust-AcOH, yielding dehydroandrosterone (I), m.p. 148-150° (semicarbazone, m.p. 280°; benzoate), and pregnanolone (II), m.p. 190° (semicarbazone, m.p. 255-259°; benzoate). Cholesteryl acetate dibromide affords similarly (CrO<sub>3</sub>) an unsaturated OHketone (III) [ ? = (I)], C<sub>19</sub>H<sub>28</sub>O<sub>2</sub>, m.p. 148° [semicarbazone, m.p. 280° (decomp.)]. Sitosteryl acetate dibromide at 45° or room temp. gives (I), also obtained from cholesterol Me ether dibromide. Oxidation of cholesteryl benzoate dibromide gives a benzoate, hydrolysed to (III), whilst the mother-liquors contain the benzoate of (II). Cholesteryl acetate hydrochloride (prepared by HCl-AcOH), m.p. 146°, gives a substance, converted into (III) by N-KOH-MeOH or by boiling C<sub>5</sub>H<sub>5</sub>N. Sitosteryl chloride dibromide gives an unsaturated Cl-ketone, m.p. 157° (semicarbazone, m.p. 275)°, converted [? into (I)] by KOAc in AcOH or EtOH at 180-200°. R. S. C.

Production of ketocyclopentanodimethyltetradecahydrophenanthrol. SCHERING-KAHLBAUM A.-G. (B.P. 449,326, 24.12.34. Ger., 23.12.33).—Pregnanolone and MgPhBr give a dihydric alcohol, the sec.-OH of which is esterified by  $Ac_2O-NaOAc$ , whilst the side-chain containing the tert.-OH loses  $H_2O$ . The resulting unsaturated acetate, when ozonised, yields the acetate (I), hydrolysed by KOH-EtOH to 17-ketodimethyltetradecahydrophenanthr-3-ol (II). (I) and (II) have the physiological properties of the male sex hormone. R. S. C.

Manufacture of physiologically active derivatives of cyclopentanophenanthrol. A. CARPMAEL. From SCHERING-KAHLBAUM A.-G. (B.P. 449,461, 23.11.34).— Hydrogenation (Pt) of 8 : 12-dimethylperhydrocyclopentanophenanthra-3 : 17-dione or the  $\Delta^4$ -unsaturated diketone (I) in AcOH-HBr gives mainly cis-8 : 12-dimethylperhydrocyclopentanophenanthra-3 : 17-diol (II) with increased pharmacological activity. (I) and Na-EtOH give 80% of the trans-form of (II). R. S. C.

Medicinal [mercurial] preparations having a diuretic effect. CHINOIN GYÓGYSZER ÉS VEGYÉSZETI TERMÉKEK GYÁRA R.T. (KERESZTY & WOLF) (B.P. 447,877, 12.11.35. Hung., 12.3.35).—Mercurials suitable for rectal administration are obtained by partly (70—85%) neutralising compounds containing both  $CO_2H$  and Hg OH groups in the same mol., e.g., mercurated camphoric  $\alpha$ -allylamides, with alkali (NaOEt) in a solvent (EtOH). The products give clear, stable solutions in H<sub>2</sub>O which are not strongly alkaline. S. C.

Stabilised organo-mercuri-sulphur compounds. M. S. KHARASCH, Assr. to E. LILLY & Co. (U.S.P. 2,012,820, 27.8,35. Appl., 17.2,34).—Salts R·Hg·S·Ar·CO<sub>2</sub>Na are best stabilised against oxidation and the catalytic effect of traces of metals by small amounts of aliphatic  $\alpha\beta$ -diamines, probably by removal of the metal as complex. R. S. C.

[Manufacture of] organic arsenic compounds. W. W. TRIGGS. From PARKE, DAVIS & Co. (B.P. 444,882, 25.7.34).—A C-As-substituted phenol is condensed with an alkylene or aralkylene halogenohydrin to give try-panosides or spirochæticides of high therapeutic index. E.g., p-OH·C<sub>6</sub>H<sub>4</sub>·AsO<sub>3</sub>H<sub>2</sub> with C<sub>2</sub>H<sub>4</sub>Cl·OH and aq. NaOH at 20° gives p- $\beta$ -hydroxyethoxyphenylarsinic acid, m.p. 128°; the corresponding  $\gamma$ -hydroxypropyl ether has m.p. 146°. H. A. P.

Scent mixtures. G. M. CLARK. From HEINE & Co. A.-G. (B.P. 449,211, 16.7.35).—2-*n*-Hexylidene- and -heptylidene-cyclopentanone, 2-*n*-hexylidenecyclohexanone, and 5-*n*-heptylidene-3-methylcyclopentanone are used in scent mixtures, which are detailed. Other alkylidene derivatives may also be used.

