# BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY SEPT. 27 and OCT. 4, 1935.\*

## I.-GENERAL; PLANT; MACHINERY.

watering of the calorific value of rechnical

Boiler practice in H.M. Office of Works, with special reference to the use of blended fuels. A. C. PALLOT (J. Inst. Fuel, 1935, 8, 250-276).-Data relating to the design and performance of the more important boiler installations are recorded. The performance of boilers with thermostatically-controlled fan draught is discussed ; in general, fans have been found preferable to steam jets, giving greater flexibility and less risk of external corrosion. Charts for giving the leading characteristics of a fuel in terms of its proximate analysis and calorific val. are illustrated. The importance for furnace control of continuous records of flue temp. and CO<sub>2</sub> content of the flue gases is emphasised. Tests with bituminous slacks and small coals gave good combustion efficiencies in some installations, but proved unsatisfactory in others; in nearly all cases the smoke emission was serious and difficulty due to grit emission was experienced. Some improvement in both respects was effected by burning a suitable blend of coals, in particular a blend of Kent smalls and Scottish anthracite, but care in stoking was necessary. Preliminary experiments on reducing the smoke emission by injecting preheated secondary air into the furnace have given good results. A. B. M.

Principles, development, and examples of technical firing calculations. III. Formulæ for calculating the heat developed by the combustion gases in a furnace. H. SCHWIEDESSEN (Arch. Eisen-hüttenw., 1935-6, 9, 23-30; cf. B., 1933, 289).--Mathematical. A. R. P.

Safety precautions in chemical manufacture. T. J. DIXON (Proc. Chem. Eng. Group, 1934, 16, 35-39). -A discussion.

Effect of viscosity variation on the rupture of plastic bodies. R. K. SCHOFIELD and G. W. S. BLAIR (Nature, 1935, 136, 147).-The difference in behaviour of viscous materials, e.g., golden syrup, which run out continuously from an orifice and those like clay paste which run out in blobs is attributed to a difference in the rate of change of  $\eta$  with shearing stress. This factor is important in the "shortness" of dough, and the behaviour of metals on extension. L. S. T.

Oils and plastic materials. M. MATHIEU (Chim. et Ind., 1935, 33, 1326; cf. B., 1935, 257).-Points in the previous paper are further explained and some corrections made. A. B. M.

Motion of granular or pulverous materials in a horizontal rotating cylinder. II. Mechanism of mixing and determination of mixing velocity. Y. OYAMA (Bull. Inst. Phys. Chem. Res. Japan, 1935, 14,

570-583).-Mechanism of mixing was investigated for circular, parabolic, and spiral motion. The relation between mixing grade and time was determined and plotted by photographic photometry. N. M. B.

and preferably increases in diam, so that

Conveyance of solid particles by fluid suspension. R. F. DAVIS (Engineering, 1935, 140, 1-3, 124-125).-Equations are derived for the velocity required by a horizontal fluid stream to keep solid particles in suspension with and without taking into account the ratio of solid to fluid. Examples are given. D. K. M.

Simple method of determining refractive indices of liquids. II. A. MAYRHOFER [with E. SOMMER] (Sci. Pharm., 1934, 5, 105—108; Chem. Zentr., 1935, i, 1094; cf. B., 1933, 525).—The method described previously may be applied to sugar solutions, fatty oils, tinctures, etc., using glycerol + H<sub>2</sub>O, paraffin + C<sub>5</sub>H<sub>11</sub>·OH, cineole, or 1-C<sub>10</sub>H<sub>7</sub>Br as standards.

J. S. A.

Distillation and rectification .- See III. Adsorbents .- See VII. Determining sulphate in H2O .--See XXIII.

## PATENTS.

Catalytic apparatus. W. J. EDMONDS, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,980,718, 13.11.34. Appl., 18.12.29).-To regulate the reaction temp. in an exothermic process at high temp. and pressure part of the ingoing gases is subjected to heat-exchange with (1) the outgoing gases and (2) the catalyst mass through the tube walls; another part exchanges with (2) only, and a third part is not preheated at all. B. M. V.

Elements for use in tubular heat-exchange apparatus. SUPERHEATER Co., LTD. From SUPER-HEATER Co. (B.P. 431,606, 15.5.34).—A closure for the clean-out aperture in a bottle-shaped return bend is described. B. M. V.

Artificial refrigeration. H. S. BOOTH and G. G. TORREY (U.S.P. 1,960,368, 29.5.34. Appl., 25.5.31).--Claim is made for a refrigerating system comprising SO, as refrigerant and Be acetylacetonate or ethylacetoacetate as absorbent. A. R. P.

Mills or grinders for paints or other substances in viscous or pasty form. ANC. ETABL. LE CLEZIO (B.P. 431,530, 31.1.34. Fr., 31.1.33).-A conical worm in a conical hopper forces the material to a space between two grinding bars in contact with a single roll. [Stat. B. M. V. ref.]

Capillary colloid mill. S. F. ACREE (U.S.P. 1,980,589, 13.11.34. Appl., 11.9.24. Renewed 27.6.32).—The space between the rotor and the fixed part decreases in the direction of travel of the fluid

\* The remainder of this set of Abstracts will appear in next week's issue. 833

and preferably increases in diam. so that the shearing rate is increased. Several forms of construction are described. B. M. V.

**Concentrator.** M. J. LIDE (U.S.P. 1,980,490, 13.11.34. Appl., 4.11.29).—The deck and air pan of a pneumatic table are mounted on springs and shaken both horizontally and vertically by means of an unbalanced rotating wt., bumpers being provided to resist the horizontal motion in one direction. B. M. V.

Filtration. I. M. PERKINS, ASST. to ATLANTIC REFINING CO. (U.S.P. 1,980,431, 13.11.34. Appl., 31.1.31. Renewed 14.2.34).—A percolation filter having means for wetting the filter material (M) (during formation of the bed) with liquid of the same nature as that to be filtered is described. If the liquid is dirty it is caused to emerge clear by depositing the wetted Mupon a layer of dry M. B. M. V.

Filters. W. A. and F. C. HOVEMAN (B.P. 431,686, 8.1.34).—A filter bed is divided into sections by partitions which are alternately high and low, the latter being surmounted by collecting gutters which stand slightly above the surface of the sand or the like and serve as outlets during the back-washing, which may be effected section by section. B. M. V.

Apparatus for obtaining a dry substance from solutions, emulsions, or suspensions. J. M. VISSER (B.P. 431,756, 11.1.35. Switz., 15.1.34).—A vac. pan is provided with a flat bottom and with stirring and rolling devices; it also contains a quantity of dry powder to prevent the feed liquid (F) from coming directly in contact with the heated surfaces. F is supplied through a rotating nozzle. B. M. V.

Aëration of liquids or dispersion of gases or vapours in liquids. DISTILLERS Co., LTD., and J. LOCKEY (B.P. 431,674, 27.3.35. Addn. to B.P. 387,486 and 393,551; B., 1933, 336, 657).—The wire (of the diffusing device) is of flattened cross-section, serrations of the same or different pitch and/or angle are formed on the flat faces, and the spiral is wound with those faces touching. B. M. V.

Apparatus for separation of gases. H. DUGGAN and I. R. MCHAFFIE, Assrs. to GRASSELLI CHEM. Co. (U.S.P. 1,980,791, 13.11.34. Appl., 18.10.33. Can., 24.8.33).—A triple-pass heat exchanger with allowance for differential expansion is described. B. M. V.

Apparatus for treatment of gases. C. G. HAWLEY, Assr. to CENTRIFIX CORP. (U.S.P. 1,980,522, 13.11.34. Appl., 19.1.31).—The gas is passed downwards through fountains of liquid, impinges on the surface of a pool, and passes upwards through moisture-eliminating means. B. M. V.

[Springs for rollers of] sugar-cane or other rolling mills. FAWCETT, PRESTON & Co., LTD., J. C. MACGILLIVRAY, and G. CHAPMAN (B.P. 431,655, 17.2.34).

Heat-insulating cement.—See IX.

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

British coals and their analyses. ANON. (Fuel Econ., 1935, 10, 946-947, 977-979).-Data for 36 coals are given.

Calculation of the calorific value of technical fuels. M. BRUTZKUS (Compt. rend., 1935, 200, 2168–2170; cf. B., 1935, 133, 179).—A method of calculation, based on the  $O_2$  required for combustion, is described. Observed and calc. vals. are compared. H. J. E.

Carbonising properties and constitution of Alma bed coal from Spruce River No. 4 mine, Boone County, W. Va. A. C. FIELDNER, J. D. DAVIS, R. THIESSEN, E. B. KESTER, W. A. SELVIG, D. A. REYNOLDS, F. W. JUNG, and G. C. SPRUNK (U.S. Bur. Mines, Tech. Paper 562, 1935, 41 pp.).-The sample of coal, which was tested by the method previously described (cf. B., 1931, 6), contained H<sub>2</sub>O 2·1, volatile matter 37·7, fixed C 53.1, and ash 7.1%. The yield and quality of the products obtained at carbonising temp. of 500-1100°, and the results of chemical, physical, and petrographic studies of the coal, are recorded. Petrographically it is classed as attrital-anthraxylous; it is largely a bright translucent coal containing layers of splint and semisplint. It tends to give a high yield of gas and tar, and is particularly adapted to carbonisation at the higher temp. The coke was improved by raising the charging density from the normal (50 lb./cu. ft.) to 56 lb./cu. ft. A. B. M.

Present position of the wood-carbonising industry. K. KIETAIBL (Oesterr. Chem.-Ztg., 1935, 38, 105—108).—Improvements in the processes of wood distillation, and in the methods of working up the products, in particular the recovery of AcOH by extraction with heavy oil obtained from the tar, are briefly discussed. Such developments, together with the extension of the field of application of the active C prepared from the solid residue, permit the process to compete successfully with the synthetic methods of preparing AcOH and MeOH. A. B. M.

Reactivity of coke. A. REIS (Chim. et Ind., 1935, 33, 1297—1306).—The methods that have been used for determining the reactivity of coke, and the results thereby obtained, are critically discussed. By modifying the carbonising conditions or by pretreatment of the coal the reactivity of the coke produced may be varied to suit its method of utilisation. A. B. M.

Improvement of the adsorption capacity of wood charcoal. H. HERBST (Kolloid-Beih., 1935, 12, 184—301).—Examination of many kinds of C shows that poorly adsorbent samples are also feebly wetted by H<sub>2</sub>O. These properties can be improved by removing tarry impurities. Experiments are described, which show the beneficial effects of removing such impurities by (1) extraction with solvents ( $C_6H_6$ , Et<sub>2</sub>O, CS<sub>2</sub>,  $C_5H_5N$ , COMe<sub>2</sub>), (2) distillation, (3) chemical treatment (e.g., heating in H<sub>2</sub> or Cl<sub>2</sub>, or molten Na<sub>2</sub>CO<sub>3</sub>, K<sub>2</sub>CO<sub>3</sub>, or Mg). The technical processes of activation of C are reviewed in the light of the above experiments, and the influence of the porosity of the adsorbent is discussed. When C is heated above 1100° a certain amount of graphite is formed and the adsorptive power decreases. E. S. H.

Electrical distillation of coal. C. FIGAI (Chim. et Ind., 1935, 34, 22–28).—Mainly a review. Neither a furnace with a vertical electrode nor a multiple-electrode furnace gave satisfactory carbonisation, which was obtained only in a furnace having two electrodes inclined at 60° to the vertical. In districts having cheap power the method may be useful to supplement existing methods. J. W.

**Extraction, cracking, and hydrogenation of coal.** E. MOEHRLE (Angew. Chem., 1935, **48**, 509—513).— Formation of liquid products from coal is discussed as a combination of decomp., extraction, and hydrogenation. For production of motor fuel by pressure hydrogenation, the coal-tar fraction of b.p. 260—320° is much superior to that of b.p. 230—260°. E. W. W.

Sampling and analysis of entrained matter in [combustible] gases. F. B. VARGA and R. H. NEWTON (Ind. Eng. Chem. [Anal.], 1935, 7, 240–242).—The procedure is designed for the determination of  $H_2O$ , volatile org. constituents, and constituents sol. and insol. in  $C_6H_6$  in coke-oven gases. The accuracy is 10%. Typical results are given. E. S. H.

Application of compressed gas as fuel for motor vehicles. ECKERT (Oesterr. Chem.-Ztg., 1935, 38, 108). —The present position is reviewed. Recent tests show that for the development of the same power compressed gas is 33% cheaper than petrol. A. B. M.

**Detonation of coal [dust].** A. A. VAN DER DUSSEN (Chem. Weekblad, 1934, **31**, 721-722).—Inert powders have the same influence on coal dust-air mixtures as on anthracene powder-air mixtures. Stone dust and fuller's earth have little inhibiting effect,  $(NH_4)_2SO_4$ has rather more, and NaHCO<sub>3</sub>, KCl, and K<sub>2</sub>SO<sub>4</sub> are very effective. The influence of the chemical nature of the powder (if any) is masked by that of physical characteristics, of which freedom from agglomeration is one of the most important. The tendency of a coal dust to explode appears to be related to the % of volatile matter, which suggests that the predistillation theory is valid. Al dust explosions may involve the formation of H<sub>2</sub>, or volatilisation of metal. H. F. G.

Prevention of gas explosions in coal mines. P. A. JONQUIERE (Chem. Weekblad, 1934, 31, 714—717). —Measures applicable to the prevention of explosion of gas mixtures which have already formed, either by dilution or admixture of an inhibitor, and to the arresting of the explosion wave, are discussed. PrBr (3%) will prevent the explosion of a 9.5% CH<sub>4</sub>-air mixture, but will promote the explosion of a non-explosive 4.5%CH<sub>4</sub>-air mixture. The explosion ranges of CH<sub>4</sub>-isoamyl bromide-air and CH<sub>4</sub>-POCl<sub>5</sub>-air mixtures are described ; 1.1% of POCl<sub>3</sub> in the latter inhibits explosion. The physiological effects of inhibitors are of great practical importance. H. F. G.

Prevention of gas explosions by powders. A. J. DIJKSMAN (Chem. Weekblad, 1934, 31, 718-721).— The blanketing action of inert powders (KCl,  $BaSO_4$ ) on  $CH_4$ -air explosions, as determined by the limiting quantity required for inhibition, is an approx. linear function of the particle size. The most explosive mixture is not necessarily that which requires the greatest quantity of powder. KCl, NaCl, and NaHCO<sub>3</sub> are very effective;  $Na_2SiF_6$ ,  $BaSO_4$ , and Al silicate are somewhat less effective. Both groups are more active than is stone dust. The theory of the action is obscure.

H. F. G.

**Explosion of ethylene.** H. I. WATERMAN (Chem. Weekblad, 1934, **31**, 713—714).— $C_2H_4$  heated to 350° at 175 kg./cm.<sup>2</sup> pressure exploded, the temp. rising to 500° and the pressure to about 500 kg./cm.<sup>2</sup> The products were C having marked adsorptive power,  $H_2$ , and a little CH<sub>4</sub>. The cause of the explosion is unknown. H. F. G.

Tendency to explode of various forms of acetylene. W. C. DE LIEFDE (Chem. Weekblad, 1934, 31, 711-713).—The influence of various factors, e.g., pressure, temp., and nature of container, on the explosion of  $C_2H_2$ , both alone and admixed with air, is discussed briefly. The solid polymeride produced at 300° has a heat of combustion of about 10 kg.-cal., and on account of its strong adsorptive properties may form the basis of a valuable explosive; it is stable and burns without explosion. Liquid  $C_2H_2$  may be handled safely at -80°, but is dangerously unstable at room temp. The solid (m.p. -81.5°, sublimation point -83.8°) is relatively stable and may, e.g., be compressed into blocks; its heat of decomp. is 48.6 kg.-cal.

H. F. G.

Generation of acetylene from calcium carbide. G. M. MASON, A. H. ANDERSEN, and R. S. JANE (Canad. Chem. Met., 1935, 19, 183-186, 192).-Recovery of the Ca content of  $CaC_2$  used for  $C_2H_2$  production implies generation without excess of  $H_2O$  by a dry process. If H<sub>2</sub>O is simply sprayed on lump CaC<sub>2</sub> the rate of evolution of C<sub>2</sub>H<sub>2</sub> decreases unless Ca(OH)<sub>2</sub> is continuously removed, e.g., by a rotary screen. If, however, CaC, is crushed to  $\gg \frac{1}{8}$ -in. mesh it can be successfully used by a spray process without screen. A ball mill forms a suitable mixer in this case. Dry generators on both principles are illustrated. In each case the correct operating temp. is 115° if condensation of H<sub>2</sub>O in pipe lines is to be avoided. Dry Ca(OH), is briquetted and re-used for CaO production. Both generators have been in full-scale operation for several years. C. I.

Chemical composition, properties, and methods of treatment of primary tars of Cheliabinsk brown coal. II. Composition of the light tar fraction and the stabilisation of "benzine" by hydrogenation. M. K. DJAKOVA, A. V. LOZOVOI, and S. I. TSCHERTKOVA (Compt. rend. Acad. Sci. U.R.S.S., 1935, 2, 399-403).-The "benzine" fraction, b.p. < 230°, contains 49.4% of phenols and carboxylic acids, 19.9% of aromatic hydrocarbons (PhMe-xylene fraction 3.8%), 11.4% of paraffins, 17.2% of unsaturated hydrocarbons, and 0.8% of N bases. Elimination of S from the neutral part of the "benzine" cannot be accomplished by the usual methods but is effected by treatment with H<sub>2</sub> at 400°/100 atm. for 2 hr. in presence of MoS<sub>3</sub> or CoS. Under these conditions the aromatic hydrocarbons are little changed whereas the unsaturated compounds are converted mainly into paraffins in presence of MoS<sub>3</sub>, or mainly into naphthenes in presence of CoS. 86-87% of the charge is recovered, which may be increased to 95%, the expenditure of  $H_2$  being 2-4% of the wt. of the material. The stabilised benzine contains 42-48% of aromatic hydrocarbons, 5-17% of unsaturated, 10-26% of naphthenic, and 26-30% of paraffin hydrocarbons; S is absent. H. W.

Low-temperature tar from brown coal. R. SCHMIDT (Brennstoff-Chem., 1935, 16, 241-247).--Progress during the period 1928-35 in knowledge of the composition and properties of brown-coal tar, in the methods of working it up and of utilising the products obtained, is reviewed. A. B. M.

High-pressure hydrogenation of low-temperature tar. III. Effects of catalysts and hydrogenating conditions. S. ANDO (J. Soc. Chem. Ind., Japan, 1935, 38, 196—199 в; cf. В., 1935, 259).—Results of experiments in a 0.6-litre autoclave, using 100 g. of tar, H. at an initial pressure of 100 atm. (i.e., 3.78 wt.-% of the tar charged), reaction temp. of 420-475°, and a catalyst consisting of  $NH_4$  molybdate,  $SnCl_2$ , or  $NiO + Fe_2O_3$ , supported on pumice, are recorded. The original tar contained 29.4 wt.-% of phenols, no gasoline, and 26.6% of kerosene. The product contained 6.0— 15.8% of phenols, the neutral oil consisting of 19-44% of gasoline, 32-38% of kerosene, and 25-45% of heavy oil; 1.4-11.6% of spirit was recovered from the gas by means of active C. The three catalysts showed no conspicuous differences in activity. The higher was the reaction temp. the greater was the yield of gaseous products, the greater the consumption of H<sub>2</sub>, and the greater the gasoline content of the neutral oil and the aromatic hydrocarbon content of the gasoline.

A. B. M.

Methods of examining mineral oils, especially the high-boiling components. J. C. VLUGTER, H. I. WATERMAN, and H. A. VAN WESTEN (J. Inst. Petroleum Tech., 1935, 21, 661-676).-Curves are given relating sp. refraction (R) (which is linearly related to H content) of different saturated hydrocarbon series, *i.e.*, paraffins and naphthenes, to mol. wt., so that for a given mol. wt., R of the corresponding paraffin and polycyclic naphthene can be obtained and the % of naphthene rings in a mixture can be calc. from R of the latter. The method does not hold when aromatic rings are present. The latter are determined by measuring the H<sub>2</sub> consumed during complete hydrogenation. A pure aromatic oil without side-chain consumes 7.8 wt.-% of H2. The difference in NH2Ph points of the hydrogenated and the original oils  $\infty$  the H absorbed. Curves are given showing the relation between NH<sub>2</sub>Ph point and R for mixtures of paraffins and naphthenes of varying mol. wt. The % of aromatic and naphthene rings and paraffinic side chains in an oil are therefore obtained from measurements of mol. wt., R, and NH<sub>2</sub>Ph point before and after hydrogenation. To determine mol. wt., the cryoscopic method in C<sub>10</sub>H<sub>8</sub> or C6H6 is used. Hydrogenation is carried out in a small rotating autoclave at 250-300°/100-120 kg. per cm.<sup>2</sup> in presence of Ni-kieselguhr. C. C.

Effect of the pressure in the process of cracking. M. S. NEMTZOV (J. Inst. Petroleum Tech., 1935, 21, 644-652).—A discussion with numerous references to literature (cf. B., 1934, 866).

Pyrolytic decomposition and hydrogenation of saturated hydrocarbons. R. FUSSTEIG (Chim. et Ind., 1935, 34, 3—9).—In an attempt to find a use for mineral oil by-products and natural gas three gases were submitted to cracking (15 atm.) and hydrogenation (200 atm.): (a) "Skellgas" ( $C_3H_8$  42,  $C_4H_{10}$  48,  $C_5H_{12}$  7%); (b) gas freed from gasoline; (c) dry gas. (b) and (c) gave poor results owing to the production of  $CH_4$ , but they are suitable for direct catalytic decomp. (a) was rather better. On cracking it gave at low temp.  $C_nH_{2n}$  and at higher temp. aromatic hydrocarbons (max. yields at 780° and 840° respectively). Hydrogenation decreased the yields of aromatic compounds and coke and increased those of liquid products. J. W.

Separation of paraffins and petroleum products from other oils by means of ethylene glycol monoacetate. K. B. EDWARDS and R. LACEY (J.S.C.I., 1935, 54, 253-254 T).-OH·[CH<sub>2</sub>]<sub>2</sub>·OAc is recommended as a selective solvent for the separation of the above from high-boiling aromatic and unsaturated hydrocarbons, the advantages being the recovery of both portions of the mixture in a state allowing further examination. The results of tests, using 8 vols. of solvent, show a considerable degree of accuracy in quant. separation of petroleum spindle oils from anthracene oils. The use of the solvent for the separation of low-temp. carbonisation tar oils into their constituents etc. is suggested.

Petroleum developments applicable to the protective coating industry. J. R. M. KLOTZ (Paint, Oil, and Chem. Rev., 1935, 97, No. 12, 16-17).--A review of recent developments in petroleum thinners. D, R. D.

Investigation of the cyclopentane hydrocarbons in naphtha-benzine by acetylation in presence of aluminium chloride. N. D. ZELINSKI and L. LEDER-PACKENDORFF (Annalen, 1935, 518, 260-274).-The paraffin and pentamethylene constituents of four samples of commercial benzine (I) have been investigated by fractionation, dehydrogenation of the cyclohexane to benzenoid hydrocarbons with Pt-C at 350°, removal of aromatic hydrocarbons with 7% oleum, and refractionation over Na. The physical consts. of the various fractions are tabulated. Treatment of the fraction b.p. 87-89.5° with AcCl-AlCl3 at 35° affords a ketone (36%), b.p. 66-90°/20 mm., giving a semicarbazone, m.p. 153-155°, and reduced (Pt-C-H2) to a ketone giving a semicarbazone, m.p. 157°. Similar treatment of a fraction of (I), b.p. 90-92°, affords, finally, a saturated ketone, b.p.  $65^{\circ}/16$  mm.  $(19 \cdot 2^{\circ})$  (semi-carbazone, m.p.  $151-152^{\circ}$ ), reduced (H<sub>2</sub>-Pt-C at 180-200°) to dimethyl*cyclo*pentane, b.p.  $137-139^{\circ}/755$ mm. A fraction of (I), b.p. 100-102°, similarly affords a saturated ketone, b.p. 85-86°/16 mm. (58.2%) (semicarbazone, m.p. 148-150°), reduced to diethylcyclopentane, and a fraction of (I), b.p. 101-102°, gives a ketone, b.p. 75-77°/15 mm. (20%), reduced to 1: 3-diethylcyclopentane. Similar treatment of a petroleum C6H14 fraction, b.p. 64-67°, gives a ketone, probably methylcyclopentyl Me ketone, since, after reduction, the hydrocarbon is separated into 1-methyl-2- and -3-ethylcyclopentane. The parent hydrocarbon present in (I) cannot be 3-methylpentane since this undergoes no cyclisation and affords no ketonic products with AcCl-AlCl<sub>3</sub>. Although contrary to the evidence of the physical consts. of the original fractions, it is suggested that the large yields of cyclic ketones arise from the presence of a considerable amount of methylcyclopentane in (I), since the data shows that cyclisation of large amounts of paraffin hydrocarbons is unlikely. J. W. B.

Procedure for classification of hydrocarbons. S. P. MULLIKEN and R. L. WAKEMAN (Ind. Eng. Chem. [Anal.], 1935, 7, 275-278).—A method for the detection of hydrocarbon type dependent on a knowledge of its miscibility with MeNO<sub>2</sub>, NH<sub>2</sub>Ph, and CH<sub>2</sub>Ph·OH, degree of unsaturation (Br), and its m.p., b.p., and d is outlined. F. N. W.

Use of gas oils in explosion motors. L. A. BORRUAT (An. Soc. cient. Santa Fe, 1934, 6, 4-16; Chem. Zentr., 1935, i, 1320-1321).—Tests of various gas oils in tractor motors with heavy-oil vaporisers are described.  $\lt$  85-90% of a suitable oil should distil at  $\lt$  320°. H. J. E.

**Prevention of explosions in pipes.** W. R. VAN WIJK (Chem. Weekblad, 1934, **31**, 726–728).—Means of preventing the propagation of an explosion wave along the pipes which interconnect the vapour spaces in  $C_6H_6$ storage tanks, in the event of fire in one tank, are discussed, with special reference to the use of an inhibitor such as KCl in conjunction with Raschig rings in the intermediate arrestors. H. F. G.

Apparatus for measuring the b.p. of lubricating oils and other compounds of high mol. wt. at reduced pressures. S. T. SCHICKTANZ (J. Res. Nat. Bur. Stand., 1935, 14, 685—692).—The apparatus is illustrated and examples of its use are described. The temp. are determined by means of three thermocouples located at different heights in the vapour column so that the differences in their readings afford an indication of the purity of the sample. A. R. P.

Boundary friction of oxidised lubricating oils. E. R. REDGROVE (J. Inst. Petroleum Tech., 1935, 21, 612-633).—A detailed account of work already noted (B., 1935, 484).

 $p_{\rm H}$  control of rotary drilling fluids. D. N. MEHTA and S. K. K. JATKAR (J. Indian Inst. Sci., 1935, 18A, 101–107).—The effects of adding acid, alkali, and tannin extract have been determined. C. W. G.

Boiler practice. Oils and plastics.—See I. cycloPentane hydrocarbons in naphthabenzine.—See II. Adsorbents.—See VII. Control of kiln atm.—See VIII. Explosions of solids.—See XXII.

#### PATENTS.

Plant for distillation of coal and other carbonaceous substances. Low TEMPERATURE CARBONISATION, LTD., and W. A. BRISTOW (B.P. 431,772, 14,10.33).— A vertical or inclined retort having a narrow crosssectional dimension and provided with heating flues adapted for low-, medium-, or high-temp. distillation has branched inlet and outlet fittings, one branch of each being fitted with a drum. These drums have a cylindrical recess in one side, which is co-axial with the drum and large enough to take containers of dimensions just small enough to pass through the retorts. Coal etc. can be carbonised in containers by utilising these attachments, or loose by utilising the other branches. D. M. M. **Regenerative coke oven.** C. Otro (U.S.P. 1,977,201, 16.10.34. Appl., 11.1.30. Ger., 4.5.29).—In an oven with parallel heating walls, each containing two series of vertical combustion flues, a flue of one series being adjacent to a flue of the other and communicating with it at the top, and the partition wall having a communicating passage (P) at the bottom, improved control is obtained by sliding stones at the foot end of the flues for regulating the inlet openings of gas and air ducts. P is at a greater height than these openings, being adjustable by another sliding member. D. M. M.

Destructive hydrogenation of carbonaceous materials. INTERNAT. HYDROGENATION PATENTS Co., LTD., Assees. of I. G. FARBENIND. A.-G. (B.P. 430,069, 2.10.34. Ger., 11.11.33).—The walls of the vessel in which the vapours and gaseous products are separated from the liquid (and possibly solid) products are continuously flushed with a liquid medium to prevent the formation of solid deposits thereon. This is preferably effected by cooling the walls, *e.g.*, by means of a pipe coil, below the temp. in the interior of the separator so that part of the vapour product is condensed thereon. A. B. M.

Treatment of carbonaceous materials with hydrogenating gases. H. E. POTTS. From INTERNAT. HYDROGENATION PATENTS Co., LTD. (B.P. 431,795, 18.1.34).—The carbonaceous materials and hydrogenating gases are passed through a cylindrical reaction vessel for treatment in the liquid phase, then into a vessel for separation of liquid products, and the remaining gases and vapours are passed into a second reaction vessel containing stationary catalyst in which the hightemp. gas-phase treatment is carried out. D. M. M.

Purification of hydrogen for destructive hydrogenation. H. E. POTTS. From INTERNAT. HYDROGEN-ATION PATENTS CO., LTD. (B.P. 431,970, 7.7.34).—The  $H_2$ , after preliminary purification by low-temp. cooling, washing, etc., is freed from residual foreign gases, e.g., CO and N<sub>2</sub>, by scrubbing at low temp. and/or pressure with liquefied hydrocarbons ordinarily gaseous; these are subsequently removed by oil-washing. When the process is a cyclic one the gases are periodically subjected to this treatment to remove N<sub>2</sub> and CO added during the reactions. D. M. M.

Separation of ammonia and hydrogen sulphide from gases. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 430,007, 4.12.33).—The NH<sub>3</sub> is removed from the gas as  $(NH_4)_2SO_4$  by washing with  $H_2SO_4$ . Then the  $H_2S$  is removed by a suitable scrubbing process, *e.g.*, by absorption in  $NH_2$ ·C<sub>2</sub>H<sub>4</sub>·OH. The H<sub>2</sub>S is recovered from the spent scrubbing agent, burned, and converted into  $H_2SO_4$ , which is used for the removal of NH<sub>3</sub> in the first stage. A. B. M.

[Agents for] breaking petroleum emulsions. M. DE GROOTE, B. KEISER, and (A) A. F. WIRTEL, (B) W. C. ADAMS, ASSTS. to TRETOLITE CO. (U.S.P. 1,975,839 and 1,976,602, 9.10.34. Appl., [A] 5.2.34, [B] 3.4.33).— The emulsion is treated (A) with the product of sulphonation of anthracene oil in presence of a fatty acid (oleic), aldehyde, ketone, or lower alcohol ( $\gg C_{12}$ ), (B) with a dibasic acid ester of a OH-fatty acid or its derivatives,

e.g., the product of interaction of triricinolein with  $o-C_6H_4(CO)_2O$ . H. A. P.

Breaking of petroleum emulsions. (A) M. DE GROOTE and (A-c) C. H. M. ROBERTS, Assrs. to TRETO-LITE Co. (U.S.P. 1,977,048, 1,977,089, and 1,977,146, 16.10.34. Appl., [A, B] 7.12.33, [C] 3.8.33).-Petroleum emulsions, e.g., from wells or storage tanks, are broken by treatment with a substituted polyhydric alcohol partly or completely esterified so that one or more OH is replaced by (A, B) one or more monobasic, detergentforming carboxylic acid residues (oleic, naphthenic, abietic) and/or one or more OH is replaced by an aliphatic polybasic acid residue, e.g.,  $H_2C_2O_4$ ; (c) one or more polybasic carboxylic acid residues, e.g., phthalic acid, and also the alcohol and/or the acid are chemically united to ricinoleic acid or its equiv. C. C.

Purification of hydrocarbon mixtures with removal of paraffin wax. EDELEANU GES. M.B.H. (B.P. 432,168, 24.1.35. Ger., 24.1. and 23.4.34).-Hydrocarbon oils are treated with a mixture of 15-35 vols. of liquid SO2 and 85-65 vols. of liquid aromatic hydrocarbons or other low-boiling org. solvents completely miscible with  $SO_2$ , e.g., CHCl<sub>3</sub>, Et<sub>2</sub>O. The mixture is cooled to  $-20^\circ$  to  $-30^{\circ}$  by evaporation of part of the SO<sub>2</sub>, and the paraffin wax which separates is removed. Lubricating oil of pour point approx. comparable to the filtration temp. is obtained. The oils may be refined before or after dewaxing by the above process by extraction with a solvent mixture similar to the above but containing an excess of SO<sub>2</sub>, without removing the solvent between the two treatments. C. C.

Stabilisation of liquid hydrocarbons. E. I. DU PONT DE NEMOURS & Co. (B.P. 432,121, 22.1.34. U.S., 20.1.33).—Liquid hydrocarbons (excluding non-benzenoid polymerisation products of  $C_2H_2$ ), e.g., cracked gasoline, crude benzol, Diesel oil, transformer oil, are stabilised against the formation of gummy or resinous products and sludge by the addition of 0.001-1.0% of 1:2-, 1:4-, 1:5-, 1:7-, or  $2:3-C_{10}H_6(OH)_2$  or mixtures of these.

#### C. C.

Manufacture of low-boiling petroleum distillates. A. M. McAFEE, Assr. to GULF REFINING Co. (U.S.P. 1,976,507, 9.10.34. Appl., 15.1.32).—High-boiling petroleum hydrocarbons, preferably containing a high % of naphthenes, are heated to just < cracking temp. (315—400°) and > 1% (2%) of AlCl<sub>3</sub> is added in the form of a heated mixture containing 80% of AlCl<sub>3</sub> and 20% of oil. The mixture passes to a reaction zone at 75—300 lb./sq. in. in which an approx. const. level of oil is maintained. Aq. NaOH is sprayed into the distillate vapours from this zone before condensation. Coke is removed by introducing cool oil until the temp. is  $< 100^\circ$ , followed by H<sub>2</sub>O or aq. alkali. C. C.

Hydrolysis of acid sludge [from refining of hydrocarbon oils]. H. W. THOMPSON and J. T. RUTHERFORD, ASSTS. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,975,131, 2.10.34. Appl., 27.5.30).—Acid sludge is hydrolysed by mixing with  $H_2O$  and steam and digesting in a brick-lined retort at a temp. sufficient for hydrolysis and > that at which coke is formed from the tar. Acid is continuously removed (by siphon) from the bottom of the retort while tar and coke are continuously withdrawn from the interface between tar and acid layers. Separated acid and tar may be further digested. C. C.

Self-compensated motor fuel. C. ELLIS, Assr. to ELLIS-FOSTER CO. (U.S.P. 1,976,696, 9,10.34. Appl., 29.4.24. Renewed 2.7.32).—Petroleum oil, e.g., gas oil, is cracked at  $370-480^{\circ}/250-350$  lb., a small proportion of air being introduced with the oil. The vapours are withdrawn from the cracking zone, oxidised by air or O<sub>2</sub> at  $120-245^{\circ}$  in presence of a catalyst, e.g., Fe coated with V oxide, and passed to condensers. The crude product is distilled to obtain gasoline. The high knockrating of the oxidised and cyclised compounds permits the inclusion of a higher proportion of heavy ends than is usual. C. C.

Manufacture of fuel composition. L. F. Hovr and W. A. SMITH (Assee.) (U.S.P. 1,975,755, 2.10.34. Appl., 23.5.31).—S-free gasoline, pretreated with CuSiO<sub>3</sub>, is protected against colour- and gum-formation respectively by addition of 0.01-0.06% of quinhydrone and 0.01-0.06% of an aromatic primary amine containing an additional NH<sub>2</sub> or CO<sub>2</sub>H, *e.g.*, C<sub>6</sub>H<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub>, *o*-NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·CO<sub>2</sub>H. [A. W. B.

Production of oils from liquids or meltable solid carbonaceous materials composed of constituents having different solubilities in light hydrocarbon solvents. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 430,080, 6.12.33).—The initial material (A) is treated with a light hydrocarbon solvent, e.g., C3H8, under conditions of temp. and pressure at which the solvent would exist in the vapour phase in absence of A, and either simultaneously or subsequently choosing such conditions that two distinct liquid (or liquid and solid) phases are formed. The initial temp. is preferably > the crit. temp. of the solvent. A high yield of the desired product is thereby extracted from A. The process may be used to extract high-quality lubricating oil from petroleum residues etc. A. B. M.

(A) Dewaxing system. R. N. GILES. (B) Propane dewaxing process. W. J. McGill and C. E. ADAMS, (A, B) Assrs. to STANDARD OIL CO. (U.S.P. 1,977,054-5, 16.10.34. Appl., [A] 27.12.32, [B] 23.1.33).-(A) Petroleum lubricating oils are diluted with C3H8 and cooled in a series of stages, shock chilling being prevented by condensing vapours from the first stage in the second stage to equalise pressure and temp. The cooled slurry from the first stage is transferred to the second, where it is recombined with condensed C3H8 and further cooled by again evaporating  $C_3H_8$ . (B) When equilibrium between the two stages is reached  $C_3H_8$  is forced by a compressor from the first to the second, whereby the temp. of the first is further reduced while that of the second is raised. The solution of oil in C3H8 is then introduced into the second stage, which is then cooled by removing C<sub>3</sub>H<sub>8</sub> vapours. C. C.

Treatment of mineral lubricating oils. S. H. DIGGS and J. M. PAGE, JUN., ASSTS. to STANDARD OIL CO. (U.S.P. 1,976,544, 9.10.34. Appl., 6.5.32).—The  $\eta$  index and resistance to sludge formation of petroleum fractions are improved by extraction with a halogenated ether  $[\beta\beta' - O(C_2H_4Cl)_2]$  mixed (optionally) with a ketone (COMe<sub>2</sub>) or with COMe<sub>2</sub> + PhNO<sub>2</sub> (at room temp.)

which removes naphthenes, and treatment of the residue with 80–98 (93)%  $H_2SO_4$  (1–1.5 lb./gal. of original oil). H. A. P.

Oxidation of hydrocarbons.—See III. Road surfacing.—See IX. Emulsifying waxes etc.— See XII.

## III.—ORGANIC INTERMEDIATES.

Analytical properties of commercial sulphated alcohols. F. M. BIFFEN and F. D. SNELL (Ind. Eng. Chem. [Anal.], 1935, 7, 234–237).—Tentative qual. methods for the analysis of commercial alkyl sulphates are suggested. F. N. W.

Analysis of sulphonated aliphatic alcohols and similar condensation products. E. SWINDELLS (Dyer, 1935, 73, 120—121).—Esters in which the SO<sub>3</sub>H is attached to O are determined by the Herbig method. Condensation products (Adulcinol, Igepon) in which the SO<sub>3</sub>H is attached partly or wholly to C are incompletely decomposed by acid. The undecomposed portion can be extracted by BuOH. Analytical procedure is described. CH. ABS. (p)

Determination of water in alcohol-ketone mixtures. Anon. (Iva, 1935, No. 1, 2–5).—The accuracy of the  $Mg_3N_2$  method (Dietrich and Conrad, B., 1931, 708) was confirmed. CH. Abs. (e)

Distillation and rectification of dilute solutions of volatile substances. Efficiency of laboratory columns. P. JAULMES (Chim. et Ind., 1935, 33, 1321—1325; cf. B., 1935, 609).—0.1N-AcOH was distilled with various types of laboratory column, using a simple apparatus for regulating the reflux. The ratio of [AcOH] in the vapours leaving and entering the column was plotted as a function of the proportion of liquid refluxed. From the curves so obtained the relative efficiencies of the columns can be estimated. A. B. M.

Determination of propionic and butyric acids in acetic acid. A. V. VINOGRADOV and E. A. OSTROU-MOVA (J. Appl. Chem. Russ., 1935, 8, 378–382).—The acids are converted into Na salts, which are fractionally titrated with N-HCl in presence of  $\text{Et}_2O$ , the  $\text{Et}_2O$ extracts are titrated with N-NaOH (phenolphthalein), and  $\text{FeCl}_3$  is added to the aq. layers, when a yellow ppt. indicates  $\text{PrCO}_2\text{Na}$ , a brown solution yielding a yellow ppt. when heated indicates  $\text{EtCO}_2\text{Na}$ , and a brown ppt. AcOH. The approx. contents of the acids are determined by comparison with known solutions. R. T.

Separation of cresols [from coal tar]. S. MASAI (J. Soc. Chem. Ind., Japan, 1935, 38, 217 в).—The crude phenols separated from middle oil (b.p. 175—235°) are distilled in vac. and the *o*-cresol is separated by three vac. fractionations of the fraction b.p. 186—187°. The fraction 200—203° is heated with  $H_2C_2O_4$ , whereby the *p*-cresol is esterified and separated from the *m*-cresol.

Mechanism of salicylaldehyde reaction for detection of fusel oils and ketones. K. TAÜFEL, H. THALER, and O. BAUER (Z. Unters. Lebensm., 1935, 69, 401-405; cf. B., 1932, 515).—The reaction is shown to be sp. for dialkyl ketones. *sec.*-Alcohols give the reaction only after prolonged heating at 100°. The effect of  $[H_2SO_4]$  on this and on the Komarovsky reaction, and the mechanism of the latter, are discussed. E. C. S.

Pb glycerate.—See IX. BuOH-COMe<sub>2</sub> fermentation. Lactic acid. Citric acid.—See XVIII.

#### PATENTS.

Production of olefine hydrocarbons and derivatives thereof. H. B. HASS and P. E. WESTON, ASSTS. to PURDUE RES. FOUNDATION (U.S.P. 1,975,456, 2.10.34. Appl., 8.1.32).—n-Alkyl chlorides or bromides having  $C_{4-5}$  (BuCl,  $C_5H_{11}$ Cl) are subjected to pyrolysis at 450— 700° in absence of a catalyst, and the olefine formed is hydrated to alcohol by known methods. Apparatus is claimed. H. A. P.

Oxidation of hydrocarbons. W. K. LEWIS and P. K. FROLICH, ASSTS. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,976,790, 16.10.34. Appl., 12.5.27).— Gaseous or low-boiling liquid hydrocarbons, e.g., natural gas, are oxidised to aldehydes, acids, and (mainly) alcohols by passing mixtures containing  $\geq 15\%$  O<sub>2</sub> over a catalyst (carbide-forming metals or their oxides, silicates; e.g., Ag, Pt, steel, clay), at 4000—40,900 c.c. per hr. and  $\geq 100$  (1000—3500) lb./sq. in. at 260—600°. A. W. B.

Manufacture of ethylene oxide. Soc. FRANÇ. DE CATALYSE GÉN. (B.P. 431,966, 9.7.34. Fr., 10.7.33).—  $C_2H_4$ ,  $O_2$  (air), and an inert gas (CO<sub>2</sub>;  $\lt$  50 vol.-%) are passed over Ag, Ag-Au, or Ag-Cu catalysts at 300— 400°/> 50 atm. H. A. P.

Manufacture of chloroform. A. A. LEVINE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,975,727, 2.10.34. Appl., 16.11.33).—A mixture of MeCl and  $C_2H_2Cl_2$  is chlorinated at 400—650°. Preferably a mixture of MeCl,  $C_2H_2Cl_2$ , and  $Cl_2$  in the vol.-ratio 2:2:1-4:3:1 is heated at 400—500° until chlorination is complete, cooled to 200°, 1—1.5 vols. of  $Cl_2$  are added, and the mixture is reheated to 400—500°. The product contains 15—30% CHCl<sub>2</sub>. H. A. P.

Manufacture of halofluoro-hydrocarbons. A. L. HENNE, Assr. to GEN. MOTORS CORP. (U.S.P. 1,978,840, 30.10.34. Appl., 30.1.31. Renewed 9.9.33).—SbF<sub>3</sub>Cl<sub>2</sub>, prepared by treating SbF<sub>3</sub> with Cl<sub>2</sub> (at > 70°) in a bal, mill, is claimed as an active fluorinating agent not requiring a catalyst. Examples include :  $C_2Cl_6 \rightarrow C_2F_4Cl_2 +$  $C_2F_3Cl_3 + C_2F_2Cl_4$ ; CCl<sub>4</sub>  $\rightarrow$  CF<sub>2</sub>Cl<sub>2</sub>. A. W. B.

Preparation of [ethyl] alcohol denaturant. L. J. FIGG, JUN., Assr. to EASTMAN KODAK Co. (U.S.P. 1,975,091, 2.10.34. Appl., 11.8.32).—A mixture, largely of b.p.  $< 85^{\circ}$ , comprising fractions of acid, oil of b.p. 75—160°, washed alcohol oil of b.p. 75—160°, and allyl alcohol of b.p. 66—91° from distillation of crude pyroligneous acid, mixed with a solvent, *e.g.*, COMe<sub>2</sub>, EtOH, Pr<sup>g</sup>OH, is used as a denaturant for EtOH. A. W. B.

Denaturant for ethyl alcohol. L. J. FIGG, JUN., Assr. to EASTMAN KODAK CO. (U.S.P. [A] 1,975,090 and [B] 1,975,092, 2.10.34. Appl., [A] 11.9.31, [B] 12.12.31). —Offensive-smelling non-toxic oils, b.p. 75—190°, obtained on dilution of the crude MeOH fraction, b.p.  $41-230^\circ$ , from the destructive distillation of hardwood and washing free from MeOH, are added  $(0\cdot 1-0\cdot 2^\circ)$ to EtOH to give an unpalatable product. It is preferable

to use a common solvent, (A)  $COMe_2$ , (B) liquid hydrocarbons, e.g.,  $C_6H_6$ , gasoline, kerosene, or mixtures of these. A. W. B.

Catalytic dehydrogenation of primary alcohols. W. A. LAZIER, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,975,853, 9.10.34. Appl., 11.4.31).—The use of Cu mixed with difficultly reducible oxides, *e.g.*, MnO, MgO, ZnO, as a catalyst for dehydrogenation of alcohols (EtOH) to yield esters, *e.g.*, EtOAc, at 200—500°/50—500 atm., is claimed. An example gives Cu 77, MnO 15, and MgO 8 mols. A, W. B.

Manufacture of primary phosphates of glycocol [glycine] alkyl esters and N-alkyl derivatives thereof. A. CARPMAEL. From SCHERING-KAHLBAUM A.-G. (B.P. 432,155, 18.9.34).—The following salts are prepared by mixing the components alone or in org. solvents (MeOH, EtOH, EtOH-Et<sub>2</sub>O): NH<sub>2</sub>·CH<sub>2</sub>·CO<sub>2</sub>Et,H<sub>3</sub>PO<sub>4</sub>, m.p. 134—135°, and NHMe·CH<sub>2</sub>·CO<sub>2</sub>Me,H<sub>3</sub>PO<sub>4</sub>. They have tonic properties. H. A. P.

Manufacture of acetic acid. W. O. WALKER and U. KOPSCH, ASSTS. to A. O. SMITH CORP. (U.S.P. 1,976,756, 16.10.34. Appl., 7.5.32).—MeCHO, mixed with an excess of air or other gas containing  $O_2$ , together with inert gases if desired, is passed into a solution (2%) of  $Co(OAc)_3$  in AcOH (at 50—80°), the partial pressure of AcOH above the liquor being maintained by temp. adjustment so as to remove the product as quickly as it is formed. Apparatus is described. A. W. B.

Separation of optically active glutamic acid without racemisation. S. KANAO, ASST. to KABUŚHIKI KAISHA SUZUKI SHOTEN (U.S.P. 1,976,997, 16.10.34. Appl., 6.4.33. Jap., 12.4.32).—Solutions of mineral acid, e.g., HCl, salts of d-glutamic acid (I) are treated with an N-alkylglycine (II), e.g., sarcosine, betaine. (I) is pptd. without reduction of rotatory power. (II) is easily recoverable. A. W. B.

**Preparation of thiourea.** W. SCHULENBURG, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,977,210, 16.10.34. Appl., 29.8.30. Ger., 14.9.29).—An aq. solution of a cyanamide, e.g., Ca(NH·CN)<sub>2</sub>, is treated at  $< 40^{\circ}$ (< room temp.) with H<sub>2</sub>S in presence of CO<sub>2</sub>. Yields of > 87% and elimination of hazard are claimed.

#### A. W. B.

Manufacture of alkylated cyclic amidines. Soc. CHEM. IND. IN BASLE (B.P. 431,808, 16.5.34. Switz., 18.5.33).—Higher fatty glycerides are condensed with o- or peri-diamines (or the corresponding nitroamines in presence of a reducing agent), under hydrogenating conditions if desired. Eg., Cacao-butter is heated with  $o-C_6H_4(NH_2)_2$  at 200—220° or  $1:2-C_{10}H_6(NH_2)_2$  at 240—245° in N<sub>2</sub> or with  $o-NH_2\cdot C_6H_4\cdot NO_2$  and Ni in H<sub>2</sub> at 90—100° and 200—220°, linseed or olive oil is heated with  $o-C_6H_4(NH_2)_2$  and Al at 200—240°, or hydrogenated fish oil is heated with  $1:8-C_{10}H_6(NH_2)_2$  at 240°.

H. A. P.

Manufacture and application of new reagents [for the carbonyl group]. LABORATOIRES FRANÇ. CHIMIOTHÉRAPIE, A. GIRARD, and G. SANDULESCO (B.P. 431,165, 1.3.34. Ger., 27.3.33).—Quaternary  $NH_4$ hydrazides are prepared by interaction of  $N_2H_4$  with a betaine ester, or with an  $\alpha$ -halageno-fatty acid, followed by a *tert.*-amine. They give H<sub>2</sub>O-sol., Et<sub>2</sub>O-insol. hydrazones with ketonic sexual hormones. E.g.,  $N_2H_4$ is added to the product of interaction of NMe<sub>3</sub> and CH<sub>2</sub>Cl-CO<sub>2</sub>Et in abs. EtOH to give hydrazidoacettrimethylammonium chloride; similarly, with C<sub>5</sub>H<sub>5</sub>N hydrazidoacetpyridinium chloride is formed. H. A. P.

Manufacture of carbon disulphide reaction products. D. H. POWERS, ASST. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,975,588, 2.10.34. Appl., 27.6.29).—Products of indefinite composition for use as acid-corrosion inhibitors and vulcanisation accelerators are prepared by interaction at 40—70° of CS<sub>2</sub>, MeCHO, and primary amines, e.g., NH<sub>2</sub>Ph, NH<sub>2</sub>Bu. H. A. P.

Production of 1:3:5-trimethylbenzene [mesitylene]. H. DOHSE and C. SCHUSTER, ASSTS. to I. G. FARB-ENIND. A.-G. (U.S.P. 1,977,178, 16.10.34. Appl., 21.2.33. Ger., 10.3.32).—Mesitylene is obtained in 40—60% yield by heating COMe<sub>2</sub> to  $250-450^{\circ}$  (330—370°)/50—300 (150—250) atm. over a natural hydrate of Al<sub>2</sub>O<sub>3</sub>, e.g., bauxite, boehmite, previously heated at 300—1000° (600—900°) for about 1 hr. until it is at least partly dehydrated. A. W. B.

Manufacture of polyphenyls. W. H. WILLIAMS, Assr. to Dow CHEM. Co. (U.S.P. 1,976,468, 9.10.34. Appl., 2.1.34).—A mixture of  $C_6H_6$  and  $Ph_2$  ( $\geq 20$  wt.-%) containing S or a volatile S compound ( $CS_2 \equiv 0.15$ — 0.5% S) is passed over an electrically heated C surface at 650—950°. After removal of  $C_6H_6$  and  $Ph_2$ , which are recirculated, the product has b.p. 121—250°/6 mm. and is thermally stable at 450°. H. A. P.

Manufacture of perylene. F. KUHRMANN, Assr. to GEN. ANILINE WORKS, INC. (U.S.P. 1,977,768, 23.10.34. Appl., 22.9.33. Ger., 8.6.32).—2:2'-Dinaphthyl is heated with AlCl<sub>3</sub> at 130—160°, with or without a solvent such as PhCl or  $C_6H_4$ MeCl. Yields of 40—50% are claimed. A. W. B.