R. S. C.

Condensation products. Substituted  $\beta$ -phenylethylamines. Amidines.—See III.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic properties of gelatin. I. Labile sulphur and its relation to the preparation of photographic emulsions. V. A. BEKUNOV (Photo-Kino Chem. Ind., 1935, No. 1–2, 13–20).—Dichroic fog was found to consist of  $Ag_2S$ . The photographic activity and fogging power of gelatin are directly related to the labile S. During ripening the amount of Ag<sub>2</sub>S on the emulsion grains increases. CH. Abs. (e)

Production of photographic silver halide-gelatin emulsions. B. CLAUS (Z. tech. Physik, 1935, 16, 109— 115; Chem. Zentr., 1935, ii, 958).—AgBr + a little AgI, free from or containing only a small amount of binding agent, is dispersed in gelatin by the action of ultrasonic radiation, and subjected to simultaneous or subsequent thermal treatment. Sensitising dyes are adsorbed on the grains before dispersion to a greater extent than by the finished plates. J. S. A.

Mercury salts in photographic emulsions. A. STEIGMANN (Phot. Ind., 1936, 34, 791).—Various experiments on adding HgCl<sub>2</sub>, in solution in H<sub>2</sub>O or in 10% aq. gelatin, to emulsions show that fogging is reduced, but more or less desensitisation is also produced. Na mercurisulphite produces similar effects, and if applied as a bath for films or papers after exposure and before development it will in many cases accelerate development. J. L.

Green and brown developing emulsions. A. STEIG-MANN (Phot. Ind., 1936, 34, 764—766).—Formulæ are given for the prep. of sensitive emulsions which will develop to give green and brown tones. Substances retarding crystallisation and org. I compounds with labile I, for forming the AgI emulsions required, are specified under the names Fesatinol I and II, and Fesa-iodine salt, respectively. J. L.

Influence of acid substrata on the photographic properties of emulsions. M. I. SCHOR and A. V. BORIN (Photo-Kino Chem. Ind., 1935, No. 1—2, 78—83).—The desensitising effect of substrata depends on the stabilising acid retained in the layer. The effect  $\infty$  the mol. concn. The amount of acid left depends on its concn. in the substrating fluid and on the operation of the machine. AcOH ( $\gg$  12 g. per litre) was better than phthalic or salicylic acid. CH. ABS. (e)

Connexion between exposure and blackening on exposure [of photographic emulsion] to X-rays. II. J. E. DE LANGHE (Z. wiss. Phot., 1936, 35, 171–174).— Theoretical. An extension of earlier work (B., 1935, 783) to rays of > one  $\lambda$ , and including fogging of the emulsion. A. J. M.

Adsorption photography. F. L. BURMISTROV (Phot. J., 1936, 76, 452-459).-Residual "images" on the washed glass of old plates are shown to be due to adsorption of Ag by thin films of SiO<sub>2</sub> gel on the surface of the glass. A new type of photographic layer may be made by treating SiO<sub>2</sub> layers with AgNO3, Ag silicate (I) being formed ; (I) is sensitive to light, like Ag halides, and can be similarly developed, using acid developer. Films of (1) can be prepared by pouring a freshly pptd. gel (before setting) on to a glass plate, or the film may be formed from the glass itself by treatment of the surface with, e.g., CuSO<sub>4</sub>, FeCl<sub>3</sub>, or Pb(NO<sub>3</sub>)<sub>2</sub>. The film is then treated with AgNO<sub>3</sub> solution. Some preliminary experimental results are given, and the theory of the process is fully The thin layers have a highly disperse discussed. structure, and the images have high resolving power. The process is suitable for the production of fine graticules, and improved neutral photometric filters and wedges for the ultra-violet. J. L.

Modern photographic positive papers. W. REIN DERS and J. BONTENBAL (Chem. Weekblad, 1936, 33, 486 -492).—The theory of correct photographic rendering of tone vals. is discussed, and the characteristic curves and other properties of 16 printing papers are compared. J. L.

Measurements of glossiness and smoothness of photographic papers. A. KLUGHARDT (Phot. Ind., 1936, 34, 761—762, 792—793).—The theory of the reflexion of light from ideal and actual surfaces is discussed at length. The author's method, previously used for textiles and paper, is applied to photographic papers, and results with glossy, semi-matt, and matt postcards are shown graphically. The max. vals. for the glossiness with a basic brightness ( $h_0$ ) of 10 can be used as a measure of the smoothness. J. L.