Production of diamino-alcohols of aromatic series. Chem. Fabr. Dr. J. Wiernik & Co. A.-G. (B.P. 431,786 and Addn. B.P. 431,848, [A, B] 12.1.34. Ger., [A] 12.1. and 1.2.33).-(A) A primary or sec.-3amino- $\alpha$ -aryl-ethanol (-propanol, etc.) is condensed with a dialkylaminoalkyl halide or arylsulphonate; alternatively, the normal NH2-alcohol syntheses are carried out using as bases the as-dialkylalkylenediamines (I). 1-OH·CHPh·CHMe·NHMe is heated with E.q., NEt<sub>2</sub>·[CH<sub>2</sub>]<sub>2</sub>Cl in C<sub>6</sub>H<sub>6</sub> to give 1-α-phenyl-β-methyldiethylaminoethylaminopropan-a-ol (II), b.p. 140°/0.5-1 mm.  $(+2HI, \text{m.p. } 166-167^\circ; +2H_3PO_4, \text{m.p. } 167-168^\circ);$ dl- $\alpha$ -phenyl- $\beta$ -diethylaminoethylaminopropan- $\alpha$ -ol, b.p. 140°/0·5-1 mm. (+ 2HI, m.p. 176-178°), is similarly produced using p-NEt<sub>2</sub>·[CH<sub>2</sub>]<sub>2</sub>·OSO<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>Me,HCl;  $\alpha$ -phenyl- $\beta$ -(benzyl- $\beta$ '-diethylaminoethylamino) propan- $\alpha$ -ol (+2 HCl) and  $\alpha$ -p-methoxyphenyl- $\beta$ -(methyl- $\beta$ -diethyl-aminoethylamino)ethanol, b.p. 180°/3 mm. (+2 HCl)are similarly prepared. The dl-form of (II), b.p. 140-141°/1.5-2mm.(+2HCl), is also obtained by interaction of OMe CHPh CHMeBr with NHMe [CH2]2 NEt2 (III) and hydrolysis (HBr) of the Me ether produced, and by reduction (H<sub>2</sub>; Pd-BaSO<sub>4</sub>) of the corresponding ketone, b.p. 132-134°/1 mm. [from COPh CHMeBr and (III) in Et<sub>2</sub>O]. (B) A compound COArCOMe or OH·CHAr·COMe is condensed with (I), and the product reduced. E.g., COPh COMe, NH, [CH2]2 NEt2 (IV), Zn, and SO<sub>2</sub> interact in aq. MeOH to give dl- $\alpha$ -phenyl- $\beta$ -( $\beta$ '-diethylaminoethylamino)propan- $\alpha$ -ol, m.p. 176—178° (oxalate, m.p. 180—182°), also formed by condensation of (IV) with OH-CHPh-COMe in Et<sub>2</sub>O and reduction (H<sub>2</sub>-Pd) of the product. H.A. P.

Manufacture of polynuclear phenols. E. I. DU PONT DE NEMOURS & Co. (B.P. 431,945, 18.1.34. U.S., 19.1.33).—The condensation of PhOH or its homologues with aldehydes or ketones in acid medium (H<sub>2</sub>SO<sub>4</sub>), *e.g.*, the production of 4:4'-CMe<sub>2</sub>(C<sub>6</sub>H<sub>4</sub>·OH)<sub>2</sub>, is carried out in presence of an acid-sol. B compound (H<sub>3</sub>BO<sub>3</sub>; 0.5— $5\cdot0$  wt.-% of PhOH). H. A. P.

Production of substituted phenols. RöHM & HAAS Co., Assees. of J. B. NIEDERL (B.P. 431,487, 14.3.34. U.S., 28.3.33).—Diisobutylene (I) is condensed with polyhydric phenols or their ethers having a free o- or p-position with the aid of a cationoid condensing agent (H<sub>2</sub>SO<sub>4</sub>). E.g., H<sub>2</sub>SO<sub>4</sub> is added to (I) in abs. AcOH at  $O-15^{\circ}$ , followed by  $m-C_6H_4(OH)_2$  in AcOH at  $< 15^{\circ}$ , with good stirring; the product is  $\alpha x \gamma \gamma$ -tetramethylbutylresorcinol, m.p. 101—102°.  $\alpha x \gamma \gamma$ -tetramethylbutylresorcinol, m.p. 101—102°.  $\alpha x \gamma \gamma$ -tetramethylbutylresorcinol, m.p. 101—225°/7 mm., -pyrogallol, m.p. 103°, and -guaiacol, b.p. 138—144°/4 mm., are similarly prepared. They possess high bactericidal properties.

Preparation of salts of pyrocatechol borate. I. WILLIAMS and A. M. NEAL, ASSTS. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,975,890, 9.10.34. Appl., 17.6.32).— $o-C_6H_4(OH)_2$  (2 mols.),  $H_3BO_3$  (1 mol.), and the appropriate base are fused together. Di-o-tolylguanidine, m.p. 167°, diphenylguanidine, m.p. 230°, dibutylamine, m.p. 163°,  $\beta$ -naphthylamine, m.p. 206°, piperidine, m.p. 255°, m-tolylenediamine, m.p. 227°, butylamine, m.p. 232°, tributylamine, m.p. 134°, and 2-methylpiperidine dipyrocatechol borate, m.p. 238°, are described. H. A. P.

Phenolic derivatives of diaryl sulphides. [Germicides.] T. B. JOHNSON, ASST. to SHARP & DOHME, INC. (U.S.P. 1,976,732, 16.10.34. Appl., 10.3.28).—Aromatic diazonium salts (I) are coupled with arylthiols (II), (I) and/or (II) containing an alkoxy-group or groups, and the intermediate diazo-thiols are decomposed followed by dealkylation (by HBr in AcOH). The products are obtained in a high state of purity. Examples include : p-methoxydiphenyl, m.p.  $50-51^{\circ} \rightarrow p$ -OH·C<sub>6</sub>H<sub>4</sub>·SPh, o-methoxydiphenyl, b.p.  $151-153^{\circ}/3$  mm.  $\rightarrow o$ -OH·C<sub>6</sub>H<sub>4</sub>·SPh, b.p.  $138-140^{\circ}/4$  mm., p-tolyl pmethoxyphenyl, m.p.  $45-46^{\circ}$ ,  $\rightarrow$  p-tolyl p-hydroxyphenyl, m.p.  $67-68^{\circ}$ , sulphide. A. W. B.

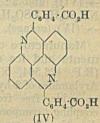
Preparation of ketone-amine [condensation products]. L. MEUSER, ASST. to NAUGATUCK CHEM. Co. (U.S.P. 1,975,167, 2.10.34. Appl., 6.5.32).—The ketone (COMe<sub>2</sub>) and (sec.-)amine (NHPh<sub>2</sub>,  $\beta$ -NHPh·C<sub>10</sub>H<sub>7</sub>) are heated in an autoclave at about 250° in presence of the bromide or iodide of a heavy metal other than Pb (FeI<sub>2</sub>,4H<sub>2</sub>O, SnI<sub>2</sub>). H. A. P.

[Nickel] salts of keto-aromatic acids. L. C. DANIELS and A. O. JAEGER, ASSIS. to AMER. CYANAMID & CHEM. CORP. (U.S.P. 1,977,047, 16.10.34. Appl., 9.12.30). —Acids of the type R·CO·R'·CO<sub>2</sub>H in which R is an aliphatic or mononuclear aromatic and R' an aromatic or, when R is aromatic, an aliphatic residue, e.g.,  $Bzr[CH_2]_2 \cdot CO_2H$ ,  $C_6H_4Bz \cdot CO_2H$ , are treated in solution [in presence of an org. base, e.g.,  $C_5H_5N$ ,  $N(C_2H_4 \cdot OH)_3$ ] with a solution of a  $H_2O$ -sol. Ni salt. The Ni salts thus obtained are of use in plastic and coating compositions. A. W. B.

Purification and hydrogenation of pyridines, I. GUBELMANN and C. O. HENKE, ASSTS. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,975,843, 9.10.34. Appl., 3.7.31).—C<sub>5</sub>H<sub>5</sub>N or C<sub>5</sub>H<sub>5</sub>N bases is/are converted into a H<sub>2</sub>O-sol. salt (salts) with a non-volatile acid (H<sub>2</sub>SO<sub>4</sub>) in H<sub>2</sub>O, and the solution distilled or evaporated to remove volatile impurities. The recovered base may be fully hydrogenated by use of ordinary (Ni) catalysts. H. A. P.

Manufacture of nitrogenous condensation products. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 431,790, 16.1.34).—N- $\beta$ -Arylethylphthalimides, in which the aryl has at least one free *o*-position, when heated with AlCl<sub>3</sub> give *iso*quinoline derivatives convertible by further treatment with acid condensing agents

into cyclic ketones. E.g., interaction of N- $\beta$ -phenylethylphthalimide with NaCl-AlCl<sub>3</sub> at 160° gives 1 - phenylisoquinoline - 2' - carboxylic acid, m.p. 285-287° (picrate, m.p. 186°), which with 23% oleum at 100° gives Bz-3-azabenzanthrone, m.p. 186°. Other examples include the prep. of 3 : 4-benzo-1-phenyl-(I), m.p. 266-267° [from 2-aminodiphenyl



and  $o-C_6H_4(CO)_2O]$ , 4'-chloro-1-phenyl- (II), m.p. 230° (from 4-chloro- $\beta$ -phenylethylphthalimide, m.p. 112—114°), and 7-chloro-1-phenyl-isoquinoline-2'-carboxylic acid (III), m.p. 242—243° (decomp.), the compound (IV) [from 2 : 2'diaminodiphenyl and  $o-C_6H_4(CO)_2O]$ ; Bz-2-benzo-, m.p. 221° [from (I)], 6- and 7-chloro- (mixed) [from (II)], and 4chloro-Bz-3-azabenzanthrone, m.p. 168—170° [from (III)], and flavanthrone [from (IV) and NaCl-AlCl<sub>3</sub> at 200°]. H. A. P.

Preservation of unstable organic substances. [Antioxidants for rubber.] WINGFOOT CORP. (B.P. 430,335, 9.11.33. U.S., 7.12.32).—Ethers, sulphides, selenides, or tellurides containing the *sec.* (or its *N*-NO-derivative) or *tert.*-aminoaryl (or aralkyl) group are claimed as antioxidants for rubber, petroleum, etc. Examples are : *p*-tolyloxyphenyl*iso*propylnitrosoamine, *p*-NHBu·C<sub>6</sub>H<sub>4</sub>·OEt, *p*-NMe<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·Te·C<sub>10</sub>H<sub>7</sub>, and *p*-OH·C<sub>6</sub>H<sub>4</sub>·O·C<sub>6</sub>H<sub>3</sub>Me·NHPr<sup>\$#</sup> (3:4). H. A. P.

Waterproofing etc. composition.—See VI. Plasticisers. Condensation products from phenols.— See XIII. Esters of substituted diphenyls. Anethole. —See XX.

## IV.-DYESTUFFS.

Rayon, union, and hair dyes. Dyes for the toy maker.—See VI. Colouring substances of cane sugar.—See XVII.

#### PATENTS.

to light and levelling power, are prepared from o-NH2·C6H4·SO3H (I) and its substitution products (other than OH- or 6-halogeno-derivatives) as first components and heteronuclear acylamidoderivatives of 1: 3-OH C10H6 SO3H. Examples are the dyes: (I)  $\rightarrow 1$  : 8 : 6-NHAc·C<sub>10</sub>H<sub>5</sub>(OH)·SO<sub>3</sub>H  $OMe \cdot CH_2 \cdot CO \cdot NH \cdot C_{10}H_5(OH) \cdot SO_3H$ , (III)  $\rightarrow 2:8:6$ -NHAc·C10H5(OH)·SO3H (IV) or its N-Me derivative,  $3:1:6:4-\mathrm{NH}_2\cdot \mathrm{C}_6\mathrm{H}_2\mathrm{MeCl}\cdot\mathrm{SO}_3\mathrm{H}(\mathrm{V}) \rightarrow (\mathrm{IV}), \text{ and } 1:2:6 \mathrm{NH}_{2} \cdot \mathrm{C}_{6}\mathrm{H}_{3}(\mathrm{OMe}) \cdot \mathrm{SO}_{3}\mathrm{H} \rightarrow 1:5:7 \cdot \mathrm{NHAc} \cdot \mathrm{C}_{10}\mathrm{H}_{5}(\mathrm{OH}) \cdot \mathrm{SO}_{3}\mathrm{H}$ (VI) (all red);  $1:5:4-\mathrm{NH}_2 \cdot \mathrm{C}_6\mathrm{H}_3(\mathrm{NHAc})\cdot \mathrm{SO}_3\mathrm{H} \rightarrow (\mathrm{VI})$ or the corresponding Bz compound, (V)  $\rightarrow 2:5:7$ -NHAc·C<sub>10</sub>H<sub>5</sub>(OH)·SO<sub>3</sub>H (VII),  $\rightarrow 1:5:2$ -NH<sub>2</sub>·C<sub>6</sub>H<sub>3</sub>Cl·SO<sub>3</sub>H or 5:1:2:4:6-NH<sub>2</sub>·C<sub>6</sub>HMe<sub>3</sub>·SO<sub>3</sub>H (IV), 4:1:3:5-NH<sub>2</sub>·C<sub>6</sub>H<sub>2</sub>Me<sub>2</sub>·SO<sub>3</sub>H  $\rightarrow$  (VII),  $\rightarrow$  (VI), or  $\rightarrow$  (IV), its N-Me derivative (VIII), or the corresponding Bz compound (IX), 2:1:5:3-NH<sub>2</sub>·C<sub>6</sub>H<sub>2</sub>MeCl·SO<sub>3</sub>H (X)  $\begin{array}{l} \rightarrow \quad (VI), \quad (IV), \quad (VIII), \quad or \quad (IX) \quad (orange-red); \quad and \\ 5:1:2:4:3:6 \cdot or \quad 5:1:3:4:2:6 \cdot OH_2 \cdot C_6 Me_3 Cl \cdot SO_3 H \end{array}$  $\rightarrow$  (VII), and 2:1:5:3-NH<sub>2</sub>·C<sub>6</sub>H<sub>2</sub>MeCl·SO<sub>3</sub>H, 1:5:2- $NH_2 \cdot C_6H_3Cl \cdot SO_3H$ , or  $5:1:2:4:6-NH_2 \cdot C_6HMe_3 \cdot SO_3H$  $\rightarrow$  (IV) (orange). H. A. P.

Manufacture of water-insoluble azo dyes [pigments and ice colours]. I. G. FARBENIND. A.-G. (B.P. 426,564, 5.10.33. Ger., 5.10. and 1.11.32).-Arylamides of 2-hydroxy-3-carboxydiphenylene sulphide or oxide, free from solubilising groups, are used as coupling components. Browns are obtained with diazotised 5-nitro-p-toluidine, o-nitroaniline, etc.

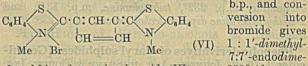
C. H.

Manufacture of azo dyes containing copper. I. G. FARBENIND. A.-G. (B.P. 425,990, 23.9.33. Ger., 23.9.32).-A trisazo dye of the type: 4:4'-diamino-3:3'-dimethoxyazobenzene  $\rightarrow 2$  mols. of an o-coupling phenol- or naphthol-sulphonic acid (I) is treated with a coppering agent until both OMe are demethylated. In the examples (I) is  $\alpha$ -naphthol-4:7-disulphonic acid (blue on cotton). C. H.

Manufacture of [aminoanthraquinone] dyes [for acetate silk]. Soc. CHEM. IND. IN BASLE (B.P. 425,517, 20.7.33. Addn. to B.P. 423,256; B., 1935, 397).-The dyes of the parent patent are made by condensing leuco-4-p-hydroxy- or -p-amino-anilino-1-hydroxyanthrawith a primary hydroxyalkylamine, or quinone a leuco-1-hydroxyalkylamino-4-hydroxyanthraquinone with a *p*-hydroxy- or *p*-amino-arylamine. C. H.

Production of compounds [dyes] containing (A) a methine or polymethine chain, (B, C) a polymethine chain. J. D. KENDALL (B.P. 431,141 and 431,186-7, [A] 23.12.33 and 6.11.34, [B, C] 23.12.33). -(A) Acids of the type CO<sub>2</sub>H·CH<sub>2</sub>·[CX:CH]<sub>n</sub>·CO<sub>2</sub>H, X being H or a substituent, are condensed with cyclic quaternary salts containing as reactive groups (a- or y-)thio- (I) or seleno-ether (II), amino- or arylamino-, acetamido-, or acetarylamido-vinyl groups. The products from (I) or (II) are dyes of true cyanine (n = 0), carbocyanine (n = 1), dicarbocyanine (n = 2), etc. type. E.g., condensation of 1-methylthiolbenzthiazole metho-p-toluenesulphonate (III) with CH<sub>2</sub>(CO<sub>2</sub>H), in C<sub>5</sub>H<sub>5</sub>N at 100° gives 1 : 1'-dimethylthiocyanine p-toluene-

sulphonate (bromide, m.p. 305°); similarly, 2-methylthiolquinoline metho-p-toluenesulphonate and glutaconic acid give 1:1'-dimethylcarbocyanine (bromide, m.p. 290°). Other examples, possibly new, are: 1:1' (or 3:3')-dimethyl-4:4'-quinazocyanine iodide, m.p. 290°; 1:1'-dimethyloxacyanine iodide, m.p. 305°; 1:1'-dimethyl-(Na)-thio-naphthacyanine and -carbocyanine p-toluenesulphonates, m.p. 297° and 268°; 1:1'-dimethyl- and 1:1'-dimethyl- and 1:1'-diethyl- $\psi$ -cyanine iodides, m.p. 278—279° and 263°; 1:5:1':5'-tetramethylthiazolecyanine iodide, m.p. 315°; 1:1'-diethylthio-, m.p. 311°, and 4:4'-dimethylthiol-2 : 2'-dimethyl-2 : 3-thiodiazolo-, m.p. 230°, and 3:3'-dimethylthiol-2:2'-dimethyl-1:1'-thio-(2:4)-diazolocyanine iodide, m.p. 240°; 2:2'-dimethylthiol-3:3'(or 1:1')-dimethyl-4':4'-pyrimidocyanine perchlorate, m.p. 245-247°; 1:1'-diethyl-, m.p. 249°, and 1:1'-dimethylthiocarbocyanine iodide, m.p. 277° (bromide, m.p. 272°); and 1:1'-dimethyloxacarbocyanine iodide, m.p. 277°. 1:4-Dimethylthiol-ββ'-thiodiazole, b.p. 170-171°/23mm., is prepared by direct methylation (Me<sub>2</sub>SO<sub>4</sub> and NaOH) of the (SH),-compound. Condensation of 1-acetanilidobenzthiazole ethiodide with CH<sub>2</sub>(CO<sub>2</sub>Et)<sub>2</sub> gives a yellow dye (sensitisation max.  $\lambda$  4650 mµ). (B) A similar condensation is carried out using anhydrides of acids of the general formula Me·[CX]<sub>2n-1</sub>:CH·CO<sub>2</sub>H, n being 1 or 2, e.g., crotonic anhydride (to give carbocyanines) or sorbic anhydride (dicarbocyanines). Condensation of 2 - methylthiolbenzoxazole metho - p - toluenesulphonate with dimethylacrylic anhydride in  $C_5H_5N$  gives 1 : 1 : 8'trimethyloxacarbocyanine iodide. (c) Condensation of cyclopentadiene (IV), indene (V), or their derivatives with (I) or (II) gives dyes of carbocyanine type in which the [.CH.], chain forms part of the nucleus of (IV). E.g., condensation of (III) with (IV) in  $C_5H_5N$  at the



b.p., and conversion into bromide gives

thenylthiocarbocyanine bromide (VI), m.p. 252° (decomp.) (iodide, m.p. 265°). In place of (I) the corresponding N-alkyl-2-(or 4-)thione and an alkyl halide etc. may be used. Other examples are: 1:1'-dimethyl-, m.p. 208°, and 1:1'-diethyl-9:9'-endodimethenyl-, m.p. 268°, and 1:1'-diethyl-7:7'-endodimethenyloxa-carbocyanine bromide, m.p. 240°, 4:5:4':5'-tetramethoxy-1:1'-dimethyl-, m.p. 296°, and -1:1'-diethyl-, m.p. 280°, 4:5:4':5'-bismethylenedioxy-1:1'-dimethyl-, m.p. 308°, and -1: 1'-diethyl-, m.p. 295°, and 1: 1'-dimethyl-5: 6: 5': 6'-dibenz-, m.p. 226°, -7: 7'-endodimethenylthiocarbocyanine p-toluenesulphonate, and carbocyanine iodides. m.p. 224° and 202°, from (IV) and the Me (VII) and Et p-toluenesulphonates (VIII) respectively, of dimethylthiolthio-ββ'-diazole (IX); [from (V)] 1:1'-dimethyl-(p-toluenesulphonate, m.p. 273°) and 1:1'-diethyl-9:9'-ophenylene-2: 2'-carbocyanine (bromide, m.p. 215°), 1:1'dimethyl- [iodide, m.p. 245° (decomp.)], 1:1'-diethyl-(p-toluenesulphonate, m.p. 226-228°), 4:5:4':5'-bismethylenedioxy-1: 1'-dimethyl- and -1: 1'-diethyl- (p-toluenesulphonates, m.p. 244° and 295°), 4:5:4': 5'tetramethoxy-1: 1'-dimethyl- and -1: 1'-diethyl- (bromides, m.p. 260° and 258°), and 1:1'-dimethyl-5:6:5':6'dibenz-7:7'-o-phenylenethiocarbocyanine (bromide, m.p.

208°), 1 : 1'-dimethyl-, m.p. 270°, and 1 : 3 : 5 : 1' : 3' : 5'hexamethyl-7 : 7'-o-phenyleneoxacarbocyanine iodide, m.p. > 300°, dimethyl-o-phenylenephthalazo- (iodide), 1 : 1'dimethyl- (p-toluenesulphonate, m.p. 298°), and 1 : 1'diethyl-9 : 9'-o-phenylene-4 : 4'-quinazo- (iodide, m.p. 222°), 3 : 3'-dimethyl- (p-toluenesulphonate, m.p. 306°), and 3 : 3'-diethyl-2 : 2'-dimethylthiol- (bromide, m.p. 270°), and 3 : 3'-dimethyl-2 : 2'.6 : 6'-tetramethylthiol-7 : 7'-o-phenylene-4 : 4'-pyrimido-carbocyanine- (bromide), and -carbocyanine bromides, m.p. 268°, 202°, and 258°, from (IX), (V), and (VII) and (VIII), and from dimethylthiolthio- $\alpha\beta$ 'diazole, (VII), and (V), respectively. H. A. P.

Production and use of [photosensitising] dyes having a methine or polymethine linkage. J. D. KENDALL (B.P. 425,609, 12.9.33).-The dyes contain **≮** 3 N, 2 being in one hetero-ring (in which only one is ortho to the methine bridge). The Me2 thioether (I), b.p. 170-171°/23 mm., of 2:5-dithiol-1:3:4-thiodiazole is condensed with quinaldine (II) (both as ethotoluenesulphonates) to give a dye, m.p. 220°; the dye from (I) and 2-methylbenzthiazole (III) has m.p. 237° (decomp.). Dyes are similarly obtained from ethotoluenesulphonates or ethiodides of: 3:5-dithiol-1:2:4-thiodiazole Me<sub>2</sub> thioether and (III)) [m.p. 257° (decomp.)], or (II) [m.p. 225° decomp.)]; Me thioether (IV), m.p. 68°, of 4-thiolquinazoline, m.p. 312°, with (III) [m.p. 295° (decomp.)], or (II) (m.p. 292°), or 2-methylbenzoxazole [m.p. 284–285° (decomp.)], or lepidine [m.p. 278° (decomp.)]; Me<sub>2</sub> thioether, b.p. 156°/20 mm., or 2:4-dithiolpyrimidine, m.p.  $> 300^\circ$ , with (III) [m.p. 249° (decomp.)] or (II) [m.p. 243° (decomp.)]; 4-methylquinazoline, m.p. 37°, b.p. 138°/10 mm., with 2-methylthiolbenzthiazole (V) [m.p. 295° (decomp.)], or CH(OEt)<sub>3</sub> [m.p. 286° (decomp.], or (IV) (m.p. 290°); 2:4-dimethylquinazoline, b.p. 143°/20 mm., with CH(OEt)<sub>3</sub> (m.p. 250°), or (V) [m.p. 276° (decomp.)], or 2-ethylthiolbenzthiazole [m.p. 283° C. H. (decomp.)].

Manufacture of sulphurised dyes. Soc. CHEM. IND. IN BASLE (B.P. 431,976, 15.8.34. Switz., 19.8.33).— Yellow to red, brown, and black dyes, fast to  $Cl_2$ , are obtained by sulphurising a polynuclear hydrocarbon having  $\lt$  3 rings (except anthracene, acenaphthene, and perylene) or their OH-derivatives. *E.g.*, pyrene and hydroxyphenanthrene are heated with S at 260—265° and 250—260°, respectively (brown-black), and fluoranthene and chrysene are heated with S and I (trace) at 270—290° (orange). H. A. P.

Coloured rubber.—See XIV. Bleaching-out layers.—See XXI.

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

Sulphur content of Merino wool, its distribution and relation to fineness and quality. F. N. BONSMA and P. J. JOUBERT (S. African J. Sci., 1934, 31, 347— 370).—The S content of wool is primarily dependent on hereditary factors. There is a general increase in vals. from fore- to hind-quarters. Cystine deficiency in the diet lowers the % of S in wool. This is associated with incomplete keratinisation of the entire fibre plasma. Relationships between S content and fineness are examined. CH. ABS. (p) Correlation between the quality of wool and the calcium and sodium content. R. SMIT and J. P. PEPER (Chem. Weekblad, 1935, 32, 65—68).—The % of inorg. constituents in wool from six different sources, and of Ca in 5 other samples (a) after washing with Triamol soap and (b) after extracting with light petroleum and washing with saponin, are recorded. The Ca content varies greatly with the method of washing, and no simple method of washing is without influence. No relationship is apparent between the quality of the wool and the Ca content. It is impracticable to examine by purely chemical means the theory of Krantz and Pohlmann regarding the relation between wool quality and salt content of the foodstuffs. H. F. G.

Oiling of wool. J. B. SPEAKMAN and J. R. GREEN-WOOD (J. Text. Inst., 1935, 26, P 271-288; cf. B., 1934, 667).—Objections to the use of drying oils are reduced by the addition of antioxidants, and to the use of mineral oils by the addition of substances such as oleyl alcohol which facilitate removal during scouring. A suitable oil should both lubricate the fibres and so reduce breakage during opening, and act as an adhesive and so reduce fly and facilitate condensing, drafting, and spinning. Rapid breaking of oiled roving in a ballistic tester shows that the work increases less rapidly with the amount of olive oil when this is > 4%, agreeing with the general practice of using  $3\frac{1}{2}\%$ . The oxidation of oil on tops was followed by measurements of the resultant increase in breaking load. Under these conditions 0.2% of  $\alpha$ -C<sub>10</sub>H<sub>7</sub>·OH is not an effective antioxidant; possibly it is adsorbed at the wool-oil interface. A. G.