Variation of the optical density of photographic plates with the conditions of drying. J. Roig and J. THOUVENIN (Compt. rend., 1936, 202, 1982–1984).— The density of blackening increases with the time of drying (for slow drying). H. J. E.

Spectral sensitograph for recording coloursensitivity curves of photographic coatings. K. FOIGE (Phot. Korr., 1935, 71, 50–52; Chem. Zentr., 1935, ii, 960).—The method is described. J. S. A.

Chemistry of sensitisers and photography in the infra-red. H. WAHL (Chim. et Ind., 1936, 36, 16— 28).—A review. J. L.

Fine-grain [photographic] developers. Lüppo-CRAMER (Phot. Korr., 1935, 71, 49–50; Chem. Zentr., 1935, ii, 959).—A review. J. S. A.

Alkali in developers. G. P. FAERMAN and N. N. SHISHKINA (Photo-Kino Chem. Ind., 1935, No. 1–2, 30–36).—The rôle of  $p_{\rm H}$  in photographic developers is discussed. For a given [Ag] there is an optimum  $p_{\rm H}$  for physical development. CH. Abs. (e)

Deterioration of photographic developers by the action of bacteria, and the removal of this phenomenon. H. BORST (Phot. Ind., 1936, 34, 873-874).— Five species of bacteria commonly occur ; they grow on the Na<sub>2</sub>SO<sub>3</sub>, producing H<sub>2</sub>S. Developing tanks can be disinfected by treatment overnight with 2-3% CH<sub>2</sub>O; other chemicals are often ineffective. The requirements of a disinfectant suitable for addition to developer are discussed; "Voigtländer X" is recommended, being effective with 0.1% addition, without affecting the properties of the developer. J. L.

Fine-grain development and sensitivity. H. FRÖT-SCHNER (Phot. Ind., 1936, 34, 820—822).—For fine-grain development, all of four films investigated required 2—4 times normal exposure, for correct rendering. The sensitivities claimed for the films were within the prescribed tolerance (3/10° DIN), but were all on the high side; more accuracy is therefore desirable.

J. L.

Hot-weather processing [in X-ray photography]. C. W. FORD (X-Ray Tech., 1935, 7, 9–13).—More KBr is added to the developer to diminish fog. An acid stopbath is used. The acidity of the fixing bath is maintained by adding AcOH when necessary. Rapid washing in a liberal flow of  $H_2O$  is essential. CH. ABS. (e)

CL. XXII.-EXPLOSIVES; MATCHES. CL. XXIII.-SANITATION; WATER PURIFICATION.

Timer Demension

Determining Fe in used engine oils.—See II. Determining Cu in brass etc.—See X.

## XXII.—EXPLOSIVES; MATCHES.

Upper temperature limit of explosion of explosives. K. K. ANDREEV (Acta Physicochim. U.R.S.S., 1935, 3, 119—126).—The variation in the intensity of the explosion with the temp. to which an explosive is heated was investigated in the case of trotyl, picric acid, tetryl, trinitroresorcinol, pentaerythritol tetranitrate, nitroglycerin, and dynamite. For all these substances the explosion was less intensive at higher temp. It is suggested that intermediate products, the concn. of which depends on temp., play an important part in determining the intensity of explosion.

#### A. J. M.

## Priming mixture. J. D. MCNUTT, Assr. to WIN-CHESTER REPEATING ARMS CO. (U.S.P. 2,009,556, 30.7.35. Appl., 6.11.31).—A mixture of tetrazene (I) and diazonitrophenol (II) with other ingredients is employed. A suitable composition for centre-fire cartridges contains (I) 1—4, (II) 12—18, Ba(NO<sub>3</sub>)<sub>2</sub> 25—40, Sb<sub>2</sub>S<sub>3</sub> 8—18, PbO<sub>2</sub> 15—25, and CaSi<sub>2</sub> 8—20%, and for rim-fire cartridges (I) 4—7, (II) 15—20, basic Pb azide 6—12, Ba(NO<sub>3</sub>)<sub>2</sub> 20—30, PbO<sub>2</sub> 12—20, and ground glass 20— $28\%_0$ . W. J. W.

PATENTS.

Manufacture of explosives. LIQUID OXYGEN EX-PLOSIVES, LTD., and A. E. LANCE (B.P. 447,887, 10.12.35). —The absorbent material for a liquid  $O_2$  explosive consists of cellulosic material, preferably wadding or soft paper, coated with a hydrocarbon of high b.p., *e.g.*, petroleum jelly or soft paraffin wax, and enclosed in a paper wrapping to form a cartridge. After impregnation the explosive may be fired with a Pb azide detonator.