Oiling of wool. RINOLDI (Boll. Laniera, 1934, 48, 231-235; Chem. Zentr., 1935, i, 1316).—The oleins should be free from olive oils, metals, and  $H_2SO_4$ , and have  $\gg 1\%$  of unsaponifiable matter, sap. val. 197-203, I val. about 84. A. G. P.

Photography in wool research. P. W. CUNLIFFE (Phot. J., 1935, 75, 439—444).—Important applications of photography, *e.g.*, location of inequalities, measurement of the cross-sections of fibres, determination of pigment distribution, etc., are described. J. L.

[Determination of] cotton maturity. W. E. STEVENS (Text. World, 1935, 85, 1475—1477).—The quality (maturity) of raw cotton can be accurately determined by its appearance through a polarising microscope using selenite plate. Fully mature fibres appear yellow to green, and immature fibres blue to indigo. These tests are shown to correspond with the physical properties of the resulting yarns. A. J. H.

Determination of [cotton] fibre maturity by polarised light. E. R. SCHWARZ and G. H. HOTTE (Text. World, 1935, 85, 1477—1478).—The theoretical basis of the test (see above) is discussed and apparatus is described. The objection that the test does not allow distinction between fine mature and coarse immature fibres is invalid because the wall thickness of the finest cotton fibres (Sea Island) in the mature state is > that of the coarsest (Indian) partly immature fibres.

A. J. H.

Boiling of dry-spun flax yarns. H. A. ELKIN (J. Text. Inst., 1935, 26, P 201-214).—The residual

impurities in flax yarns after alkali boiling may be measured by the loss in wt., by the alkali-solubility under standard conditions, or by the Cu no., there being an approx. const. relation between these three quantities. The loss in wt. increases with increasing amount of Na<sub>2</sub>CO<sub>3</sub> or of NaOH, and is considerably greater for the latter ; a const. moderate loss in wt. is more easily obtained with Na<sub>2</sub>CO<sub>3</sub>. With increasing amount of Na<sub>2</sub>CO<sub>3</sub> the breaking load of the boiled yarn rises to a max. and then diminishes; with NaOH the rise is very slight. When allowance is made for the loss in wt. the strength rises to a max., which is higher for NaOH than for Na<sub>2</sub>CO<sub>3</sub>. For a given loss in wt. yarn is more resistant to micro-organisms when boiled with Na<sub>2</sub>CO<sub>3</sub> than with NaOH. A. G.

Bleaching and catalysis defects of flax. I. Flax at different stages. (i) The flax stem. (ii) Retting of flax. J. PROVOOST (Het Vlas, 1934, 14, 63-64, 79-80, 87, 94-96, 103).-(i) The construction and composition of the flax stem in various growth stages are described.

(ii) The chemistry and biology of retting are considered. CH. ABS. (p)

Existence of transverse subdivision of vegetable fibres. R. HALLER (Helv. Chim. Acta, 1935, 18, 800-807).-The swelling of bamboo fibres observed by Hess and Lüdtke (A., 1928, 1361) cannot be due to any peculiarity in the structure of a natural fibre, as it is also observed, not only with cotton and hemp, but also with artificial silk fibres on treatment with H<sub>2</sub>SO<sub>4</sub>.

F. R. G.

Possible application of a modified single-stage process as a general method of determining cellulose. Y. C. TANG and H. L. WANG (Naturwiss., 1935, 23, 577) .- Impurities are much more rapidly removed from cellulose if the concn. of the solvent is raised to 1.5% of ClO<sub>2</sub> and 1-1.5% of C<sub>5</sub>H<sub>5</sub>N. Thus, after shaking with this solvent for 24 hr. and then extracting for 16 hr. with a solution of 7% of NaOH and 3% of NaCl, wheat straw is white and free from lignins and pentosans. A. G.

Heat of dissolution and of swelling of cellulose derivatives. I. Calorimetric phenomena. II. Calorimetric determination of degree of solvation. III. Heat of dissolution of acetates. T. NAKASHIMA and N. SATTO (J. Soc. Chem. Ind., Japan, 1935, 38, 232-234 B, 234-235 B, 235-237 B).-I. The heats of dissolution of cellulose acetates and nitrates in org. solvents are positive, and the heat of solvation is thus > the heat absorption due to expansion of the lattice, to dissociation of associated solvent mols., and to separation of dissolved particles. The heat of dissolution of nitrates in COMe2-C6H6 mixtures increases rapidly as the % of COMe, increases from zero, and reaches a max. It does not fall when the solubility increases rapidly and the heat absorbed by separation must therefore be small.

II. By measuring the heat evolved when acetates containing known amounts of solvent are added to excess of solvent, it is shown that the triacetate combines with 2 mols. of COMe<sub>2</sub> or CHCl<sub>3</sub> per C<sub>6</sub>, whereas the sol. acetate "Cellit " combines with only 1 mol.

III. In various solvents the heats of dissolution or

swelling of triacetate and of cellulose-dextrin acetate are nearly the same, whereas those of glucose penta- and cellobiose octa-acetate are strongly negative. A. G.

Action of nitrating acids on components of technical wood cellulose. I. M. NAIMAN and N. D. Ткоттаку (J.S.C.I., 1935, 54, 255-262 т).-The action of nitrating acids has been studied under the usual conditions of nitrocellulose (I) manufacture. The presence of non-cellulosic impurities in wood cellulose is shown to diminish its val. as material for nitration because they influence unfavourably the quality and the vield of (I). Lignin and resins, which are not completely citrated, make the nitrating acids impure and adversely effect the (I), and are the most undesirable admixtures. Pentosans give nitrates of inferior properties if compared with (I), but are in general not so harmful as lignin or resins. The thermal effect accompanying the nitration of components of wood cellulose in different conditions has been studied. The generally accepted physicochemical consts. are said not to be sufficient to character ise wood cellulose from the point of view of its suitability for nitration. Other conditions being equal (bleaching nitration, cellulose consts., etc.), the hardness of wood cellulose before bleaching (i.e., the cooking conditions) exerts a considerable influence on the quality of the resulting (I). Soft cellulose is greatly to be preferred for nitration.

Viscose. LXI. Isolation and purification of highly esterified cellulose xanthates. G. KITA, S. JIMBO, and H. TAKADA (J. Soc. Chem. Ind., Japan, 1935, 38, 252 B).-Cellulose xanthate is more completely pptd. from viscose by MeOH-C<sub>6</sub>H<sub>6</sub> than by MeOH-AcOH, and by this method xanthate treated a second time with CS<sub>2</sub> yields a product containing 1.1 Na and CS, per cellulose (C6) unit. A. G.

Micro-organisms causing spoilage of viscose material. A. IMSCHENETZKI (Microbiology U.S.S.R., 1933, 2, 369-376).-Penicillium oxalicum, Torulopsis rufula, and T. globosus from packing materials caused loss of elasticity in viscose bottle caps. The caps may be preserved in 0.5% aq. NaHSO3. Organisms are destroyed by heating at 60° for 3 min. Сн. Авз. (р)

Fine rayon and staple fibre. A. ZART (Chem.-Ztg., 1935, 59, 661-664) .- By diminishing the singlefilament denier softer yarns and fabrics are obtained. Cuprammonium rayon can be obtained in fine filaments more easily than can viscose rayon because the spinning solution is more viscous and the pptn. slower, rendering stretching during pptn. easier. Fine-filament viscose can be obtained by reducing the size of the spinning jets, by diminishing the concn. of the spinning solution, or by modifying the pptg. bath so that pptn. is slower. In practice a filament denier of 1 is obtainable. Acetate rayon is readily stretched during pptn. and the denier can be diminished to 0.1, but in practice it does not fall below 3. A. G.

Determination of methoxyl groups in wood. K. STORCH and I. WENZEL (Angew. Chem., 1935, 48, 513-514).-The method of Ender (B., 1934, 541), using vanillin, is shown to give OMe vals. 10-20% too low, owing to formation of Me<sub>2</sub>SO<sub>4</sub>; when, however, the

 $H_2SO_4$  distillate is made alkaline and after some time acidified, correct vals. are obtained for vanillin, and vals. for wood which approx. to those by the Zeisel method. E. W. W.

Effect of pretreatment of wood on the lignin determination. Distribution of methoxyls in wood. G. J. RITTER and J. H. BARBOUR (Ind. Eng. Chem. [Anal.], 1935, 7, 238—240).—Preliminary treatment with 95% EtOH in addition to the usual  $EtOH-C_6H_6$ and hot-H<sub>2</sub>O extractions, is necessary in the determination of lignin by the 72% H<sub>2</sub>SO<sub>4</sub> method in woods containing pyrocatechol tannins. The OMe in redwood (5.71%) and white oak (6.38%) is contained in the lignin and to a smaller extent in the holocellulose and extraneous material. F. N. W.

Effect of  $p_{\rm H}$  on the freeness of chemical and groundwood pulps. E. C. LARY and D. S. DAVIS (Paper Ind., 1935, 17, 249—250).—By suitable adjustment of the  $p_{\rm H}$  of unsized wood-pulp stocks, the freeness of which was then determined, it is shown that freeness reaches a min. at  $p_{\rm H} 5.4$  for (unbleached ?) sulphite, between  $p_{\rm H} 5$  and 6 for soda and kraft, and about  $p_{\rm H} 4.4$ for mechanical pulps. Data on the last-named were very erratic. Slower stocks are considerably more sensitive to changes in  $p_{\rm H}$  than are freer stocks. The  $p_{\rm H}$ determinations were carried out on the clear white-H<sub>2</sub>O from the freeness test, and not on the stocks themselves.

H. A. H.

**Erratum.** Page 719, col. 1, line 12 from bottom, for WURTZ read WURZ.

Rice straw and its utilisation in the paper industry. L. STANGALINI (Ind. Carta, 1934, 1, 503—510; Chem. Zentr., 1935, i, 1146—1147).—Vals. are given for the composition of rice straw and the length and thickness of the cellulose threads. The ash is about 14%(mainly SiO<sub>2</sub>), and the pulping process must be such as to leave much of this (combined) SiO<sub>2</sub> in the fibre, since complete removal of SiO<sub>2</sub> results in brittleness. The best process is treatment with Ca bisulphite followed by dil. NaOH. R. N. C.

Wet electro-filtration in the sulphite pulp indusiry. W. Hoss (Papier-Fabr., 1935, 33, 241–245).— The advantages of using electro-filters, especially for the elimination of  $H_2SO_4$  mist, and impurities such as Se, As, and Fe from the  $SO_2$ , are discussed. D. A. C.

Hydration theories and associated phenomena [connected with papermaking]. F. A. SIMMONDS (Paper Trade J., 1935, 101; T.A.P.P.I. Sect., 33-37).--A résumé of recent literature. H. A. H.

Relation of some properties of cotton rags to the strength and stability of experimental papers made from them. M. B. SHAW, G. W. BICKING, and M. J. O'LEARY (J. Res. Nat. Bur. Stand., 1935, 14, 649-665).—Papers made from new rags are more stable and much stronger than those made from old rags owing to the greater degree of cellulosic purity of the former. Rosin sizing decreases the folding-endurance but has little effect on bursting, tearing, or tensile breaking strength; at  $p_{\rm H} 5 > 1\%$  of rosin tends to cause deterioration and increase in the yellowing of the paper on heating. Increase in acidity has little effect on unsized but a deleterious effect on rosin-sized papers.

Starch beater sizing increases the bursting strength of papers from new rags but has no effect on those from old rags; starch does not diminish the stability of the papers in the heat test. Surface sizing with glue or starch retards chemical deterioration of the less stable papers but has no effect on papers of high stability.

A. R. P.

Sizing of hand-made laboratory [paper] sheets. VON POSSANNER (Papier-Fabr., 1935, 33, 257-260).-The influence of drying temp. and  $p_{\rm H}$  variation on the resistance to ink penetration of laboratory sheets was studied, using both resin and wax sizes. In the case of every resin size resistance to penetration rose sharply with rise in temp., and with a 23% free resin content was greatest at 120°, whereas with a colloidal resin size with a very high free resin content it continued to rise up to 130°, which was the max. temp. used. With wax size rise in temp. caused a decrease in penetration, which was greatest with dark-coloured sizes. With mixtures of resin and wax sizes the resistance  $\infty$  the quantities of each size present, and since wax size is ineffective in acid solution, the  $p_{\rm H}$  of the H<sub>2</sub>O for max. resistance should be 7.5-7.7. Drying the sheets under vac. by the "rapid Köthen" method was deleterious, but good results were obtained by introducing a current of air under the sheet. D. A. C.

Bewoid size system. B. WIEGER (Paper Trade J., 1935, 101; T.A.P.P.I. Sect., 51-55).-The method of manufacturing Bewoid size is described, and a no. of advantages are pointed out. Sizing theories in general are discussed. The membrane theory is rejected, as is also the electrostatic theory in its present form, since neither can account for the efficiency of the Bewoid process. It is suggested that the small amount of Na rosinate present in Bewoid size plays a part in mordanting the free rosin particles on to the fibres. Sizing is not a complete elimination of the capillary absorbency of the fibre structure, but is only an accumulation of obstructions against the speed of the H<sub>2</sub>O flow in the individual capillary spaces. Differences in the sizing capacity of various types of fibres are attributed to differences in fibre structure and mordanting power.

H. A. H.

Surface phenomena in the production of paper and cellulose. II. Wetting as a measure of the degree of paper sizing. I. I. KOVALEVSKI (Bumazhn. Prom., 1934, 13, Nos. 10—11, 21—33).—The quality of sizing is indicated by the selective wettability with  $C_6H_6$  or other non-polar liquids. Physical characteristics are discussed. CH. ABS. (p)

Internal structure of paper. A. J. BAILEY (Paper Trade J., 1935, 101; T.A.P.P.I. Sect., 38-40).—Photomicrographs of different kinds of paper in cross-section are given, and their significance is discussed. Fibrillation, considered alone, appears in general to be indicative of weakness, coincident with loss of structure (cf. B., 1931, 966; 1934, 447). Possible explanations are advanced. H. A. H.

Dependence of reflectance and opacity [of paper] on thickness; relation between contrast ratio and printing opacity. D. B. JUDD (Paper Trade J., 1935, 101; T.A.P.P.I. Sect., 58-59).—From graphs constructed according to the Kubelka-Munk formula, the increase in opacity due to increase in thickness and to decrease in reflectivity (e.g., by adding dye) may be determined. H. A. H.

Instrument for formation measurement [of paper]. M. N. DAVIS, W. W. ROEHR, and H. E. MALM-STROM (Paper Trade J., 1935, 101; T.A.P.P.I. Sect., 43—48).—The instrument, which measures the perfection of formation ("look-through") of any sample of paper in terms of a single coeff. termed the "formation no.," is described, all such vals. falling on an arbitrary scale. The instrumental gradings of a range of papers were in better agreement with the mean of two sets of visual gradings than were each of the latter themselves, provided the papers were all of one class. Compensation is made, by means of a "high pass filter," for variations due to differences in thickness. Very good reproducibility between one instrument and another is claimed, provided sufficient readings be taken.

H. A. H.

Instrumentation in brightness grading [of paper]. M. N. DAVIS (Paper Trade J., 1935, 101; T.A.P.P.I. Sect., 2—10).—The General Electric reflexion meter for determining brightness is described, and in particular factors affecting calibration and limits of accuracy are discussed. H. A. H.

Dyeing paper.-See VI.

#### PATENTS.

**Treatment [carbonisation] of raw wool.** DEUTS. HYDRIERWERKE A.-G. (B.P. 431,367, 19.4.34. Ger., 19.4. and 23.5.33).—Preliminary alkaline scouring is avoided by addition to the carbonising bath  $(H_2SO_4)$  of aliphatic ( $\langle C_{10} \rangle$ ) or alicyclic sulphonic acids that are stable to the carbonising liquid, *e.g.*, hexadecane, naphthenyl-,  $\beta$ -octadecenyloxyethane-, or *p*-dodecylthiolbenzene-sulphonic acids. H. A. P.

Washing process for textile materials. E. FRANZ (B.P. 432,018, 13.12.33).—Impurities in raw or manufactured textile waste (especially wool soiled with pitch) are removed by steeping the material in  $\lt 0.5$  pt. of an undiluted H<sub>2</sub>O-insol. carboxylic acid (*e.g.*, a fatty acid such as oleine or arachis-oleic acid), then removing most of this and converting the residue into a soap by treatment with an alkali (NH<sub>3</sub>), followed by washing. The acid is recovered from the washing liquor by treatment with a strong acid. A. J. H.

Cleaning [greasy] cotton sweepings. J. W. BOWERSOX, ASST. to BURTON-DIXIE CORP. (U.S.P. 1,980,747, 13.11.34. Appl., 24.1.30).—Used cotton waste is freed from grease and  $H_2O$  by agitation with powdered absorbent limestone. B. M. V.

Saponification of filaments, threads, yarns, fabrics, and the like having a basis of organic ester of cellulose. H. DREYFUS (B.P. 431,701, 15.1.34).— Introduction of 0.2—1% of a mono- or poly-hydric phenol [PhOH, C<sub>6</sub>H<sub>3</sub>(OH)<sub>3</sub>], or a weak aromatic acid or OH-acid (B2OH, o-OH·C<sub>6</sub>H<sub>4</sub>·CO<sub>2</sub>H), into the medium for saponification of cellulose esters reduces damage to the material. A. W. B.

Preparation or reduction of wood or other fibrous material. R. B. RESPESS, Assr. to RESPATS, INC. (U.S.P. 1,976,297, 9.10.34. Appl., 15.1.32).—Means for continuously defibring wood in a practically air-dry condition in a mill working on the kollergang principle are claimed. D. A. C.

Recovery of pulp from printed paper. A. B. DAVIS, J. F. THOMPSON, and A. T. GARDNER, ASSTS. to L. L. ALSTED (U.S.P. 1,975,771, 9.10.34. Appl., 24.10.31).—Paper printed with inks containing as pigment the Fe or Mn lakes of tannins or logwood extracts, which are capable of being bleached by oxidising or reducing agents, is shredded, bleached, *e.g.*, with  $H_2SO_3$  (as either  $SO_2$  or a sulphite), and optionally neutralised with borax. D. A. C.

Transparent sheet material. E. M. KRATZ, Assr. to MARSENE PRODUCTS Co. (U.S.P. 1,976,027, 9.10.34. Appl., 8.7.27).—A continuous sheet comprised of a mixture of gelatin and sulphonated castor oil, and formed on fabric or baked drying oils from which it can be readily stripped when dry, is coated with a waterproofing solution of cellulose nitrate and acetate, containing also dammar gum and  $(C_6H_4Me)_3PO_4$ .

D. A. C.

Treating and bleaching cellulose etc.—See VI. Plasticising cellulose esters.—See XIII. Adhesive. —See XV. Insecticide.—See XVI.

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

**Textile Calgon.** E. B. BELL (Amer. Dyestuff Rep., 1935, 24, 427–431).—Uses of Calgon  $(Na_6P_6O_{18}; B., 1935, 224)$  in the wet-processing of textile materials are described with reference to wool scouring, silk degumming, dyeing, kiering, and H<sub>2</sub>O-softening. A. J. H.

Unwanted creases in woven and knitted fabrics. F. P. W. WILSOME (Amer. Dyestuff Rep., 1935, 24, 385—386, 409).—The influence of fibre plasticity on fabric creasability is discussed, with especial reference to poplin and twill woven-cotton fabrics (I) and viscose and acetate rayon Milanese knitted fabrics (II). Creases in (I) as produced by bleaching in rope form can be removed by subsequent mercerisation; creases in (II) similarly produced cannot be removed without destroying the characteristics of the fabric. Fabrics containing tightly twisted yarns have an increased tendency to acquire permanent creases ("crowsfeet") during wetprocessing. A. J. H.

Sulphur bleaching of woollen materials. E. P. SANTCROSSE (Amer. Dyestuff Rep., 1935, 24, 418–420, 438).—Details are given of the construction of bleaching chambers suitable for wool yarn and fabric using  $SO_2$ derived from burning S. Wood, concrete, brick, sheet Pb (for ceiling), and Al (for hooks) are satisfactory, except that concrete and brick induce undesirable pptn. of sublimed S unless previously heated. A. J. H.

Textile uses of urea and thiourea. R. A. HAGUE (Text. Colorist, 1935, 57, 538).—Patented processes for using urea and thiourea in dyeing, printing, delustring of cellulose acetate rayon, and retting are reviewed.

A. J. H. Japanese dyeing tannins. XIV. Absorption of Korean " acer tannin " and commercial tannin by cellulose. Y. UYEDA (J. Soc. Chem. Ind., Japan, 1935, 38, 274-275B).—Absorption by cellulose of cryst. is < that of non-cryst. tannin, lower temp. increasing absorption. The effect of varying concn. of solution has been determined. F. R. S.

Rayon dyes. Anon. (Text. World, 1935, 85, 1278-1279).—Direct dyes which give level shades on unequal qualities of viscose rayon are classified. A. J. H.

Union dyes for half-wool piece goods. C. W. NELSON (Amer. Dyestuff Rep., 1935, 24, 437-438).-Practical notes are given. A. J. H.

Harmlessness of modern hair-dyes. C. GRIEBEL (Z. Unters. Lebensm., 1935, 70, 61-76).-Henna, reng, and catechu are harmless. The action of certain preps. reputed to contain henna is due to oxidising dyes. Of the latter, the NH2-compounds and polyphenols, particularly p-C<sub>6</sub>H<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub>, are harmful. The alleged restoration of the original colour to hair by Bi preps. is unfounded. E. C. S.

Dyes for the toy maker. S. I. OBERMANN (Amer-Dyestuff Rep., 1935, 24, 435-436).-Suitable types of dyes and methods are described. A. J. H.

Dyeing bright shades [on cotton] without using basic dyes. F. W. BERWICK (Text. World, 1935, 85, 1293) .- The difficulty of obtaining well-penetrated brilliant shades on lightly twisted yarn or closely woven fabric with basic dyes is avoided by applying selected acid dyes from a boiling bath after first mordanting the cotton with Katanol or other sulphurised phenol mordant.

A. J. H.

[Colour] spots on [silk] hosiery. H. WHITE (Text. World, 1935, 85, 1465).-Wet silk hose dyed with Nerol 2B (C.I. 304) and Sulphon Cyanine Black B (C.I. 307) developed red and green spots when exposed to  $SO_2$ . The presence of traces of alkali (e.g., Na<sub>2</sub>CO<sub>3</sub>) promotes the discoloration. A. J. H.

Mechanism of dyeing paper. R. E. ROSE and J. C. SCHMIDT (Paper Trade J., 1935, 101; T.A.P.P.I. Sect., 55-57).-Dyeing theories are briefly discussed, particularly as they affect the application of acid, basic, and direct dyes to paper fibres. It is believed that paper dyeing is more akin to lake making than to textile dyeing. H. A. H.

Dyeing of sponges and loofahs. C. T. HORRTING (Text. Colorist, 1935, 57, 561-562).-Sponges are dyed at  $\geq 60^{\circ}$  in a bath containing 1-3% (on the sponge) of AcOH and acid milling, acid, basic, or direct dyes, followed by soaping at 35°. Loofahs are chiefly dyed with direct dyes. A. J. H.

Effect of mercerisation (with stretching) on the tensile strength and extensibility of cotton yarn. K. LANGER (Textilber., 1934, 15, 165-169).-Changes in the tensile strength and extension at breaking point of a 2/40's cotton yarn as produced by mercerising (with graded amounts of stretching) in the usual manner after a pressure boil and also by direct mercerisation with the aid of Mercerol as a wetting agent are given. The method of mercerising has little influence on the resulting changes, but increased stretching progressively diminishes extension. Mercerising increases tensile

strength 35% (average) and diminishes extension 35 and 26% (without and with Mercerol respectively under the same max. stretching). Development of weakness in places after mercerisation was traced to irregularities in spinning the yarn and not to mercerisation. A. J. H.

Effect of mercerisation on the tensile strength. length, and fineness of cotton fibres. K. LANGER (Textilber., 1935, 16, 507-508).-Changes in the tensile strength, length, and fineness of the individual fibres present in a soft spun 2/40's cotton yarn mercerised as previously described (see above) are given. The resulting average increase of strength was 33%. Tensile strength was 7% higher when mercerisation was effected without Mercerol. Under min. and max. stretching conditions during mercerisation the resulting changes in length were -1.4 and +3.9%, and -2.9 and +1.9%, and in fineness +2.8 and -8.4%, and +2.6 and -8.2% (without and with Mercerol respectively).

A. J. H.

Dyeing of sheep skins.—See XV.

## PATENTS.

[Agents for] improving wetting-out properties of [porous or fibrous] materials. C. CHAPMAN, and MONSANTO HOLDINGS, LTD. (B.P. 431,645, 12.12.33) .--The material (e.g., cotton wool) is impregnated with a solution (in H<sub>2</sub>O) of a monohalogenated homologue of PhOH  $(1:6:3-C_6H_3MeCl·OH, 1:3:2:5-C_6H_2Me_2Cl·OH)$ and dried. H. A. P.

[Manufacture of] wetting agents and the like. W. J. TENNANT. From HENKEL & Co. (B.P. 431,682, 7.12.33).—Known detergents, wetting agents, etc., e.g., the higher Na *n*-alkyl sulphates, sulphonated glyceryl monocetyl ether, are mixed with salts of an acid intermediate in composition between HPO3 and H<sub>3</sub>PO<sub>4</sub> (Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub>). (Cf. B.P. 408,708; B., 1934, 534.) H. A. P.

Cleaning of washing fluids and recovery of solvents. A. AGRONOFSKY (U.S.P. 1,977,107, 16.10.34. Appl., 12.12.31).-Apparatus for recovery of dry-cleaning solvents from air effluent from drying chambers is claimed. A. W. B.

Bleaching of cellulose. F. R. SEAVEY, A. J. PHILLIPS, and F. Olsen, Assrs. to Cellulose Res. Corp. (U.S.P. 1,975,590, 2.10.34. Appl., 10.4.29).—A slurry of cellulose (for paper making) is led through an electrolytic cell containing aq. NaCl so that it is constantly in contact with the anode, where it is bleached with the nascent O and Cl there formed. A. J. H.

Colouring of organic solvents, lacquers, fats, oils, resins, waxes, and products obtained therefrom. I. G. FARBENIND. A.-G. (B.P. 431,649, 12.1.34. Ger., 25.2.33).-Azo dyes from benzenediazonium salts. free from NO2, CO2H, or SO3H, coupled with 4-hydroxy-2-quinolones containing an alkyl (> $C_3$ ) attached to the N, e.g.,  $NH_2Ph \rightarrow 4$ -hydroxy-1-butyl-2-quinolone, are claimed to have high solubility in the above media.

A. W. B.

Treatment of textile material. I. G. FARBENIND. A.-G. (B.P. [A] 431,524 and [B] 431,703, [A] 10.1.34, [B] 15.1.34. Ger., [A] 10.1.33, [B] 14.1.33).—Resistance to creasing is imparted by treating textiles with urea, a polymeride of  $CH_2O$ , and an agent which yields (A) alkali, e.g., alkali starch, NaOEt, NaHCO<sub>3</sub>, (B) an acid or acid salt only during the condensation, e.g., org. acid esters, amides, aldehyde-bisulphite compounds; the materials are dried and heated to 70-130°.

A. W. B.

Manufacture of dressed textile materials. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 431,704, 15.1.34).—Resistance to creasing of textiles (artificial silk) is afforded by treating with an aq. solution of a mixture of a highly polymeric compound, *e.g.*,  $(CH_2:CH\cdotOH)_n$ ,  $[C_3H_5(OH)_3]_n$ , esters and ethers thereof, mixed polymerides, *e.g.*, from  $CH_2:CH\cdotOAc$  and  $CH_2(CO_2H)_2$ , and an aldehyde, *e.g.*,  $CH_2O$ , glyoxal, followed by heating to 70—150°. A. W. B.

Rendering woollen or other animal hair textiles repellent to water. STUDIENGES. F. FASERVEREDLUNG M.B.H. (B.P. 431,670, 27.11.34. Ger., 4.12.33).— Thoroughly cleansed wool, preferably after immersion in a boiling acid dye-bath followed by neutralisation, is treated with a 0.1-1.0% (0.1-0.4%) soap solution at  $\leq 25^{\circ}$  (25-30°) for 5-60 min. and rinsed in cold soft H<sub>2</sub>O. A. W. B.

Waterproofing and fire-retarding composition. C, F. BOOTH, ASST. to SWANN RESEARCH, INC. (U.S.P. 1,975,072, 2.10.34. Appl., 1.7.31).—Chlorinated ( $\lt$  60% Cl) diaryls (Ph<sub>2</sub>), mixed with a metallic soap (Al stearate) and a natural or artificial wax or resin (paraffin wax), or a plasticiser, *e.g.*, (C<sub>6</sub>H<sub>4</sub>Me)<sub>3</sub>PO<sub>4</sub>, are claimed as water- and fire-proofing agents for fibrous materials. A. W. B.

Treatment [delustring] of [textile] material containing organic esters of cellulose. C. DREYFUS and W. WHITEHEAD, ASSTS. to CELANESE CORP. OF AMER. (U.S.P. 1,976,218, 9.10.34. Appl., 8.9.28).—Cellulose acetate and like materials are delustred by exposure to boiling aq. liquors or steam while under tension, so that distortion is avoided. A. J. H.

**Process of mercerisation.** Soc. CHEM. IND. IN BASLE (B.P. 431,662, 19.6.34. Switz., 20.6.33).—Acid sulphates (or their salts) of hydroaromatic, aromaticaliphatic, or heterocyclic alcohols, *e.g.*,  $C_6H_{11}$ OH, terpineol, CH<sub>2</sub>Ph·OH, alone or mixed with solvents, *e.g.*, BuOH, pine oil, OH·[CH<sub>2</sub>]<sub>3</sub>·OBu, or dispersing agents, *e.g.*, PhOH,  $C_6H_4$ Cl·OH, alkali-stable sulphonated fatty acids or their derivatives, *e.g.*, castor oil sulphonated in presence of o- $C_6H_4$ (CO)<sub>2</sub>O, are claimed as wetting agents for mercerising baths. A. W. B.

Insecticide [for fabrics]. R. E. SPOKES, ASST. to E. A. MURRAY CO. (U.S.P. 1,977,412, 16.10.34. Appl., 11.2.33).—To a solution of a cryst. chlorohydroxydiphenyl in petroleum naphtha is added a mineral wax (e.g., petroleum scale) and stearanilide (to prevent crystallisation of materials when sprayed on to fabric). The prep. serves to protect materials against the common clothes moth. A. G. P.

Fabric treatment [coating or filling apparatus]. BRIT. CELANESE, LTD., A. MELLOR, and R. J. MANN (B.P. 432,429, 26.1.34).

Dressings etc.—See XX.

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

Mechanism of lead chamber reaction. I. Reaction between sulphurous and nitrous acids in a dilute system. E. ABEL and J. PROISL [with J. SCHAFRANIK and R. SMRŽ] (Monatsh., 1935, 66, 6—34).— The reaction between  $H_2SO_3$  and  $HNO_2$  in dil.  $H_2SO_4$ solution has been investigated under various conditions. In the concn. range studied  $N_2O$  and NO are formed, but no  $N_2$ . The yield of  $N_2O$  is increased by a high ratio of  $SO_2$ :  $HNO_2$  in the reaction products. The intermediate formation of HNO is probable.

O. J. W. **Preparation of hydrochloric acid from hydrogen and chlorine.** I. S. MOROZOV (Trans. State Inst. Appl. Chem. U.S.S.R., 1934, No. 20, 69–76).—Acid free from  $Cl_2$  is obtained by burning the gases (5–10% excess of  $H_2$ ) in presence of a catalyst, *e.g.*, quartz.

CHEM. ABS. (p)

Production of salt. J. E. HYLER (J. Chem. Educ., 1935, 12, 203-207).—Manufacturing processes are described and illustrated. L. S. T.

Salt systems of Chili nitrate mineral. A. GRAZIADEI (Chim. et Ind., 1935, 33, 1307–1316).— The polytherms of the system NaCl-NaNO<sub>3</sub>-H<sub>2</sub>O are applied to a quant. treatment of the extraction of NaNO<sub>3</sub> from Chili saltpetre. J. S. A.

Salt systems of Chili nitrate mineral. A. GRAZIADEI (Chim. et Ind., 1935, 34, 29–36; cf. preceding abstract).—The system  $NaNO_3-Na_2SO_4-H_2O$  is considered in relation to the extraction of  $NaNO_3$  from Chili saltpetre. R. S.

Chlorination of calcium phosphate. B. A. ROSHDESTVENSKI (Trans. State Inst. Appl. Chem. U.S.S.R., 1934, No. 20, 47-58).—Chlorination of Ca phosphates in bone charcoal and apatite (750°) yields POCl<sub>3</sub>, which on passing over red-hot coal [? C] is converted into PCl<sub>3</sub>. CH. ABS. (p)

"Water-solubility" of basic slag. STEHER and DITTRICH-BACH (Superphosphat, 1934, 10, 121–122). —Considerable proportions of  $PO_4$ " may be extracted from basic slag by many extractions with  $H_2O$ . The presence of  $CO_2$  in the  $H_2O$  increases the amount of  $PO_4$ " obtained per extraction. CH. ABS. (p)

Adsorbents and permutites. A. L. LIUBIMOV (Trans. Inst. Econ. Mineral. U.S.S.R. [10-year vol.], 1933, 73—90).—Gumbrin (floridin) activated by heating (350°) for 1.5 hr. in a mixture of petroleum combustion gases and air, and Askanite (bentonite) activated by treatment with dil. H<sub>2</sub>SO<sub>4</sub> and drying at 100°, are used for filtration and bleaching of petroleum oils. The prep. of Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub> gel from nephelite is described. The material is used in H<sub>2</sub>O-softening and also has adsorptive power (C<sub>6</sub>H<sub>6</sub>, SO<sub>2</sub>) equal to that of SiO<sub>2</sub> gel. CH. ABS. (p)

Preparation of aluminium chloride from the oxide. P. P. FEDOTEEV and A. A. TSCHISHIK (Trans. State Inst. Appl. Chem. U.S.S.R., 1934, No. 20, 59-68). —The formation of AlCl<sub>3</sub> from kaolin is facilitated by preliminary treatment with HCl or HNO<sub>3</sub> (warm).

Сн. Авз. (р)

(A) Preparation of concentrated solutions of hydrogen peroxide. (B) Preparation of hydrogen peroxide. A. F. SAGAIDATSCHNI and S. N. LURE (Trans. State. Inst. Appl. Chem., 1934, 19, 23-35, 35-47).---(A) Working conditions for the prep. from  $K_2S_2O_8$  are investigated.

(B)  $K_2S_2O_8$  (100 kg.) is mixed with 60%  $H_2SO_4$  (28.5 kg.) and heated to 100° in a vac. After 15—20 mm. steam is passed into the vessel at the rate of 40 kg. per hr.  $H_2O_2$  at 30—35% concn. distils. CH. Abs. (p)

Sulphur and sulphuric acid. I. V. RABSCHINSKI and M. A. MENKOVSKI (Trans. Inst. Econ. Mineral. U.S.S.R. [10-year vol.], 1933, 170—194).—The recovery of S from pyrites flotation tailings is described. S is best obtained from SO<sub>2</sub> by use of a bauxite catalyst and C or CO. S is also obtained from ores by subliming in an atm. of CO<sub>2</sub> and N<sub>2</sub>. CH. ABS. (p)

Determination of water in by-product sulphur. L. SHNIDMAN (Ind. Eng. Chem. [Anal.], 1935, 7, 246– 248).— $H_2O$  distils with the oil when S is heated in a Cu still with gas oil and light oil. The wt. of S and vol. of distilled  $H_2O$  are determined. The results agree closely with those obtained by the air-drying method at  $47\pm2^\circ$ . E. S. H.

Preparation of phosphorus oxychloride. V. A. MAZEL and M. B. GOLDBERG (Trans. State Inst. Appl. Chem. U.S.S.R., 1934, No. 20, 41-46).—POCl<sub>3</sub> can be obtained from its elements in glass or Sn-plated equipment. CH. ABS. (p)

Electro-filtration.—See V. Mortars for  $Ca(ClO_3)_2$ industry.—See IX. Treating solutions containing U.—See X. Chrome liquors.—See XV. Determining K in fertilisers.—See XVI.

#### PATENTS.

Production of nitrate of lime through absorption, at elevated temperatures, of nitrous fumes. G. LEFORT DES YLOUSES, ASST. to L'AZOTE FRANÇ. (U.S.P. 1,959,480, 22.5.34. Appl., 6.8.31. Fr., 8.8.30).—The gases produced by catalytic oxidation of  $NH_3$  are passed through vessels filled with porous CaO prepared by passing hot gases through granules of Ca(OH)<sub>2</sub> until the H<sub>2</sub>O is expelled. A. R. P.

Manufacture of superphosphate and like products. D. WELLISCH (U.S.P. 1,959,973, 22.5.34. Appl., 20.8.29. Ger., 7.2.29).—The reaction between the  $H_2SO_4$  and the phosphate rock is carried out in a horizontal cylindrical chamber rotating about a stationary hollow axis through which heat can be applied.

A. R. P.

Production of precipitated silica. S. S. SVENDSEN, Assr. to CLAY REDUCTION Co. (U.S.P. 1,959,747-9, 22.5.34. Appl., [A] 3.2.30, [B] 8.6.31, [C] 29.6.31).— (A) Vapours of SiF<sub>4</sub>,2NH<sub>3</sub> (I) are brought in contact with aq. NH<sub>4</sub>F to produce gelatinous SiO<sub>2</sub> and NH<sub>4</sub>F solution, which is used for the decomp. of clay to produce more (I). (B) (I) is passed into boiling H<sub>2</sub>O and the gelatinous SiO<sub>2</sub> is collected, dried, and heated at 500° to produce a product containing about 2% of H<sub>2</sub>O and having an apparent d of 10—14 lb./cu. ft. (c) (NH<sub>4</sub>)<sub>2</sub>SiF<sub>6</sub> is decomposed with dil. aq.  $NH_3$  at  $< 34^{\circ}$  (0–10°) to give a product which is opaque in oil and has  $n \cdot 1 \cdot 4$ . A. R. P.

Manufacture of alumina. A. FLEISCHER, ASST. to KALUNITE CO. (U.S.P. 1,960,336, 29.5.34. Appl., 30.6.33).—Basic K alum is heated in a multiple-hearth furnace at 400—700° in an atm. of NH<sub>3</sub> to volatilise as  $(NH_4)_2SO_4$  the SO<sub>4</sub>" combined with the Al<sub>2</sub>O<sub>3</sub>. The product is then leached to remove K<sub>2</sub>SO<sub>4</sub> and leave a light, reactive form of Al<sub>2</sub>O<sub>3</sub>. A. R. P.

Extraction of impurities from mineral raw materials [e.g., clays, bauxite, asbestos]. R. STAUFER and K. KONOPICKY, ASSTS. to ALTERRA A.-G. (U.S.P. 1,959,448, 22.5.34. Appl., 2.5.30. Austr., 6.5.29).—The material is digested at 100° with 5—20 (10%) H<sub>2</sub>SO<sub>4</sub> containing FeSO<sub>4</sub> (10%), to extract the Fe<sub>2</sub>O<sub>3</sub>, and the resulting solution is filtered, treated with SO<sub>2</sub> to reduce Fe<sup>TT</sup> to Fe<sup>TT</sup>, cooled to remove part of the Fe<sup>TT</sup> as FeSO<sub>4</sub>,7H<sub>2</sub>O, and used again after addition of more H<sub>2</sub>SO<sub>4</sub>. A. R. P.

Hydrolysing titaniferous solutions. B. D. SAKLATWALLA and H. E. DUNN, Assrs. to SOUTHERN MINERAL PRODUCTS CORP. (U.S.P. 1,959,765, 22.5.34, Appl., 29.11.32).—The  $Ti(SO_4)_2$  solution from decomp. of ilmenite with  $H_2SO_4$  is passed under pressure through a parallel-flow type of heat exchanger, in which it is heated at 125° for sufficient time to hydrolyse about 85—90% of the Ti<sup>IV</sup>, and is then discharged through spray nozzles into a cooling chamber. A. R. P.

Purifying H<sub>2</sub>. NH<sub>3</sub> and H<sub>2</sub>S from gases.—See II.

## VIII.-GLASS; CERAMICS.

**Removal of iron oxide from silica sands.** F. W. ADAMS (J. Soc. Glass Tech., 1935, **19**, 118-124 T).— Fe<sub>2</sub>O<sub>3</sub> as a surface impurity on the grains is removed by agitation at  $26-65^{\circ}$  with a solution of NaHC<sub>2</sub>O<sub>4</sub> or K quadroxalate (0.25-2%) containing 0.05-1% of FeSO<sub>4</sub>,7H<sub>2</sub>O. The necessary plant may be built of wood and the disposal of the effluent is easy. J. A. S.

Influence of carbon dioxide on the melting of glass. M. A. BESBORDOV, A. A. SOKOLOVA, and G. A. SHINKÉ (J. Soc. Glass Tech., 1935, **19**, 143–150 T).— A glass (SiO<sub>2</sub> 74, CaO 10, Na<sub>2</sub>O 16%) was made from batches having the Na<sub>2</sub>O introduced as either Na<sub>2</sub>CO<sub>3</sub> and/or Na<sub>2</sub>SO<sub>4</sub> + C. The melting was carried out for 4 hr. at temp. ranging from 900° to 1300° in atm. of air and CO<sub>2</sub>. The amount of CO<sub>2</sub> retained by the glass from the Na<sub>2</sub>CO<sub>3</sub> batch melted in CO<sub>2</sub> decreased from 0.13% for the 900° melt to 0.05% for the 1300° melt. Glasses from the Na<sub>2</sub>SO<sub>4</sub> + C melt contained a max. (at 1100°) of 11.99% of the original Na<sub>2</sub>SO<sub>4</sub> and 0.12% of CO<sub>2</sub>, and for 1300° the corresponding vals. were 0.68% of SO<sub>3</sub> and 0.05% of CO<sub>2</sub>. When melted in air the amounts of residual CO<sub>2</sub> and SO<sub>3</sub> were somewhat smaller. J. A. S.

Corrosion of crucibles for glass making; corrosion experiments on barium crown glass. M. WATATANI (Repts. Imp. Ind. Res. Inst., Osaka, 1934, 15, No. 2, 1—24).—The corrosion-resisting properties of various materials towards Ba crown glass was studied. Diaspore gave the best results. CH. ABS. (e)

British Chemical Abstracts-D

Volatility of soda-lime-silica glasses. H. W. Howes, H. LAITHWAITE, E. PRESTON, and W. E. S. TURNER (J. Soc. Glass Tech., 1935, 19, 104—117 T).— Precautions necessary to obtain consistent measurements of the volatility of Na<sub>2</sub>O at temp. up to 1400° are described. The volatility of the Pt vessel was small but measurable. The effects of surface area, shape of the containing vessel, and convection currents in the furnace were studied. During the initial volatilisation period traces of H<sub>2</sub>O, As<sub>2</sub>O<sub>3</sub>, SO<sub>3</sub>, etc. are expelled and must be taken into account. A "pure" glass, SiO<sub>2</sub>  $73 \cdot 75$ , Fe<sub>2</sub>O<sub>3</sub> + Al<sub>2</sub>O<sub>3</sub> 0·40, CaO 9·90, Na<sub>2</sub>O 15·94%, was studied and Na<sub>2</sub>O was the only volatile constituent at 1400°. J. A. S.

Basis of a standard test for the chemical durability of glass bottles. W. E. S. TURNER [with H. S. BLACKMORE, V. DIMBLEBY, H. S. Y. GILL, H. W. HOWES, and W. J. A. WARREN] (J. Soc. Glass Tech., 1935, 19, 171-188 T).-The test pieces must be of both standard capacity and dimensions and the conditions of annealing and storage must be known. Treatment with H<sub>2</sub>O at or near b.p. is best effected in steam and is little affected by variation in barometric pressure. The amount of alkali extracted by  $H_00 \propto$  the area exposed, but the concn. in the extract constantly diminishes. Conditions influencing the results for individual bottles are discussed, e.q., personal factors, time of test, storage, age, and chemical composition. The visible effects of the durability test or of weathering are discussed under the headings of pitting, flaking, and cracking. J. A. S.

Devitrification constants of some potash-leadsilicate glasses along the lead-glass line. R. GEPPERT and A. DIETZEL (Sprechsaal Keram., 1934, 67, 591—593; Chem. Zentr., 1935, i, 1287).—Rates of crystallisation as a function of temp. are recorded for 4 K<sub>2</sub>O-PbO-SiO<sub>2</sub> glasses (25, 30, 35, and 40% PbO). Zschimmer's rule was confirmed. Devitrification occurred with difficulty, the crystals being cristobalite and tridymite. H. J. E.

Brilliance exhibited by lead crystal glasses. E. PRESTON, A. J. HOLLAND, and W. E. S. TURNER (J. Soc. Glass Tech., 1935, 19, 125-138 T).-13 specimens of British and Continental glass were examined for general appearance, chemical composition, and for angle and quality of polishing of the cut. The decoloriser was generally MnO (sometimes with traces of NiO) although in three cases As<sub>2</sub>O<sub>3</sub> was found. One glass contained only 2% of PbO and could not be classed as "lead crystal," and its durability was very poor. All glasses had a sufficiently low Fe<sub>2</sub>O<sub>3</sub> content for satisfactory brilliance. Data are recorded showing the increase in Fe<sub>2</sub>O<sub>3</sub> content of a glass left in contact with the refractory. Great differences were found in the angles of cut. The wide (British) cut (120-158°) does not afford as great a brilliance as the narrow (Belgian) cut (77-86.5°). Microscopical examination showed that, in general, brilliance suffered by insufficient polishing. J. A. S.

Determination of ferrous iron in glass(continued). K. TAKAHASHI and S. SHINKAI (J. Soc. Chem. Ind., Japan, 1935, 38, 266 b).—The determination of Fe in glass containing As has been studied, using the method reported previously (B., 1935, 591). No reaction occurs between Fe and As during the analysis. R. S. B.

Ground-coat studies. V. Mill additions and consistency. E. P. CZOLGOS (Better Enamelling, 1934, 5, No. 1, 10—12).—The effects of  $H_2O$  hardness on the properties of a ground-coat enamel slip (2/3 groundcoat frit, 1/3  $H_2O$ , clay, SiO<sub>2</sub>, or felspar, borax, MgCO<sub>3</sub>, and bentonite) are described. SiO<sub>2</sub> and felspar can be interchanged without affecting the consistency. Clay settles less and reaches stability quicker in  $H_2O$  or mill-liquor solution containing 0.75% of borax. Incorporation of suitable electrolytes in the enamel slip helps to prevent settling. CH. ABS. (e)

Adhesion of base enamels containing cobalt and nickel. F. HEIMES (Sprechsaal Keram., 1934, 67, 720-722; Chem. Zentr., 1935, i, 1104; cf. B., 1935, 405).—Measurements were made of the potentials developed with an Fe electrode in an enamel melt. Addition of NiO and CoO gave variable effects. Atm.  $O_2$  was an important controlling factor. H. J. E.

"Fish eyes" [in enamel]. VIELHABER (Emailwar. Ind., 1934, 11, 394—395; Chem. Zentr., 1935, i, 1104). —This effect in Majolica enamel is due to decomp. of sulphides. H. J. E.

Determination of small quantities of fluorine in enamel. W. DAWIHL (Keram. Runds., 1934, 42, 607—608; Chem. Zentr., 1935, i, 1106).—The method of pptn. of F as  $CaF_2$  is improved by adding EtOH. Other methods are reviewed. Hackl's Ti sulphate- $H_2O_2$  method is inapplicable in presence of  $PO_4^{\prime\prime\prime}$ ; the F should be removed by heating with HClO<sub>3</sub> and titrated with Th nitrate. H. J. E.

Solubility of enamel of cooking vessels. B. WILLBERG and H. CAJANDER (Z. Unters. Lebensm., 1935, 69, 591—594).—The vessels were boiled with 4%aq. AcOH for 3 periods of 2 hr., and the total dissolved substances,  $B_2O_3$ , and F in the first and third extracts determined. The results of the boiling tests and the composition of the enamel of six brands of vessel are tabulated. E. C. S.

Sardinian kaolin, a national raw material. F. DE CARLI (Chim. e l'Ind., 1935, 17, 399-405).— The mining and refining are described. D. R. D.

Barium hydroxide to prevent scumming of ceramic products. J. O. EVERHART, W. C. RUECKEL, and C. R. AUSTIN (Ohio State Univ. Eng. Expt. Sta., 1935, Circ. 30, 15 pp.).—Comparative data for  $Ba(OH)_2$  and  $BaCO_3$  are recorded. 6 lb. of a  $3:2 Ba(OH)_2$ –  $BaCO_3$  mixture were required to replace 7 lb. of  $BaCO_3$ . Rapid drying aids scum formation. CH. ABS. (e)

Relations between fineness of particles and plasticity of ceramic bodies. R. ISOMATSU (Repts. Imp. Ceram. Expt. Inst., 1933, No. 11, 13—41).—The min. content of particles of  $< 1 \mu$  diam. for the best working conditions was approx. 20%. Bentonite had an exceptionally high % of subcolloidal (diam. 0·1— 1·0  $\mu$ ) and colloidal particles, and gave sufficient plasticity when it was added in small amounts. Acid earths are also rich in fine particles, and can be used to improve the plasticity of meagre material. Bodies containing no very small particles have practically no plasticity. CH. Abs. (e)

Comparison of wet methods used for the fineness test of sands and clays. R. C. HILLS (Trans. Amer. Foundrymen's Assoc., 1934, 5, No. 6, 101–128).— Comparative tests of various methods are recorded and discussed. CH. ABS. (e)

Electric furnaces for ceramic firing. E. J. HARRIS and B. A. BOVÉE (Bull. Amer. Ceram. Soc., 1935, 14, 219—222).—The construction and operation of a SiC resistor furnace (hearth  $16 \times 30 \times 17$  in.; 1200— 1500°) is described. Turbulence of the furnace atm. during the oxidation period was obtained by a small jet of steam or air. Capital and operating costs are given. J. A. S.

Control of atmosphere in [ceramic] kilns. M. H. MAWHINNEY (Bull. Amer. Ceram. Soc., 1935, 14, 222— 224).—Automatic devices for the control of primary and secondary air, and for the mixing of air and fuel in gas and oil firing, are described. J. A. S.

Oxidation of ceramic ware during firing. VII. Review. F. G. JACKSON (Bull. Amer. Ceram. Soc., 1935, 14, 225–229).—The fundamental principles are described and discussed. (Cf. B., 1925, 760.) J. A. S.

Characteristics of fireclay with special reference to manufacturing difficulties. C. A. G. THOMAS and J. R. ADDERLEY (J. Soc. Glass Tech., 1935, **19**, 139— 142 T).—Attempts to remove ferruginous impurities from Stourbridge clays by means of an electro-magnet or by blunging and sieving resulted in a decrease of  $Fe_2O_3$  by  $\gg 0.15\%$ . The plasticity is an inherent property of the clay and is often in inverse ratio to the depth of the seam. J. A. S.

High-grade semi-acid ladle linings. S. A. SHICHAREVITSCH and G. L. KOGAN (Ogneuporui, 1934, 2, No. 11, 12—22).—Semi-acid brick for linings for casting high-grade steels must contain additions of a highly refractory plastic clay with a sintering point of  $1110-1200^{\circ}$ . A slowly regenerating cryst. quartz containing no dust passing 900 mesh/sq. cm. must also be added. The composition of the mass is : binding clay 40, grog (0.5—1.0 mm.) 20, cryst. quartz (0.13— 2 mm.) 40%. Test requirements are described.

Сн. Авз. (е)

Apparatus for investigating the corrosive action of slags on refractory materials. A. E. J. VICKERS and R. A. BELL (J. Soc. Glass Tech., 1935, 19, 151— 155 T).—The bricks to be tested form the vertical inner surface of a cylindrical furnace which is heated by a vertical rotating burner. The powdered slag is fed into the burner and impinges against and runs down the face of the bricks. The molten slag leaves the furnace through a central hole in the hearth.

# Soapstones.-See XI.

**法部的主义** 

J. A. S.

#### PATENTS.

Glass furnace. C. C. KINKER, Assr. to OWENS-ILLINOIS GLASS CO. (U.S.P. 1,980,344, 13.11.34. Appl., 26.1.31).—In a tank furnace the gathering pool has a floor sloping to a deepest point under the place where the glass is sheared. B. M. V.