W. J. W.

## XXIII.—SANITATION; WATER PURIFICATION.

Germicidal effect of alcohols. C. E. COULTHARD and G. SYKES (Pharm. J., 1936, 137, 79—81).—MeOH, EtOH, and  $Pr^{g}OH$  have marked germicidal properties towards vegetative bacteria but are inactive towards spores. Suitable aq. solutions of these alcohols (70% in the case of EtOH) containing 1% of NaOH, KOH, HCl, HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>, H<sub>3</sub>PO<sub>4</sub>, or 10% of amyl-*m*-cresol will, however, destroy spores in a few hr. A. L.

Berlin city sewerage, 1935. F. LANGBEIN (Gesundheitsing., 1935, 58, 213-223; Chem. Zentr., 1935, ii, 898).—A review of methods of sewage treatment.

J. S. A.

Influence of dairy effluents on treatment of sewage of small towns. L. W. HAASE (Kl. Mitt. Ver. Wasser-, Boden-, Lufthyg., 1935, 11, 37-42; Chem. Zentr., 1935, ii, 898).—The interference to normal sewage treatment caused by 25-30% of dairy effluent is minimised by liming the latter effluent before mixing with the main bulk. J. S. A.

Effluents of the margarine industry. GLASER (Gesundheitsing., 1935, 58, 254-256; Chem. Zentr., 1935, ii, 898).-Measures for neutralisation of acid effluents, and for suppression of corrosion by acid fumes, are discussed. J. S. A.

Complete treatment of distillery wastes. A. M. BUSWELL and M. LEBOSQUET (Ind. Eng. Chem., 1936, 28, 795—797).—In the demonstration-scale plant described, the slop undergoes anaërobic fermentation in closed, domed tanks. The effluent is then pumped to a trickle filter packed with blast-furnace slag, after dilution with sedimented effluent from the filter, in order to allow final aërobic digestion. The operation of the plant is discussed. A final stable effluent is obtained, with relatively little sludge from which, however, the bulk of the P of the grain can be recovered. The gases from the tanks are burned, whilst the high [NO<sub>3</sub>'] in the filter prevents trouble from odours. I. A. P.

The Magno-process [of water treatment]. L. KAATZ and H. E. RICHTER (Gas- u. Wasserfach, 1936, 79, 575-576; cf. B., 1936, 398) .- A trial run for removal of CO2 from H2O with "Magnomass" (I) (partly burned dolomite in granules) showed that with fresh material the alkalinity of the treated H<sub>2</sub>O was very high. Such H<sub>2</sub>O is also liable to become turbid through the reaction  $MgO + Ca(HCO_3)_2 = MgCO_3 + CaCO_3 + H_2O$  and must be run to waste. As MgO and MgCO3 are more rapidly dissolved than is CaCO<sub>3</sub> they are washed out and the eventual reaction at the filter is merely  $CaCO_3 + CO_2 +$  $H_2O = Ca(HCO_3)_2$ . Fe is satisfactorily removed, but not Mn unless filtration is much slower than with sand filters. (I) is much more costly than  $CaCO_3$  and for the  $H_2O$ supply in question offers no advantages. C. I.

Elimination of salts from water. J. BILLITER (Trans. Electrochem. Soc., 1936, 70, Preprint 3, 35–48). —Natural H<sub>2</sub>O containing a wide diversity of salts can be readily and efficiently desalted by electrolysis, employing a 3-compartment cell; in some cases pretreatment with Cu-zeolite is recommended. The purification is primarily due to electrodialysis and not to electroosmosis. The average current efficiency is 18%, > 80%of the current being consumed in the recombination in the middle compartment of the cell of H and OH' which were formed in the outer compartments. C. J.

Problems of deferrisation and demanganisation [of water]. R. S. WESTON (J. New England Water Works Assoc., 1936, 50, 231-238).—Prolonged aëration followed by coagulation of the finely-divided particles of hydroxide is usually effective in removing Fe and Mn. Details of other recognised methods and of procedures adopted in exceptional cases are given. C. J.

Odour of iodoform in mineral waters containing iodine. O. SCHMATOLLA (Chem.-Ztg., 1936, 60, 638; cf. Lindner, B., 1936, 670).—The distribution of I in the atm. and the sea is discussed. A. J. M.

Odour of iodoform in mineral waters containing iodine. E. LINDNER (Chem.-Ztg., 1936, 60, 638; cf. preceding abstract).—The odour of  $CHI_3$  in the mineral  $H_2O$  examined occurred sporadically. Separation of I was due to the action of  $HNO_2$ . A. J. M.

Anti-gas clothing.—See VI. Halogeno-tannins.— See XX.

PATENT.

Combating insect pests.-See XVI.