Manufacture of glassware. S. B. BOWMAN and F. C. FLINT, Assrs. to HAZEL-ATLAS GLASS CO. (U.S.P. 1,980,373, 13.11.34. Appl., 5.5.31).—In a glass tank of moderate size the batch is first melted in a rotary drum (described). B. M. V.

Production of laminated glass. G. B. WATKINS, Assr. to LIBBEY-OWENS-FORD GLASS Co. (U.S.P. 1,960,520, 29.5.34. Appl., 27.3.29).—The two glass layers are separated by a thin film of cellulose acetate coated on both sides with a mixture of  $OH \cdot CH_2 \cdot CH_2 \cdot OEt$ 40, the corresponding Bu ether 30, and Bu<sub>2</sub> phthalate 30%. A. R. P.

**Production of safety glass.** G. B. WATKINS, Assr. to LIBBEY-OWENS-FORD GLASS Co. (U.S.P. 1,980,510, 13.11.34. Appl., 26.2.31).—After manufacture of the glass by known methods up to and including the application of the sealing material around the edges, the whole is subjected to fluid pressure. B. M. V.

Manufacture of cellular clay products. C. S. MATHENY, ASST. to NAT. FIREPROOFING CORP. (U.S.P. 1,980,954, 13.11.34. Appl., 7.10.32).—A fluid slip is made of clay, gypsum, dolomite, and an acid solution (the last two to release gas), also a substance (preferably combustible) that will form a paste to confine the bubbles around each dolomite particle and so form small cells. The articles are dried to harden and fired. B. M. V.

Manufacture of glazed refractory articles. R. C. BENNER and H. N. BAUMANN, JUN., Assrs. to CARBORUN-DUM Co. (U.S.P. 1,975,069, 2.10.34. Appl., 12.11.28).— SiC refractories are protected from oxidation at red heat by the application of a glaze having a maturing range of 900—1500°. Carbonaceous matter is burnt out before the glaze begins to mature. The glaze may consist of (a) AlF<sub>3</sub>,3NaF or Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub> 15 + Al<sub>2</sub>O<sub>3</sub> 85%, (b) PbO 2·5 + CuO 2·5 + SiO<sub>2</sub> 95%, or (c) Na<sub>2</sub>SiO<sub>3</sub> 5 + BaSiO<sub>3</sub> 95%. J. A. S.

Granular [abrasive] material. H. R. POWER, Assr. to CARBORUNDUM Co. (U.S.P. 1,980,432, 13.11.34. Appl., 25.2.33).—The abrasive qualities of garnet are improved by heating to incipient fusion (900°) in a nonoxidising atm. of, e.g.,  $N_2$ ,  $H_2$ , or  $CH_4$ . B. M. V.

## IX.—BUILDING MATERIALS.

Asbestos. V. P. ALEXANDROV (Trans. Inst. Econ. Mineral. U.S.S.R. [10-year vol.], 1933, 91--123).--Chemical and mechanical properties of asbestos from various formations are examined. The mechanical properties of asbestos cements are attributed to its colloidal properties and high adsorptive power. The latter is comparable with that of hydraulic materials. Colloidal solutions of asbestos in  $H_2O$ -glycerol are obtained by comminution in org. media ( $C_6H_6$ , glycerol, etc.). CH. ABS. (p)

Lead glycerate and litharge-glycerol cement. H. NITSCHMANN (Helv. Chim. Acta, 1935, 18, 759– 781).—Pb glycerate (I) is the only definite compound and is formed by the reaction  $C_3H_8O_3 + PbO =$  $C_3H_6O_3Pb + H_2O$ . In the formation of the cement

this reaction does not go to completion, due to the formation of protective sheaths of (I) around the PbO particles, and the high viscosity of glycerol. The solubility of (I) in aq. glycerol at  $25^{\circ}$  increases with increasing concn. of glycerol, whilst the conductivity decreases. The variation of setting time with composition and with dispersity of PbO has been investigated.

E. S. H.

Chemical properties of mucilage of alga, used as the cementing material of Japanese walls. T. TADOKORO and T. SATTO (J. SOC. Chem. Ind., Japan, 1935, 38, 270 B).—The mucilage contains fucosan and galactan with some araban, with  $H_2SO_4$  present as sulphate of the polysaccharide. F. R. S,

Behaviour of high-early-strength cement concretes and mortars under various temperatures and humidity conditions. L. Schuman and E. A. PISAPIA (J. Res. Nat. Bur. Stand., 1935, 14, 723-747).-Twelve commercial cements of varying chemical and compound compositions have been tested under different conditions of setting. In most cases the strength of the concrete was a linear function of the cement/H<sub>2</sub>O ratio for any particular age or storage conditions. Concrete cylinders stored at 32° developed about double the 1-day strength of cylinders stored at 21°; storage at 43° still further increased the strength, but at ages > 3 days high storage temp. reduced the strength slightly. After 1 day storage in a damp atm. gave higher strengths than storage in a dry atm. ; 300 cycles of freezing and thawing reduced the strength of cylinders of all cement/H<sub>2</sub>O ratios during the first day to about 10% < that of cylinders stored for 1 year in a damp room at 21°. Cycles of freezing, thawing, drying, and soaking caused spalling and reduction in strength in most cases. Damp storage produced expansion of 0.001-0.018%, whereas dry storage produced contraction of 0.025-0.053% in a year and alternate freezing and thawing expansion of 0.003-0.031%. The total heat evolved in adiabatic storage of concrete with a cement/ $H_2O$  ratio of 1.5 was 10-60 g.-cal./g. in 8 hr., 60-100 in 24 hr., and 104-130 in 87 days. About  $\frac{1}{4}$  of the original H<sub>2</sub>O content of concrete cylinders stored adiabatically and hermetically sealed still remained uncombined after 87 days. A. R. P.

Cement hardening : an electrostatic phenomenon. II. L. JESSER (Zement, 1934, 23, 677-682, 691-694 ; Chem. Zentr., 1935, i, 1105 ; cf. B., 1935, 547).—A discussion. H. J. E.

Resistant mortars applicable to the calcium chlorate industry. M. V. BORODULIN and R. I. ROMANOVSKAJA (Trans. State Inst. Appl. Chem. U.S.S.R., 1934, No. 21, 24—28).—Mixtures of Portland cement with sand, kieselguhr, felspar, SiO<sub>2</sub> gel, kaolin, or andesite powder, wetted with H<sub>2</sub>O or Na silicate (I), were used in tests for lining towers in the Ca(ClO<sub>3</sub>)<sub>2</sub> industry. Samples of the cement were placed in aq. Ca(OH)<sub>2</sub>, and Cl<sub>2</sub> was passed at 80—85°. Samples bonded with (I) were most resistant, 4—8% of clay being added to retard setting. CH, ABS. (e)

Development of proposed standards and tests for plaster of Paris. S. J. McDowell (Bull. Amer. Ceram. Soc., 1935, 14, 229-236).—Proposed specific-

ations for terra-cotta and pottery plasters are: (1) composition,  $\lt$  85% and 90% of calcined CaSO<sub>4</sub>,2H<sub>2</sub>O respectively; (2) setting time, according to needs but usually 20-40 sec. (A.S.T.M. test); (3) fineness, 100% through 28- (Tyler) and  $\lt$  94% through 100-mesh; (4) dry modulus,  $\lt$  700 and  $\lt$  900 lb./sq. in. respectively; (5) soaked modulus,  $\ll 400$  and  $\ll 500$  lb./sq. in. respectively. The plaster is mixed to a standard testing consistency by the following method : 300 g. of plaster are sifted through 8-mesh into H<sub>2</sub>O (15-30°), kept for 2 min., stirred with a spatula for 1 min. (120 r.p.m.), poured into a dry brass cylinder (2-in. bore  $\times$ 4 in.) standing on dry glass plate and the cylinder is at once lifted vertically, allowing the plaster to slump. The mixture is of standard consistency when the resultant pat has a diam. of 6 + 0.25 in. J. A. S.

Experiments with road mixes and surface treatments in California. T. E. STANTON and J. T. PAULS (Public Roads, 1935, 15, 253—270).—Tests are described on liquid asphaltic materials of the slow-curing type to determine their suitability in humid climates.

CH. ABS. (e) **Permeability of building and heat-insulating materials to air.** E. RAISCH and H. STEGER (Gesundheitsing., 1934, 57, 553—556; Chem. Zentr., 1935, i, 1289).—Air-permeability measurements at varying pressures are recorded for 28 materials. H. J. E.

Sap stains of Japanese woods. III. Ceratostomella piceæ, Münch, the cause of a blue stain of pine trees. Y. NISIKADO and K. YAMAUTI (Ber. Ohara Inst. landw. Forsch., 1935, 6, 539-560).—The fungus does not grow in aq.  $CuSO_4$  (1:5000) or aq.  $HgCl_2$ (1:10,000). The growth rate on culture media is  $\leq$ that of *C. pini* and *C. ips*, and O is necessary for mycelial growth and conidium germination. Immersion in  $H_2O$ at 50° for 15 min. or in aq.  $CH_2O$  (1:200) and aq.  $HgCl_2$ (1:4000) for 1 hr. kills both conidia and ascospores. P. G. M.

Coal-tar creosote oil as a wood preservative. N. A. RICHARDSON (J. Brit. Wood Pres. Assoc., 1934, 6, 64—91).—A discussion. CH. Abs. (e)

Qualities of Bakelite-treated wood. E. WEGELIUS (Tekn. Aikakaus., 1934, 24, 227-236).—Thin veneers impregnated and glued together with Bakelite give a material suitable for aeronautical use. The homogeneity and strength are better than for ordinary wood, troubles due to moisture changes are eliminated, and the wood resists fungi and bacteria. CH. ABS. (e)

Use of treated timber in railway stock construction. W. H. BROWN (J. Brit. Wood. Pres. Assoc., 1934, 6, 45-63).—The practice of the L.N.E.R. is described. CH. ABS. (e)

## PATENTS.

(A) Concrete and hydraulic cement (B) Coloured concrete. DEWEY & ALMY, LTD. (B.P. 431,679 and 431,680, [A] 7.11.33, [B] 8.11.33. U.S., [A] 21.11.32, [B] 12.12.32).—(A) Products of the type  $A \cdot R \cdot A'$ , where A and A' are aromatic nuclei (polynuclear), and R is  $\ll 1$  alkyl group, containing at least one SO<sub>3</sub>H (condensation product of 2-C<sub>10</sub>H<sub>7</sub>SO<sub>3</sub>H with CH<sub>2</sub>O), are added (0·1—0·3%) to structural concrete

mixes. Cement-peptising action, resulting in higher plasticity using less  $H_2O$ , without loss in strength, is claimed. [Stat. ref.] (B) Finely-divided coloured pigments, e.g., C black,  $Fe_2O_3$ , are incorporated in cement mixes as above; weakening effects of pigments are reduced. A. W. B.

[Heat-]insulating cement. H. N. CLARK (U.S.P. 1,959,658, 22.5.34. Appl., 30.11.31).—A mixture of mineral wool 45—65 (50), kieselguhr 10—30 (19—75), bentonite 11—18 (18), fibrous asbestos 5—20 (10), CaO  $\geq$  5 (2), and Al powder  $\geq$  1 (0.25)% is claimed. A. R. P.

Heat-insulating material. G. M. THOMSON (U.S.P. 1,980,889, 13.11.34. Appl., 21.12.31).—A composition suitable for pouring into wall spaces or the like comprises exfoliated vermiculite 40—50 and ground gypsum wallboard 60—50%. B. M. V.

Plastic composition for manufacture of conduits and other articles of manufacture by extrusion. G. S. THURMAN and K. T. ROMIE, ASSTS. to FIREPROOF WALL CO. (U.S.P. 1,977,158, 16.10.34. Appl., 10.9.30). —A setting material, *e.g.*, cement (10), a fibrous material, *e.g.*, asbestos (9), and a clay (3—4 pts. by vol.) are mixed with a liquid to the consistency of a "dry mix," which is alternately subjected to pressure, *e.g.*, by extrusion, and mixing, and the final coherent and homogeneous mix is extruded through a shaping die to the desired form. S. S. W.

Substances for surfacing of roads. Soc. DE RECHERCHES ET DE PERFECT. INDUSTR. (B.P. 430,061, 8.6.34. Switz., 12.6.33 and 24.5.34).—The material consists of an aq. emulsion of tar containing powdered coal as filler. Soap etc. is used as an emulsifying agent, and carragheen jelly etc. as stabilising agent. Preferably a small proportion of NaOH is also added. A. B. M.

[Moulding] method and apparatus for production of bricks, blocks, tiles, etc. G. HEAD (B.P. 432,799, 27.1.34).

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

Use of potassium dichromate in the determination of the iron content (total iron content and ferrous oxide) in iron ores and silicates. A. M. ZANKO and A. L. DAVIDOV (Ber. Ukrain. wiss. Forsch. physikal. Chem., 1935, 4, 89–98).—Conditions are given for the titration of Fe<sup>•</sup> with  $K_2Cr_2O_7$  potentiometrically and using the NHPh<sub>2</sub> indicator. Fe<sup>••</sup> is reduced by SnCl<sub>2</sub> and the excess removed by HgCl<sub>2</sub>. The amount of Hg<sub>2</sub>Cl<sub>2</sub> formed must be small, otherwise the NHPh<sub>2</sub> end-point is affected. Insol. silicates are treated with HCl + HF and the titration is carried out as above. The val. of the equivalence e.m.f. is affected by the presence of F' but is independent of Fe<sup>•••</sup>.

R. S.

Preparation of cupola cast iron alloyed with tin and copper. V. V. SKORCHELETTI and A. I. SHULTIN (Soobsch. Vsesoy. Inst. Met., 1931, No. 8, 29–34).— The Cu and Sn (2:1) are introduced in the ladle, and the cast Fe is added and mixed. The product should contain C 3.5, Si 1.5, Mn 0.5%, and as little S and P as possible. Sn-Cu cast Fe is exceptionally resistant to 10% H<sub>2</sub>SO<sub>4</sub> at room temp. CH. ABS. (e)

Influence of copper on grey cast iron. E. SÖHN-CHEN and E. PIVOVARSKY (Giesserei, 1934, 21, 449–453; Chem. Zentr., 1935, i, 1113).--0.5-2.0% of Cu in grey cast iron improves the mechanical properties, but does not affect corrosion-resistance except that to AcOH, which is improved. < 1% lowers and > 1% of Cu raises the thermal and electrical conductivity. The solubility of Cu is 0.5-1%. H. J. E.

Influence of aluminium on the properties of cast iron. M. PLOVÉ (Rev. Mét., 1935, 32, 248-270).— Addition of Al up to 0.02% to a cast Fe (C 3.27, Si 2.0, Mn 0.70, P 0.54, S 0.06%) enhances the casting properties. Depth of chill is decreased as Al up to 0.25% increases. The effect on microstructure is small and varies with the casting temp., if Al  $\geq 0.2\%$ . From Al 2 to 10% the amount of free graphite decreases, from 10 to 15% no free graphite is present, and from 15 to 20% graphitic C increases. The character of the pearlite is also modified if Al is  $\geq 2\%$ . W. P. R.

Vacuum distillation of metals. Refining of beryllium and iron. W. KROLL (Metal Ind., 1935, 47, 29-32).—Ductile Be cannot be produced by evaporation in high vac. although the distilled Be has no inclusions in the grain boundaries. Purification of Fe is attained by distilling off Mn, Si, Cu, P, and S which are conc. in the first part of the distillate. Ni, Co, and W remain in the residue. W. P. R.

Removal of sulphur from iron and steel melts. J. GØRRISSEN (Tidsskr. Kjemi, 1935, 15, 50–53, 71–74). —A quant. discussion of the solubility of sulphides in the molten metal, especially in the presence of Mn and C, and of the solubility of MnS and FeS in slags of various compositions. R. P. B.

Production of steel in the blast furnace combined with the smelting of acidic ores. M. FRÄNKL (Stahl u. Eisen, 1935, 55, 805–809).—Poor-quality siliceous ore is smelted with additions of Fe oxide and is added in a special refining furnace to the product of a modified blast furnace. The process produces a low-C, low-Si steel. W. P. R.

Nitrogen case-hardening of steels. S. NISHIGORI (Tech. Rep. Tôhoku, 1935, 11, 623-681).-The process has been investigated on 95 steels containing Al. Cr. Mo, Ni, and C by measurements of the hardness and wt. increment, and by X-ray and micrographic examination after heating in NH<sub>3</sub> at 500-600° for 25-100 hr. The dissociation of NH<sub>3</sub> due to the catalytic action of the surface layers slowly increases to a const. val. in 15 hr. at 500° and in < 3 hr. at 600°; the consumption of NH<sub>3</sub> at 550° is about 3 times that at 500°. Wt. increment is greatest in Al-Fe alloys and least in Ni-Fe alloys, in which latter no case-hardening occurs. The microstructure of nitrided Al-Fe and Mo-Fe alloys shows a Widmannstätten structure of an acicular compound in a deep brown matrix. Nitrided *a*-Fe has a network structure, but no needles of nitride are visible ; the presence of even very small amounts of C refines the grain structure and toughens the nitride layer. In Al-Cr steels the depth of the nitride layer decreases with increasing C content, but with const. Al and Cr the hardness is not affected by C; in absence of C these alloys tend to disintegrate on nitriding, owing to the formation of a N-rich ternary nitride which forms at the grain boundaries with a considerable increase in vol. A. R. P.

Ferro-alloys as additions to special steels.— KÄRSTEN (Chem.-Ztg., 1935, 59, 556—557).—Two types of electric furnace for the production of C-free ferrosilicon, -manganese, -chromium, -tungsten, and -molybdenum are illustrated and their operation is briefly described. A. R. P.

Physical chemistry of steel making. P. HERASY-MENKO and E. VALENTA (Trans. Amer. Foundrymen's Assoc., 1934, 5, No. 6, 21–47).—A general discussion. CH. ABS. (e)

Etching agent for coarse-structure investigation [of steel]. A. MATTING (Autog. Metallbearb., 1934, 27, 253-255; Chem. Zentr., 1935, i, 1115).—The solution contains 25 c.c. of  $H_2O$ , 3 g. of  $CuNH_4Cl_2$ , 50 c.c. of conc. HCl, and 15 g. of FeCl<sub>3</sub>. H. J. E.

Influence of non-metallic inclusions on the precipitation of primary cementite in hypereutectoid steels. E. G. MAHIN and E. F. LEE (Trans. Amer. Soc. Met., 1935, 23, 382–397).—Non-metallic inclusions affect the locus of the primary Fe<sub>3</sub>C network in hypereutectoid steels in a manner similar to the pptn. of primary ferrite in hypoeutectoid steels. Dissolved impurities segregated in the austenite near the inclusions cause the beginning of the primary phase formation.

Embrittlement of steels due to soaking at high temperatures. D. A. N. SANDIFER (Engineering, 1935, 140, 81-82).—Notched-bar impact vals. of steel containing C 0.33, Mn 1.34, Si 0.17%, which had been tempered at 575° or 600°, progressively fell during exposure at 450° from 60 to 10 ft.-lb. in 5 days. A similar steel with addition of Mo (0.2%) and also two Ni-Cr-Mo steels did not become similarly embrittled. E. H. B.

**Rapid determination of oxides in liquid steel.** M. BABSKY (Aciers spéc., Mét., Alliages, 1934, 9, 228–229; Chem. Zentr., 1935, i, 1277–1278).—Al is added to the molten steel, forming  $Al_2O_3$  with dissolved  $O_2$ . The steel is dissolved in 30% HNO<sub>3</sub> + (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub>, and the Al determined. J. S. A.

Sulphur determination [in steel] by the combustion method. H. A. KAR (Ind. Eng. Chem. [Anal.], 1935, 7, 244—246).—The sample is burned in  $O_2$  and the resulting  $SO_2$  and  $SO_3$  are absorbed in  $Na_2O_2$  solution. S is then determined as  $BaSO_4$  by pptn. with  $BaCl_2$ . E. S. H.

Determination of sulphur in ferromagnetic alloys. B. L. CLARKE, L. A. WOOTEN, and C. H. POTTENGER (Ind. Eng. Chem. [Anal.], 1935, 7, 242—244). —Fe-Ni or Fe-Ni-Co alloys are heated in H<sub>2</sub> at 1100—1200°, and the liberated H<sub>2</sub>S is absorbed in aq.  $Cd(NO_3)_2 + NH_3$ . For the range 0.005-0.02% S the accuracy is  $\pm 0.001\%$  S on the basis of a 10-g. sample. E. S. H.

Errors in quantitative spectrographic analysis of the iron-silicon system. G. SCHEIBE and A. SCHÖNTAG (Arch. Eisenhüttenw., 1934-5, 8, 533-540).-The magnitude of the errors introduced into the spectrographic determination of Si in Fe and steel by incorrect set-up of the optical system, variations in the method of excitation, and variations in the photographic and photometric technique are discussed, together with means for their prevention. The three-line method can be improved by the use of a Pt strip across the lens, which reduces the intensity of the lines over half their length to  $\frac{1}{4}$  of that over the other half; in this way only one line of Fe (2518 Å.) and one of Si (2516 Å.) are necessary for a measurement of the relative intensity, which may be made in both the strong and the weakened half of the spectrogram. Rigid adherence to standard conditions of self-induction and resistance are essential for good results since the relative intensity of the two lines remains const. only over a narrow range of electrical conditions. With a standardised technique throughout the analysis, details of which are given, Si can be determined to an accuracy of  $\pm 3\%$  in steel,  $\pm 9\%$  in chill castings, and  $\pm 11\%$  in grey cast Fe when the abs. amount varies between 0.2 and 1%.

A. R. P. Modified persulphate-arsenite method for [determination of] manganese, with special reference to steel analysis. E. B. SANDELL, I. M. KOLTHOFF, and J. J. LINGANE (Ind. Eng. Chem. [Anal.], 1935, 7, 256—258).—The HMnO<sub>4</sub> formed by the oxidation of the sample with  $(NH_4)_2S_2O_8$  in presence of  $H_3PO_4$ , using Ag as catalyst, is titrated with a reducing solution containing equiv. amounts of Na<sub>3</sub>AsO<sub>3</sub> and NaNO<sub>2</sub>. Mn<sup>VII</sup> is reduced to Mn<sup>II</sup> and the solution becomes colourless. Small amounts of Cr, V, Ni, and Mo do not interfere. Ag is pptd. as AgCl before the titration.

E. S. H.

Photometric titration. Application of the coppercuprous oxide cell and determination of vanadium in special high-speed steel. T. Sômiya and Y. NAKAMURA (J. Soc. Chem. Ind., Japan, 1935, 38, 262-266 B).-An improved form of an earlier apparatus (cf. A., 1930, 1263) is described. Two beams of light are passed through filters and two samples of solution, and fall on two photo-cells connected to give opposing currents in a galvanometer. Applications to the titration of FeSO4 by KMnO4 and Cu by Na2S2O3 are given. The light filter used depends on the colour of the solution, since the photo-cell is most active at approx. 6500 Å. 0.6% of V in steel containing W, Cr, Co, and Mo may be rapidly and accurately determined by dissolving in acid and titrating with KMnO<sub>4</sub>. It is claimed that the precision and sensitivity are > for ordinary methods, and deeply coloured solutions can R. S. B. be used.

Influence of alloy elements on the transformation of austenite and the tensile properties of alloy steels in stepped heat-treatment. H. DÖPFER and H. J. WIESTER (Arch. Eisenhüttenw., 1934—5, 8, 541—548).—The rate of transformation of quenched austenite in steels containing 2—3% of Cr, Si, Mo, W, V, Mn, or Ni has been determined on tempering at 150—600°. In all cases the rate increases with rise in temp. from above the martensite point to a max. at 300—400° and then decreases; with Cr, Mo, and V

W. P. R.

steels there is a range of comparative stability of austenite at about 500°, but in the pearlite field the rate of decomp. again increases rapidly with rise in temp. With Si steels the rate increases rapidly throughout the temp. range and with Mn, Ni, and W steels it decreases again only above 550°. The occurrence of a temp. range of austenite stability in Ni-Cr steels is therefore due to their Cr content. The transformation of austenite in most of the steels tested is preceded by a period of induction which is relatively long at low tempering temp. and in a small temp. range about 500°, and is dependent to a great extent on the alloying element and the % C present. Mechanical tests on specimens subjected to a stepped heat-treatment show that the same properties can be produced in this way as by the ordinary single-stage tempering, the only advantage of the stepped method being that internal stresses and the development of hardening cracks are reduced to a min. A. R. P.

Copper-containing steels for dynamos and transformers. V. S. MESSKIN (Soobsch. Vsesoy. Inst. Met., 1931, No. 8, 53-65).—> 0.8% and 0.6% of Cu in dynamo and transformer steels respectively exists in solid solution. This addition of Cu greatly increases the resistance to corrosion. The hardness is increased 8-10% for steel with 1% Cu. Magnetic and electrical properties are practically unchanged. CH. ABS. (e)

Improvements in weather-resisting steels. K. DAEVES (Arch. Eisenhüttenw., 1935—6, 9, 37—40).— Addition of > 0.06% P to steels with low Cu or Cu + Sn contents reduces considerably the loss by rusting on exposure to the air; the rust film formed is dark brown, smooth, strongly adherent, and free from excrescences and has a relatively high protective power. In the absence of Cu a high P content is protective only in an inland atm. The blue rolling scale on Cu–P steels flakes off more readily than that on ordinary steels on exposure to the weather, and the Cu–P steels behave better under a paint coating than do the ordinary steels. A. R. P.

Rusting and scaling of constructional steels under tensile stresses. E. W. MÜLLER and H. BUCHHOLTZ (Arch. Eisenhüttenw., 1935—6, 9, 41—45).— The rate of rusting of constructional steels with small quantities of Cu, Cr, Ni, and Mo in the ordinary atm. is unaffected by tensile stresses, even those which produce a permanent elongation of 5%. In running H<sub>2</sub>O, however, the rate of rusting is somewhat increased by the application of stresses. Scaling is unaffected by stresses which are insufficient to replace the scale or to cause it to flake off. A. R. P.

Rolling and recrystallisation texture of nickeliron alloys. W. G. BURGERS and J. L. SNOEK (Z. Metallk., 1935, 27, 158—160).—The rolling texture of the 50:50 Fe–Ni alloy is characterised by a scattering of the crystallites about all positions with the [335] direction parallel to the direction of rolling and the (135) plane in the plane of rolling. The recrystallisation texture is a true cube texture with a scattering of only  $5-10^\circ$ ; recryst. sheet has anisotropic magnetic properties, the [100] direction being the most easily magnetised (cf. following abstract). A. R. P.

Rolling and recrystallisation texture in ironnickel alloys in relation to the magnetic properties. F. PAWLEK (Z. Metallk., 1935, 27, 160-165).-The rolling texture of reversible Fe-Ni alloys consists of (110) in the rolling plane and [112] in the rolling direction, while the recrystallisation texture consists of (010) in the rolling plane and [100] in the rolling direction and appears after a reduction in thickness of > 90%, the actual amount necessary depending on the purity. Under certain annealing conditions a very coarse crystal structure is produced from alloys with a fibrous structure by heating at  $> 1100^{\circ}$ . The recovery of the mechanical and electrical properties of worked alloys follows a different course according to whether the recrystallisation leads to a re-formation of the regular orientation of the crystallites or to the development of a fibre structure. The cube texture remains const. during deformation up to 50-70% reduction, and subsequent annealing after a min. of 15% reduction leads to the restoration of a random crystallite orientation. The cube texture produced by cold deformation is of considerable technical importance since in this state the alloys exhibit the isoperm behaviour in the direction of rolling.

A. R. P.

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Alternating-current corrosion. I. S. JELLINEK. II. H. HOHN (Elektrotechn. u. Maschinenbau, 1934, 52, 577—580, 580—583; Chem. Zentr., 1935, i, 1119).— I. Observations of corrosion in water-pipes and gas meters are recorded.

II. Corrosion increases with voltage, c.d., and wavelength, log. corrosion being  $\infty$  the normal potential of the electrode metal. If anions which can form H<sub>2</sub>O-sol. compounds with the electrode material are present, passivity may occur. H. J. E.

Atmospheric exposure tests on non-ferrous screen wire cloth. G. W. QUICK (J. Res. Nat. Bur. Stand., 1935, 14, 775-793) .- Wire screens of the following have been subjected to atm. corrosion for 9 years at four localities: (a) 90:10 Cu-Zn, (b) 80:20 Cu-Zn, (c) 75:20:5 Cu-Ni-Zn, (d) 70:30 Ni-Cu, (e) Cu, (f) 98 : 2 Cu-Sn, (g) 95 : 5 Cu-Al. Failure at Pittsburgh occurred with d in 48 months, q 58, b 76, c 83, and a, e, and f 90 months. Failure of b occurred in 48 months in a temperate, and in 54 months in a tropical, sea-coast atm. No failures occurred at Washington, a mildly corrosive inland atm. Accelerated corrosion tests in the laboratory gave results from which no prediction of the behaviour in outdoor exposure could be made. A. R. P.

Marine corrosion. J. W. DONALDSON (Metallurgia, 1935, 12, 117-118).—A brief review.

[Examination of] mineral products. I. P. ALIMARIN (Trans. Inst. Econ. Min. U.S.S.R., 1933, 267—286).—A review of available methods.

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Deactivation of pyrites. M. MORTENSON (Tidsskr. Kjemi, 1935, 15, 69—70).—From investigations of the effect of various substances in hindering flotation it is concluded that Ca ions have no special activity in this respect. R. P. B.

Concentration of antimony ores from the Kadam-Dzhai deposit. L. M. ALEXEEV (Redk. Met., 1934, 3,

No. 5, 27–39).—The material was a partly oxidised sulphidic ore (Sb  $3\cdot 10$ , SiO<sub>2</sub> 85%). Table concent or flotation gave a concentrate with  $\gg 30\%$  Sb (55–65% recovery). The Sb was finely disseminated and existed partly as oxide. CH. Abs. (e)

Concentration by flotation. IX. Treatment of concentrates. A. FURIA (Chim. e Ind., 1934, 2, 83-85; Chem. Zentr., 1935, i, 1112; cf. B., 1934, 303).—The yield from cyanide treatment is improved by concentrating and roasting the concentrate. H. J. E.

Use of chlorine gas in non-ferrous metallurgy. G. G. URAZOV and I. S. MOROZOV (Trans. State Inst. Appl.Chem.U.S.S.R., 1934, No.20, 7—41).—Chlorination is best applied with sulphide ores at  $300-700^{\circ}$ , and with mixed ores containing sulphides and oxides, in the presence of H<sub>2</sub>O. CH. ABS. (e)

Treatment of uranium-containing solutions with metallic iron. I. J. BASCHILOV (Redk. Met., 1934, 3, No. 2, 25—33).—Solutions remaining from the production of Ra were treated with scrap Fe to ppt. Cu. With acidity approx. 0.5N, Cu free from U was pptd. With acidity 0.1N at  $90^{\circ}$ , U free from Cu was pptd. Strict control of acidity is necessary in the commercial application of this method. CH. ABS. (e)

Atmospheres for annealing high[-copper] brass in relation to buffing. L. KROLL and E. A. ANDERSON (Met. & Alloys, 1935, 6, 192–193).—Brass (66% Cu) tends to be covered after annealing and pickling by loose (containing C) and soft (Cu-rich) layers. The loss of wt. during the buffing away of these layers is less when annealing is carried out in dry  $H_2$  than in air, but might be higher in presence of roll oil. E. H. B.

Lead bronzes with special reference to manganese as an alloying element. J. WECKER and H. NIPPER (Z. Metallk., 1935, 27, 149-154).-Mn is more satisfactory than Sn as an alloying element for Cu in the prep. of heavily leaded bearing bronzes; whereas with 30% Pb segregation commences with only 5% Sn in the Pb-Sn-Cu alloys, > 7% Mn may be present in the Pb-Mn-Cu alloys before the castings become nonhomogeneous. Addition of 2% Ni to the 30% Pb-7% Mn alloy improves the ease of melting and facilitates the casting of large ingots, but better results are obtained by reducing the Mn to 5%. Sn and Mn have a similar hardening effect; Ni hardens only in the presence of Sn or Mn. Small amounts of As increase the hardness but P is without effect. Mn-Ni-Pb bearing bronzes have good sliding properties, a high resistance to wear and impact, and are much superior in strength to white metals. A. R. P.

Aluminium-copper drawn products. A. VIVANTI (Alluminio, 1934, 3, 268–280; Chem. Zentr., 1935, i, 1115).—The structure of Al–Cu wires of various compositions was studied after heating at 1120°, 950°, and 850°. At 1120° Stockdale's results were partly confirmed (cf. B., 1924, 299), and the  $\alpha + \beta$ , pure  $\beta$ , and  $\beta + \delta$  phases were also found as equilibrium components. H. J. E.

Black spots on tin and tinned ware. S. BRENNERT (Tech. Publ. Int. Tin Res. Dev. Counc., D, No. 2, 27 pp.). —The black spots which sometimes form on tinned dairy

equipment have been reproduced in simple salt solutions (e.g., NaCl). They consist mainly of SnO and are formed by corrosion in accordance with electrochemical principles. Black spots do not begin to form until the Sn surface reaches a crit. " dissolution potential," the val. of which varies with the composition of the solution, but the spots can continue to grow at less noble potentials. The potential of a Sn surface may rise to a val. > the dissolution potential through the further growth of the air-formed oxide film in neutral or nearly neutral solutions; at weak points in this film black spots are produced on Sn. The formation of black spots can be prevented by keeping the Sn in contact with a less noble metal (e.g., Al or Zn), thus ensuring that the potential is < the dissolution potential. E. S. H.

Impact torsion tests. II-IV, M. ITIHARA (Tech. Rep. Tôhoku, 1935, 11, 489-511, 512-527, 528-581; cf. B., 1933, 1061).-II. An apparatus for measuring the twisting moment and angle of twist ( $\theta$ ) of metal rods under impact torsion is described and impact and static torsion diagrams are given for Sn, Pb, Cu, Al, Zn, brass, duralumin, and several ferrous alloys. The curves are almost coincident for face-centred cubic metals, but for body-centred cubic Fe alloys  $\theta$  in the dynamic test is about 10-20% > in the static test and the yield point about 3 times as great. The dynamic sliding resistance of a metal is the sum of the elastic resistance and the viscous resistance. The shape of the impact diagram of Y-Fe alloys is ascribed to the decrease of elastic resistance and the coeff. of viscosity  $(\mu)$  with increase in sliding speed owing to increasing at. disorder; with the face-centred cubic metals the sliding planes are of greater symmetry and the static and dynamic curves are therefore parallel.

III. The apparent  $\mu$  of various metals determined from the difference between the moments in the impact and static diagrams, are in kg.-sec. per sq. cm. (for sliding speeds of 1700 r.p.m.): brass 5.27, Al 3.28, Zn 2.55, Cu 1.90, duralumin 0, lead 0.06, Armco Fe 20-24, C steel 18-40, Mn steel 5-9, armour-plate steel 38-50. The impact torsion diagrams of coldworked and aged metals show higher yield points than those of annealed metals to an extent which is greater the greater is the degree of ageing.

IV. The impact and static torsion diagrams of Armco Fe, mild steel, and 0.3% C Swedish steel have been determined at  $-160^{\circ}$  to  $1100^{\circ}$ . The curves show marked differences above and below the range of blue-brittleness. The effect of age-hardening on the curves is considered. A. R. P.

Transition structures in fusion welding with austenitic welding rods. F. RAPATZ and W. HUM-MITZSCH (Arch. Eisenhüttenw., 1934—5, 8, 555—556).— The use of austenitic welding rods results in the production of workable welds which are scarcely affected by thermal treatment; this is particularly advantageous when the material welded is strongly hardened by heattreatment. The transition zone between weld and basis metal is usually martensitic unless the basis metal is a mild steel, when ferritic areas may occur; in high-C steels the austenite of the weld metal penetrates the grain boundaries of the base. Ni-austenite gives tougher transition zones than Mn-austenite, probably because, owing to the slower rate of diffusion of Ni, the martensitic zones are relatively thin. The presence of Cr and Mo assists the fusibility in welding but has little effect on the weld structure. Mn alloy welding rods tend to produce hardening and contraction cracks due to the sensitivity of the alloy to overheating and to its greater coeff. of expansion. A. R. P.

Physical properties of deposited metal in the arc-welding and atomic hydrogen processes. L. MILLER and C. R. DEGLON (Sheet Met. Ind., 1934, 8, 131-133, 189-191, 252-253).—A discussion. Short arc welding with high-quality electrodes and the at. H process yields deposits which have no age-hardening tendencies. Ni has no marked detrimental effects O produces low impact vals. in the product.

Significance of anode and cathode efficiencies. C. B. F. YOUNG (Steel, 1934, 95, No. 21, 38, 51).—A discussion. In general, anode and cathode efficiency should be equal. CH. ABS. (e)

Electrochemistry applied to electrodeposition. IV. Ostwald's law of dilution. S. FIELD (Metal Ind., 1934, 45, 473-475).—A discussion. CH. ABS. (e)

Mechanism of chromium deposition from the chromic acid bath. C. KASPER (J. Res. Nat. Bur. Stand., 1935, 14, 693-709).—Since relatively large additions of salts of Zn, Ni, Cd, Fe, and Cu do not have the effect on the behaviour of  $CrO_3$  plating baths that would be expected if deposition of Cr occurred from the  $Cr^{III}$  state and cryoscopic and spectrophotometric data show that the dichromates of these metals are strongly ionised, it is concluded that Cr is deposited from  $CrO_3$  plating baths from the  $Cr^{VI}$  state. The low efficiency and high polarisation in Cr deposition are accounted for by the fact that the strong oxidising agent  $(CrO_3)$  exists in an acid solution as a large negative ion. Suggestions are made for improving Cr-plating.

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Electrolysis of cadmium sulphate. O. K. KUDRA (Mem. Inst. Chem. All-Ukrain. Acad. Sci., 1934, 1, 197— 207).—With cylindrical cathodes optimum Cd deposits are obtained with a solution containing  $CdSO_4$  5, acid 0·1, and gelatin 0·01%, using a c.d. of 0·01 amp. per sq. cm. On Al cathodes, dense smooth Cd foil of 4 mm. thickness is obtainable. CH. ABS. (e)

Deposition of zinc-cadmium alloys from alkaline cyanide solutions. L. WRIGHT and J. RILEY (Metal Ind., 1934, 45, 471-473).—Alloys of a wide range of composition were deposited from solutions of the double cyanides with excess of KCN, KOH, and an addition agent "cerelose." Cd additions to the bath were necessary to keep the Zn : Cd ratio const.

Ladle linings .- See VIII.

## PATENTS.

Rotary furnace for treating pulverised ore with gas. O. and J. B. STÅLHANE, ASSTS. to ALLMANNA SVENSKA ELEKTRISKA AKTIEB. (U.S.P. 1,959,772, 22.5.34. Appl., 22.9.32. Swed., 23.9.31).—The furnace comprises a horizontal rotary drum externally heated and provided internally with inclined scoop-like troughs decreasing in capacity towards the discharge end. Ore and hot gas are passed simultaneously into the furnace through a downwardly-inclined pipe at one end, and the rotation of the drum produces an intimate intermingling of ore and gas during the passage through the furnace. A. R. P.

Heat-treatment of metal [steel] articles. M. F. VERITY, Assr. to MASSEY-HARRIS Co., LTD. (U.S.P. 1,959,650, 22.5.34. Appl., 12.8.33).—The edges of ploughshares are hardened by immersing the shares below  $H_2O$  so that the edges are just below the surface, and playing an  $O_2$ - $C_2H_2$  flame along them to heat successive portions to hardening temp., the forward movement of the flame allowing the  $H_2O$  to re-submerge the heated portions and thereby quench them. A. R. P.

(A) Steel. (B) Manufacture of steel. J. T. WHITELEY, Assr. to I. W. HEYMAN and S. L. ZAVON (MULTI STEEL Co.) (U.S.P. 1,959,398—9, 22.5.34. Appl., [A] 10.12.29, [B] 25.2.32. Renewed [A] 28.5.32).—(A) Claim is made for steel containing C 0.05—0.3, W 0.3—0.6 (0.5), V 0.1—0.2 (0.2), Mo 0.75—1.5 (1), Ni 0.5—1 (0.75), Cu 0.3—7.0 (4), Mn 0.8—1.2 (1), Si 0.8—1.2 (1), Cr 0.5—1 (0.75), Zr 0.2—0.5 (0.3), and Ti 0.2—0.5(0.3)%, with traces of B and Co. (B) The molten steel containing all the above elements except Zr and Ti is treated with a ferro-alloy of these, and then with a powerful oxidising agent, *e.g.*, K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, KClO<sub>3</sub>, NaNO<sub>3</sub>, KMnO<sub>4</sub>, or NaBO<sub>3</sub>, to produce a strong exothermic reaction in which the Zr and Ti are oxidised. A. R. P.

Free-cutting steel. H. W. GRAHAM and S. L. CASE, Assrs. to JONES & LAUGHLIN STEEL CORP. (U.S.P. 1,959,758, 22.5.34. Appl., 13.3.33).—Molten steel is treated with  $FeSO_4$  or  $(NH_4)_2SO_4$  to introduce > 0.025%S into the finished steel. A. R. P.

Magnetic materials [aluminium-nickel-iron alloys] and heat-treatment thereof. R. A. CHEG-WIDDEN and E. S. GREINER, ASSTS. to BELL TELEPHONE LABS., INC. (U.S.P. 1,959,999, 22.5.34. Appl., 11.8.33). —The remanence and coercive force of alloys with Al 8—12 (10), Ni 25—30 (25), and Fe 65—60 (65)% are improved considerably by heating at > 1200° and quenching in oil. A. R. P.

Flotation process and reagent [for copper carbonate ores]. L. J. CHRISTMANN, ASST. to AMER. CYANAMID CO. (U.S.P. 1,960,526, 29.5.34. Appl., 11.3.29). —Suitable collectors for malachite and azurite in presence of cresol or Aërofloat frothers are  $\alpha$ -anilinopropionic acid, 2-hydroxy-3-naphthoic acid, cinnamic acid, anthranilic acid, mandelic acid, and phenylglycine. A. R. P.

Copper-base alloy. S. TOUR, ASST. to L. PITKIN, INC. (U.S.P. 1,959,509, 22.5.34. Appl., 14.6.30).—Cu alloys containing Si as a hardener, with  $\measuredangle$  1 of the metals Zn, Mn, Fe, Co, Ni, are melted with 1—6% of Bi and cast under such conditions that the Bi remains evenly disseminated throughout, so as to improve the machinability. A. R. P.

[Molybdenite] ore separation [by flotation]. J. P. RUTH, JUN., Assr. to RUTH CO. (U.S.P. 1,960,459, 29.5.34. Appl., 23.8.32).—Flotation of pyrites and

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other sulphides is inhibited by  $Zn(CN)_2$  and the use of de-aërated  $H_2O$ , and that of  $MoS_2$  promoted by addition of  $NH_3$ . Cresol and xanthates are used as frother and collector, respectively. A. R. P.

Alloys. [Dental amalgams.] D. GRAY, ASST. to ONEIDA COMMUNITY, LTD. (U.S.P. 1,959,668, 22.5.34. Appl., 9.7.32).—Claim is made for amalgams containing 1—50 (5)% In. A. R. P.

Condensation of magnesium vapours. OESTERR. AMERIKANISCHE MAGNESIT A.-G. (B.P. 431,537, 7.4.34. U.S., 7.4.33).—Mg vapours are quickly cooled, by direct contact with an inert liquid (e.g., hydrocarbon oil) which will wet Mg powder, from a high temp. down to a temp. at which Mg no longer reacts with CO and preferably down to approx. room temp. An apparatus of the falling-film type is described. B. M. V.

Magnesium-base forging alloy. J. A. GANN, F. L. REYNOLDS, and A. W. WINSTON, ASSTS. to DOW CHEM. Co. (U.S.P. 1,959,913, 22.5.34. Appl., 29.1.32).— An alloy of Mg with Al 0.5—3.0 (2), Mn 1—0.5 (0.5), and Cd 0.5—6 (2)% is claimed. A. R. P.

Apparatus for plating. K. T. POTTHOFF (U.S.P. 1,959,764, 22.5.34. Appl., 19.10.28).—Means for automatically raising and lowering the racks while the current is on are claimed. A. R. P.

Electrolytic method [for making copper sheet and strip]. R. A. WILKINS, ASST. to INDUSTRIAL DEVELOPMENT CORP. (U.S.P. 1,960,563, 29.5.34. Appl., 30.8.30).—In the continuous process of depositing Cu on a horizontal rotating drum, the surface of the drum consists of Cu coated with a thin film of CuSiO<sub>3</sub> by washing it with a solution of  $H_4SiO_4$  in EtOH.

A. R. P.

Electrodeposition of [iron-nickel] alloys. A. G. RUSSELL, Assr. to BELL TELEPHONE LABS., INC. (U.S.P. 1,960,029, 22.5.34. Appl., 19.11.31).—An electrolyte for the deposition of the 29:71 Fe–Ni alloy contains  $NiSO_4,7H_2O$  212,  $FeSO_4,7H_2O$  22,  $NiCl_2,6H_2O$  18,  $FeCl_2,4H_2O$  2.5,  $H_3BO_3$  25, and  $MgSO_4,7H_2O$  125 g. per hitre, and is operated at 50—60° with Ni and Fe anodes, the effective areas of which are adjusted to maintain a const. Fe : Ni ratio in the bath. A. R. P.

CS<sub>2</sub> reaction products.—See III. Welding rod.— See XI.

## XI.—ELECTROTECHNICS.

Wooden cores for the construction of electric furnaces. L. S. Ts'AI and H. L. CHAO (J. Chinese Chem. Soc., 1935, 3, 196—197).—Divided wooden core forms are described. R. S.

Electrical insulation of baked soapstones. W. Y. CHANG and Y. M. HSIEH (J. Chinese Chem. Soc., 1935, 3, 183–195).—The influence of baking temp. on the vol. resistivity (p) and other properties of soapstone has been studied. p increased from  $10^{6-9}$  to  $10^{8-17} \omega$  with baking. R. S.

Electrical distillation of coal.—See II. Electrofiltration.—See V. Electric furnaces for ceramic firing.—See VIII. Steels for dynamos etc. Removing gases from metals. Cr deposition. Cd electrolysis. Zn-Cd alloys.—See X. PATENTS.

**Electric furnace.** A. LOPPACKER (U.S.P. 1,980,729, 13.11.34. Appl., 26.7.29).—In a combustion assay furnace or the like the heating coil is situated in an annular space between two refractory tubes and is sealed except for supply and exhaust tubes for the maintenance of a non-oxidising atm. around the wire. B. M. V.

**Electric induction furnace.** E. F. NORTHRUP, Assr. to AJAX ELECTROTHERMIC CORP. (U.S.P. 1,980,875, 13.11.34. Appl., 1.1.30).—A metallic tube is surrounded by electrical coils and is heated by currents induced in the tube alone or in the goods and the tube. B. M. V.

[Welding rod for] electric welding. E. M. MARTIN and C. B. LANGSTROTH (U.S.P. 1,959,437, 22.5.34. Appl., 27.4.32).—To provide a gaseous atm. which absorbs the ultra-violet and infra-red rays and renders the work visible, the welding rod is coated with a mixture of a cellulose ester (40), an ester of  $H_3BO_3$  (40),  $Na_2SiO_3$  (10),  $CaCO_3$  (3), silicomanganese (3), and kaolin (4%). A. R. P.

Electrolyser and electric accumulator. H. NIEDERREITHER (U.S.P. 1,980,873, 13.11.34. Appl., 24.3.33. Ger., 23.3.32).—A Ni–Fe or other type of accumulator is provided with means, including a diaphragm, by which the  $H_2$  and  $O_2$  evolved may be collected separately. B. M. V.

Supplying and cleaning gas by electrical action. C. HAHN, ASST. to INTERNAT. PRECIPITATION CO. (U.S.P. 1,980,521, 13.11.34. Appl., 22.1.32. Ger., 19.1.31).— The gas is propelled through the precipitator solely by the action of electric wind produced by suitably inclined sharp points on the live electrodes. B. M. V.

Bleaching cellulose.—See VI. Magnetic alloys. Plating apparatus. Cu sheet etc. Electrodeposited Fe-Ni alloys.—See X.

## XII.—FATS; OILS; WAXES.

Action of commonly occurring organisms on fats. A. SEDUICH (Bull. State Inst. Agric. Microbiol. U.S.S.R., 1933, 5, 299—313).—Many organisms decompose fats yielding aldehydes and acids. Their relation to rancidity is discussed. CH. ABS. (p)

Detection and control of diacetyl in fats. H. Häffner (Z. Unters. Lebensm., 1935, 70, 117-120).— The Ni reaction is recommended for detection of Ac<sub>2</sub>. Its addition to butter should be prohibited. E. C. S.

Saponification curve of fats in alcoholic solution and its importance for analytical evaluation of fats. R. STROHECKER (Z. Unters. Lebensm., 1935, 69, 521-541).—In the first stages of saponification the reaction curve is that of neither a unimol. nor a bimol. reaction, but after 90 min. at 18°, or 10 min. at 30°, the reaction becomes strictly bimol. This is due either to alcoholysis, or to the more rapid saponification of glycerides of low mol. wt. A practical method is described for the detection of adulteration by determining the fall in conductivity in 20 min. of the fat-EtOH-KOH system at 30°. Fats rich in PrCO<sub>2</sub>H, C<sub>11</sub>H<sub>23</sub>·CO<sub>2</sub>H, C<sub>13</sub>H<sub>27</sub>·CO<sub>2</sub>H, or unsaturated acids are more rapidly

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saponified than those rich in palmitic and stearic acids. High acidity results in a lower rate of saponification. E. C. S.

Synthetic fats. I. Manufacture of synthetic tallow and butter. S. UENO (J. Soc. Chem. Ind., Japan, 1935, 38, 237—241 B).—Distilled fatty acids, prepared from the soap-stock (" oil foots ") obtained from the alkali-refining of fish oils, were hydrogenated and esterified with glycerin to yield tallow-like fats. By esterifying a mixture of hardened fish oil and  $PrCO_2H$  or coconut oil fatty acids with glycerin, in the presence of a catalyst (e.g., Sn) capable of inducing evolution of  $H_2$ , ester interchange occurred and (edible) butter-like fats were produced.

E. L.

Indian oil-seeds. F. J. F. SHAW (J. Soc. Arts, 1935, 83, 945—968).—A lecture.

Solid and liquid components of palm oil. T. A. BUCKLEY (Malay. Agric. J., 1935, 23, 315—320).—The stearin is separated from the cooled oil by filter-pressing. The crude solid is melted slowly, cooled, and pressed in a screw press, the process being repeated 2 or 3 times. During the separation, fatty acids and carotene tend to accumulate in the oleine. The colour remaining in the stearin is insufficient to necessitate bleaching for use in soap-making. A. G. P.

Physical and chemical constants of fig-seed oil A. PAIZI (Praktika, 1934, 9, 164—166; Chem. Zentr., 1935, i, 1314).—Seed contain  $23 \cdot 5\%$  of oil ( $5 \cdot 7\%$  in seed from dried figs),  $d^{15}$  0.929, sap. val.  $4 \cdot 35$ , Reichert-Meissl val. 1.04, Polenske val. 1.52, I val. 147.4, Hehner val. 87.3; the free fatty acids have m.p. 17°.

A. G. P.

**Tea-seed oil.** J. PRITZKER and R. JUNGKUNZ (Z. Unters. Lebensm., 1935, **69**, 542—547).—Tea-seed oil resembles olive oil in its consts. and reactions. It is free from saponin. The seeds are free from caffeine, but the flowers contain 1.38%. Phytosteryl acetate from samples of tea-seed oil had m.p. 156—157° and 175—176°, whilst that from olive oil has m.p. 118—119°, but adulteration of the latter with the former cannot be detected by making use of this difference in m.p. None of the colour reactions attributed to tea-seed oil is trustworthy. E. C. S.

Coula-seed oil. A. STEGER and J. VAN LOON (Rec. trav. chim., 1935, 54, 502—504).—Extraction of the airdried coula (or koumounou) seeds (*Coula edulis*, Baillon, tropical Africa) with light petroleum gave  $7 \cdot 2^{\circ}_{0}$  of oil having  $d_{78}^{-3} 0.8979$ ,  $n_{70}^{20} 1.4683$ ,  $n_{70}^{-0} 1.4511$ ,  $\pm$  val. (Wijs)  $86 \cdot 0$ , CNS-I val.  $82 \cdot 6$ , sap. val.  $189 \cdot 5$ , acid val.  $20 \cdot 3$ , Ac val. 11, Reichert-Meissl val.  $0 \cdot 4$ . The oil consisted of unsaponifiable matter  $0 \cdot 8$ , total fatty acids  $91 \cdot 4$ (saturated acids  $1 \cdot 6$ , oleic acid  $87 \cdot 1$ , linoleic acid  $2 \cdot 7^{\circ}_{0}$ ), glycerol residues  $3 \cdot 8$ , volatile matter etc.  $4 \cdot 0^{\circ}_{0}$ . The unsaponifiable matter, m.p.  $12^{\circ}$  (approx.), after removal of protein matter and vac. distillation, had m.p.  $11 \cdot 6^{\circ}$ ,  $n_{70}^{\circ} 1 \cdot 4415$ , I val. (Wijs)  $91 \cdot 1$ , acid val. 200, mean mol. wt. 281. P. G. C.

Iodine content of American cod-liver oil. A. D. HOLMES and R. E. REMINGTON (Amer. J. Dis. Children, 1935, 49, 94—100).—Vals. ranged between 3590 and 14,940 units per billion of oil. Samples from Nova Scotia, Gaspe, and Newfoundland contained more I than those from Maine or regions where deep-sea fish were available. CH. Abs. (p)

Effect of storage on the colour and on the free fatty acid content of a commercial sample of veterinary cod-liver oil. E. J. SHEEHY (Sci. Proc. Roy. Dublin Soc., 1935, 21, 243—245; cf. B., 1933, 974; 1934, 683).—Samples of an oil (1.54%) of free fatty acids) after storage in the dark for 20 months in (a) an unstoppered jar, (b) full, sealed containers, and (c) an Fe drum,  $\frac{1}{3}$  full, contained 3.20, 1.68—1.76, and 2.43% of free fatty acids, respectively. Colour changes, possibly associated with the material of the container, also occurred with increase in acidity. E. H. S.

Preservation of halibut-liver oil with quinol. W. S. JONES and W. G. CHRISTIANSEN (J. Amer. Pharm. Assoc., 1935, 24, 465—469).—Quinol (0.03%) inhibits O absorbtion and vitamin-A deterioration (measured by colour and biological tests) of refined halibut-liver oil. Maleic acid is ineffective. E. H. S.

Partial hydrogenation of fish oil. II. General course of hydrogenation of sardine oil. M. TAKANO and Y. KUMENO (J. Soc. Chem. Ind., Japan, 1935, 38, 252-254 B).—Detailed analyses of samples of Hokkaido sardine oil, drawn during the course of hydrogenation at  $220^{\circ} \pm 5^{\circ}$  over a Ni catalyst, show that the reaction is roughly selective; the amount of unsaturated acids yielding light petroleum-insol. bromides scarcely changes until all acids yielding Et<sub>2</sub>O-insol. bromides have disappeared, and saturated acids are not formed in notable amount until all the first two classes have been converted into monoethylenic acids. *iso*Oleic and oleic acids, however, appear to be converted into saturated acids at about equal rates. E. L.

Behaviour of fats and oils towards air, light, and plant enzymes. L. M. HOROVITZ-VLASSOVA, E. E. KATSCHANOVA, and A. D. TKATSCHEV (Z. Unters. Lebensm., 1935, 69, 409–421).—The action of air,  $O_2$ ,  $H_2O_2$ , diffuse daylight, direct sunlight and ultraviolet light, and of extracts of soya- and castor-oil beans on the physical properties and oxidation of soya oil is investigated. E. C. S.

Determination of iodine value. L. SHERDEVA and G. SHIRJAEVA (Grozn. Neft., 1934, 4, No. 6-7, 45-47). --Methods are reviewed. That recommended is to shake  $0\cdot1-0\cdot2$  g. of the sample with 15 c.c. of a 2:1 Et<sub>2</sub>O-COMe<sub>2</sub> mixture and 25 c.c. of  $0\cdot2N$ -I in EtOH, add 200 c.c. of H<sub>2</sub>O, shake for 5 min., and after 5 min. titrate the excess of I with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. CH. ABS. (e)

Fatty acids of Japan wax. M. TSUJIMOTO (Bull. Chem. Soc. Japan, 1935, 10, 212—219; cf. A., 1932, 313).—The acids present in the unbleached wax of *Rhus succedanea*, L., berries are palmitic 77%, stearic and arachidic 5%, oleic 12%, linoleic < 1%, dibasic acids 6%. The acids were separated by fractional distillation of their Me esters and subsequent extraction with COMe<sub>2</sub> and pptn. of the Pb salts.  $n_{20}^{\infty}$ , I and sap. vals. are recorded for the ester and acid fractions.

F. R. G.

Oiling of wool.—See V. Oil palms.—See XVI. Determining fat in milk.—See XIX.

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Cleansing preparation. R. D. GRANT (B.P. 432,140, 4.5.34).—Olive oil or other animal or vegetable oil is incorporated in cold-H<sub>2</sub>O cleansers of the known type comprising soft soap, sawdust or other fibrous material, and perfume. S. S. W.

Process for emulsifying oils, fats, waxes, resins, pitches, asphalts, and the like with water. R. O. BRATKE (B.P. 431,642, 6.12.33).—About 0.01% of an aq. alkali (KOH lye) containing about 10 wt.-% of an aromatic acid, OH-acid, or a phenol, e.g., BzOH,  $o-OH \cdot C_6H_4 \cdot CO_2H$ ,  $o-C_6H_4(OH)_2$ , is added to the substance prior to or during admixture with H<sub>2</sub>O. Emulsions of the H<sub>2</sub>O-in-oil type are obtained for manufacture of paints, inks, salad oils, road surfaces, etc. B.P. 392,237 (B., 1933, 596) is disclaimed. A. W. B.

Amidines.—See III. Wetting agents. Colouring fats etc.—See VI. Cleaning agent.—See XXIII.

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

Drying oils. S: O. SORENSON (Paint and Var. Prod. Man., 1935, 13, No. 1, 22-26).—A review of recent developments in the use of linseed, tung, soyabean, fish, safflower, and perilla oils in varnishes and paints. D. R. D.

Acidity in paints and varnishes. R. FORDER (Paint Manuf., 1935, 5, 126-129, 188-191).—A review is given of the sources of the acidity and its effects on the grinding of pigments, livering, peptising, and flow of paints, coloration of bronze powders and insulating varnishes, gelation of tung oil, fossil gum, and rosin varnishes, and the stability of liquid driers.

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Paint testing. C. D. HOLLEY (Paint and Var. Prod. Man., 1935, 13, No. 1, 28-30).—A review of known methods. D. R. D.

**Durability of paint.** A. V. BLOM (Paint and Var. Prod. Man., 1935, **13**, No. 1, 7—10).—In determining the durability of paints by exposure trials, it is better to determine the flexibility etc. of the film at intervals than to observe merely visible breakdown. The application of the Erichsen machine to testing the distensibility of weathered paint films is described. D. R. D.

Influence of pigment on paint film weathering. H. A. DEPEW (Ind. Eng. Chem., 1935, 27, 905-908).-For durable ZnO paints, the pigment particles should be relatively large coarse needles rather than fine regular "round" particles. A reinforcing "brushheap" structure (illustrated by photomicrograph) is developed in the application of paint containing acicular ZnO and the phenomenon of separation at pigment-vehicle interface is also accentuated, leading to innumerable microscopic failures which relieve the normal stresses in an exposed paint film. The " cracking "type of paint failure, usually associated with ZnO, is thus converted into the less undesirable "chalking." The other pigment properties of ZnO, particularly reactivity with acid vehicle and opacity to light of short  $\lambda\lambda$ , are shown to be less significant than hitherto supposed. S. S. W.

**Production of non-metallic paint pigments.** N. F. JUSCHKEVITSCH, V. N. SCHULTZ, M. E. ZBOROVSKI, and A. L. LJUBIMOV (Trans. Inst. Econ. Mineral. U.S.S.R. [10-year vol.], 1933, 323—345).—Prep. of BaS and of TiO<sub>2</sub> from various sources is described and their uses in paint production are examined. CH. ABS. (*p*)

Zinc chromate. I. ANON. (Paint and Var. Prod. Man., 1935, 13, No. 1, 12—17).—A detailed review of the chemistry of Zn chromate pigments (ZnCrO<sub>4</sub>, basic and hydrated chromates, double chromates with other metals, etc.). D. R. D.

Colloid nature of Chinese black ink. II. Τ. TERADA, R. YAMAMOTO, and T. WATANABE (Sci. Papers Inst. Phys. Chem. Res. Tokyo, 1935, 27, 75-92; cf. B., 1934, 370).-The relation between the coeff. of friction  $\mu$  of the stone on which the dry colour stick is rubbed with H<sub>2</sub>O and the rate of dissolution and size of colloidal particles is investigated. The rate of dissolution decreases, and for different stones  $\mu$  varies at different rates, with repeated rubbing. For metal surfaces the particle size is > for stone surfaces. The mechanism of dissolution is discussed ; µ consists of a part independent of the texture of the contact surface and a part governed by roughness. N. M. B.

Natural varnish gums. H. H. NELSON (Drugs, Oils, and Paints, 1935, 50, 242-250, 282-284).—The production, grading, and marketing of shellac, copal, dammar, and kauri gum are described. D. R. D.

Precipitation of solid driers. A. A. KRAËFF (Verfkroniek, 1935, 8, 180-181).-The pptn. of driers from varnishes on keeping may be due to (1) the absence of sufficient aromatic hydrocarbons in the thinners; (2) the use of too much drier; (3) the use of driers having too high a proportion of metals, i.e., an excess over that capable of forming a sol. compound with the org. acid, a fault common with pptd., but not with fused, driers; (4) the use of Pb linoleate, which is only sparingly sol.; (5) oxidation of the driers on keeping to sparingly sol. salts of oxidised resin and fatty acids; (6) the use of driers made from oxidised acids; (7) inaccurate temp. control during the prep. or addition of driers; (8) insufficient stirring during the prep. of pptd. driers. Notes on individual driers are appended.

D. R. D.

Benzylcellulose lacquers. L. LIGHT (Paint and Var. Prod. Man., 1935, 13, No. 1, 18-21).—The relative merits of cellulose nitrate, cellulose acetate, and benzylcellulose (I) lacquers are discussed. Suitable solvents, plasticisers, and resins for use with (I) are listed.

D. R. D.

**Evap-O-Rotor.** Device for comparing the evaporation rates of lacquer solvents. J. H. LOWELL (Ind. Eng. Chem. [Anal.], 1935, 7, 278-279).—The samples are placed in Al cups, which are slowly rotated on a circular turntable. The loss of wt. after a given time interval is determined. E. S. H.

## PATENTS.

Treated pigments or fillers for rubber mixes, plastics, paints, etc. PURE CALCIUM PRODUCTS CO., and A. H. STEVENS (B.P. 432,032, 15.1.34. Addn. to B.P. 379,942; B., 1932, 999).—The inorg. earthy material, e.g.,  $CaCO_3$ , is ground in a paint or plastic, in pebble or tube mills etc. in place of a ball mill, and the mass is discharged while still in the "free-flowing stage." S. S. W.

Coating compositions. H. A. AUDEN and H. P. STAUDINGER (B.P. 432,025, 19.12.33).—Cellulose ester, *e.g.*, acetate, lacquers containing one or more pyruvic or lævulic esters, b.p.  $\lt$  150°, are claimed. S. S. W.

Plasticising [cellulose] esters. A. M. MUCKENFUSS and V. L. HANSLEY, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,975,697, 2.10.34. Appl., 19.3.32).— Esters derived from polycyclic hydrocarbons occurring in coal tar are claimed as softeners and plasticisers for esters, e.g., nitrate, acetāte, of cellulose. Examples include : *Et indene-3-carboxylate*, b.p. 95—100°/1 mm.,  $\beta$ -butoxyethyl fluorene-9-carboxylate, b.p. 295—300°/1 mm., obtained by passing CO<sub>2</sub> into the appropriate Na derivative of the hydrocarbon and esterifying the acid obtained in known manner. A. W. B.

**Removing solvents from plastic colloids.** H. P. BASSETT (U.S.P. 1,976,873, 16.10.34. Appl., 26.6.31).— Solvents and plasticisers are removed from a cellulose ester plastic colloid by exposing it for several hr. or days to an atm. having R.H. approx. 76%, which is subsequently reduced at intervals while the temp. is raised from about 38° to 70°. S. M.

(A) [Phenol-aldehyde] synthetic resin composition. (B) Manufacture of [alkyd] synthetic resins and synthetic resin varnishes. (c) [Alkyd] synthetic resin compositions. S. L. M. SAUNDERS (B.P. 431,951, 432,103, 432,158, [A] 26.4.34, [B, C] 21.12.33).--(A) A fusible phenol-aldehyde resin is dissolved at (150° in a partial ester (containing  $\lt$  2 free OH) of a monobasic org. acid and a polyhydric alcohol, e.g., glyceryl monolinoleate; the mixture is heated with o-C<sub>6</sub>H<sub>4</sub>(CO)<sub>2</sub>O (I) or other polybasic org. acid or anhydride. The partial ester is condensed (B) with (I) in presence of heat-treated tung or other drying oil, (c) with an incomplete condensation product of (I) and a polyhydric alcohol (glycerol). All these resins are S. M. oil-sol.

Manufacture of [resinous] nitrogenous condensation products derived from phenols. Soc. CHEM. IND. IN BASLE (B.P. 432,143, 22.5.34. Switz., 24.5.33) .--- Mono- or poly-nuclear OH-aryl compounds (I) or their ethers having at least one reactive CH<sub>o</sub> are condensed with an arylamine [approx. 1 mol. per mol. of (I)], the condensation being interrupted immediately the product begins to lose solubility in EtOH or COMe2. *E.g.*, crude cresol (72 pts.) is condensed with  $CH_2O$  (125 pts. of 40 vol.-%) in aq. NaOH at  $< 10^{\circ}$ , NH<sub>2</sub>Ph (96 pts.) is added, and the product neutralised (HCl); further HCl (1 pt.) is added and the mixture heated at 95° for 1 hr. The resin is separated (Na<sub>2</sub>CO<sub>3</sub>) and heated in a current of steam at 60°/60 mm. It is sol. in EtOH and is converted by heat into an insol., infusible product. H. A. P.

Mills for paints etc.—See I. Salts of ketoaromatic acids.—See III. Colouring lacquers etc. —See VI. Emulsifying resins etc.—See XII. Flakes from plastic materials.—See XIX.

# XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Zinc oxide, kaolin, and whiting in carbon black mixtures with natural and synthetic rubber. A. ERMOLAEV and O. RAIZ (J. Rubber Ind. U.S.S.R., 1934, 11, 131—140).—Fillers for use with synthetic rubber are discussed. The optimum vol. was > for natural rubber. The use of ZnO, clay, and whiting in place of gas- and lamp-blacks in natural or synthetic rubber mixtures decreased the tensile strength and abrasion-resistance to about the same degree. Mixtures of synthetic rubber and lampblack (alone or with ZnO) did not evolve heat. CH. ABS. (e)

Use of Veimarn (Leningrad district) slate tar as a softener in rubber mixtures. N. TSCHESNOKOV and G. MICHAILENKO (J. Rubber. Ind. U.S.S.R., 1934, 11, 146-151).—Some of the tar fractions were satisfactory softeners. CH. Abs. (e)

Commercial rubber with fine pores and cells, made with carbon saturated with carbon dioxide. N. TSCHESNOKOV (J. Rubber Ind. U.S.S.R., 1934, 11, 221—227).—C black, prepared from the husks of sunflower seeds, was activated at 800—900° and saturated with  $CO_2$  at room temp. (8·2% of  $CO_2$  absorbed in 5 hr.). During vulcanisation it gave up  $CO_2$ , forming pores in the rubber. The sponge rubber ( $d \ 0.486$ —0·533) was H<sub>2</sub>O-, sound-, and heat-proof. CH. ABS. (e)

Modified rubbers. VII. Expanded chlorinated rubber. P. SCHIDROWITZ and C. A. REDFARN. Composition of some chlorinated rubbers. G. F. BLOOMFIELD, E. H. FARMER, and C. H. MILLER (J.S.C.I., 1935, 54, 263—267 т; cf. B., 1935, 643; also B.P. 424,561; B., 1935, 370).—A light, strong, cellular product, of low H<sub>2</sub>O absorption and apparent sp. gr. as low as 0.087, is obtained when chlorinated rubber is heated under pressure and the pressure is then released. A method is suggested for production on a commercial scale. Chlorinated rubber does not absorb I and it apparently undergoes no chemical alteration in the expansion process. Possibly the expansion is caused by liberation of  $CCl_4$  held in loose chemical combination. The addition of plasticisers, e.g., triacetin, and sometimes of a volatile liquid, results in an improved product on expansion. Earlier estimates of the proportion of residual CCl<sub>4</sub> in chlorinated rubber (B., 1932, 392) have D. F. T. been excessive.

Synthetic rubber and the creation of a synthetic rubber industry in the U.S.S.R. S. V. LEBEDEV (Sotz. Rekonstr. Nauk., 1933, 3, No. 1, 127–136).—The Lebedev (butadiene) process is described. CH. ABS. (e) Acetone extraction of raw rubber. I. Effect of time of heating of the alcohol solution on the

of time of heating of the alcohol solution on the determination of the acid value of raw rubber and its acetone extract. II. Influence of heating at 100° and storing of the acetone extract on the acid value. H. ENDOH (J. Soc. Chem. Ind., Japan, 1935, 38, 280–281 B, 281–284 B).—I. For direct determination of the acid val. of raw rubber the sample (approx. 1.75 g.) is heated with 50 c.c. of EtOH for 30 min. and the solution titrated with 0.1N-KOH, using phenolphthalein. For the COMe<sub>2</sub> extract the acid val. is measured by heating the extract with 50 c.c. of EtOH

for 15 min. and then titrating. The latter method gives the higher results.

II. Changes are observed in the acid val. on progressive heating of the dried COMe, extract at 100° or on storing the undried extract for several days, and it is recommended that after removal of the COMe, by distillation the flasks containing the residual extracts be dried for < 15 min. at 100° before being weighed. The acid val. of the extract is then measured as above. D. F. T.

Effect of antioxidants on the natural and accelerated ageing of rubber. R. F. TENER and W. L. HOLT (J. Res. Nat. Bur. Stand., 1935, 14, 667-684).-Tensile strength tests have been made on 5 rubber mixes with 5 antioxidants during periods of (a) dark storage, (b) weather exposure, (c) storage in air at 70°, (d) storage in air at 90°, and (e) storage in  $O_2$  at 60°/300 lb. per sq. in. The effect of antioxidants can be expressed numerically by a time index, which is a measure of the time during which a rubber mix will remain serviceable, or by a tensile index which is a measure of the increase in strength compared with a similar mix without antioxidant. The time index for the materials tested was as high as 5-6 for certain conditions and averaged  $2 \cdot 77$ , while the tensile index averaged 1.46 with a max. of 2. A. R. P.

Cold-vulcanisation method for attachment of rubber soles. G. KISELEV (Kozh. Obuvn. Prom., 1934, 13, 454-456).-A solution of rubber in naphtha is used as cement, S<sub>2</sub>Cl<sub>2</sub> dissolved in CCl<sub>4</sub> being used for vulcanisation. Сн. Авз. (е)

Attaching rubber soles with a self-vulcanising cement. LEV, GANDEL, and SEMIZOROV (Kozh. Obuvn. Prom., 1934, 13, 519, 522).-One portion of naphtha is saturated with H<sub>2</sub>S and a second (50% more by vol.) with SO<sub>2</sub>. The first is added to the solution of raw rubber in naphtha, the SO<sub>2</sub> solution being added shortly before applying the rubber cement. The rubber concn. after adding the H<sub>2</sub>S and SO<sub>2</sub> solutions is 5%. Setting of this self-vulcanising solution requires < 4 hr. CH. Abs. (e)

Cements for joining leather to rubber. B. FAB-RITZIEV, G. BUIKO, and E. PACHOMOVA (Kozh. Obuvn. Prom., 1934, 13, 522-523).-Tests with various types of cement are described. Сн. Авз. (е)

## PATENTS.

[Rubber] latex composition and its preparation. M. O. SCHUR, Assr. to BROWN Co. (U.S.P. 1,977,211, 16.10.34. Appl., 4.9.31) .- Waxy antioxidants, e.g., a mixture of ditolylamines and paraffin wax, are mixed with sufficient ZnO (e.g., 5 pts.) to form a dry freeflowing powder which is then, with the aid of a protective colloid, converted into an aq. dispersion to be eventually blended with latex. D. F. T.

Manufacture of rubber. R. M. UNGAR and P. SCHID-ROWITZ (B.P. 431,869, 17.1.34. Cf. B.P. 368,902; B., 1932, 520).-The earlier process for softening rubber is now applied to rubber as first formed from the latex, e.g., to rubber crumb obtained by spraying latex or to crumbed coagulum. D. F. T.

Manufacture of coloured rubber products. I. G. FARBENIND. A.-G. (B.P. 431, 195, 27.12.33. Ger., 23.12.32). -Tetrazotised benzidine, or one of its halogeno-, alkoxy-, or alkyl derivatives, is coupled with Me or Et 1-phenyl-5pyrazolone-3-carboxylate (I) (2 mols.), which may be similarly substituted, to give yellow to red or violet dyes stable to vulcanisation; e.g.,  $4:4':3:3'-(NH_2 \cdot C_6H_3Cl \cdot)_2$  $\equiv 2$  (I) is red. H. A. P.

Method and means of producing rubber thread. DUNLOP RUBBER CO., LTD., and J. HEALEY (B.P. 432,801, 2.2.34).

CS, reaction products. Preserving rubber.-See III. Fillers for rubber mixes.—See XIII.

# XV.-LEATHER; GLUE.

Chrome liquors. III. Penetration of various anions into the chromium nucleus. E. R. THEIS and E. J. SERFASS (J. Amer. Leather Chem. Assoc., 1935, 30, 341-369 ; cf. B., 1933, 505) .- A series of Cr liquors have been titrated conductometrically immediately after the addition of varying amounts of HCl, H2C2O4, and AcOH, and about a month later. The results show that the amount of HCl or H<sub>2</sub>C<sub>2</sub>O<sub>4</sub> which reacts with the Cr complex ∝ concn. of the acid. With HCl this amount increases with time and this is the case for H<sub>2</sub>C<sub>2</sub>O<sub>4</sub> when the solution is < 0.5N, but the reverse obtains at higher acid concns. AcOH penetrates the complex much more quickly than do the other acids and the amount diminishes slightly with time. Stiasny and Königfeld's method for the determination of the non-olated groups thus gives correct results only when the amount of HCl is at least three times as much as is required to neutralise all the OH groups. D. W.

Determination of hydrolysable (pyrogallol) tannins in presence of condensed (pyrocatechol) tannins. L. POLLAK, L. J. FISCHER, and E. POLLATSCHIK (Collegium, 1935, 333-344) .- The pyrocatechol tans are first pptd. by boiling 100 c.c. of the tannin infusion with CH2O-HCl, 3.5 g. of NH3 are added to the filtrate, and a further quantity of CH2O-HCl, when the pyrogallol tans are pptd. as a NH3-CH2O-tannin compound. The ppt. is dried, weighed, its NH<sub>3</sub> content determined, and the amount of pyrogallol tan calc. Alternatively, the NH<sub>3</sub>-CH<sub>2</sub>O-tannin ppt. may be oxidised to H<sub>2</sub>C<sub>2</sub>O<sub>4</sub>, which can then be titrated. The solid residue obtained by evaporating the NH<sub>3</sub> extract of pyrocatechol-CH<sub>2</sub>O ppt. yields the pyrogallol tans carried down therewith. The CH<sub>2</sub>O ppts. with pyrogallol tans, which contain a large amount of pyrocatechol constituents or gallic acid, e.g., oakwood, sumac, do not respond to this NH<sub>3</sub> D. W. treatment.

Reports of International Commission on quantitative tannin analysis, 1934-5. J. G. PARKER and F. STATHER (J. Soc. Leather Trades Chem., 1935, 19, 365-371).-The filter-candle method of filtration is still preferred by many sections. Higher results for insol. matter are given by the Riess method. No alteration in results was observed after keeping ready-chromed hide powder for 1, 2, and 3 years, respectively. The Baldracco modified shake method of analysis is recommended by certain sections; others have obtained slightly lower non-tannin results therewith. D. W.

Tanning with complex chromium salts. V. CASA-BURI and I. CRISCUOLO-CANTARELLA (Boll. Uff. R. Staz. Sperim. Ind. Pelli, 1935, 13, 198—227).—Tanning baths containing Cr alum, NaOH, and formic, citric, lactic, tartaric, or butyric acid were tried. Tartaric acid gives good results, the basicity of the leather produced being > 50%. Renewal of the liquor is unnecessary if the basicity of the leather is kept almost const., colloidal hydrated Cr<sub>2</sub>O<sub>3</sub> being either added or pptd. in the liquor, from time to time. Tanning is possible in complete absence of mineral acid, the Cr<sub>2</sub>O<sub>3</sub> being dissolved in an org. acid. T. H. P.

Physical chemistry of tanning. I. Electrochemical properties of vegetable tanning agents. S. I. SOKOLOV and G. E. KOLLAKOVA (Kolloid-Z., 1935, 72, 74—81).—Extracts of oak, quebracho, pine bark, and willow have negatively-charged colloid particles, the  $\zeta$  potentials of which have been determined by the electrophoretic method. On addition of acid the  $\zeta$  potential falls linearly with the  $p_{\rm H}$ , reaching zero at  $p_{\rm H}$  1. Conductometric and potentiometric titration of purified extracts of oak and pine bark show that they are acids with two stages of dissociation. The first dissociation const. is 10<sup>-5</sup>, apparently due to the CO<sub>2</sub>H; the second is 10<sup>-8</sup> (oak) and 10<sup>-10</sup> (pine bark), probably due to OH groups. E. S. H.

Effect of neutralisation of sheep pelts after pickling on the chrome-tanning process. V. MICHEEV (Kozh. Obuvn. Prom., 1934, 13, 686-687).---Neutralisation before tanning increases Cr absorption and thus permits lowering of the tannide concn. The quality of the finished pelt is improved. CH. ABS. (p)

Influence of the neutralisation of sheep skins after chrome tanning on the dyeing of its flesh side. V. MICHEEV (Kozh. Obuvn. Prom., 1934, 13, 683—684).—The spotty appearance of the flesh side of sheep pelts dyed with metanil-yellow AT was avoided by neutralising the pelts with a solution of  $Na_2S_2O_3$ and  $Na_2CO_3$  (2 and 5 g. per litre respectively).

Сн. Авз. (е)

Theory of the tanning process. (MISS) D. J. LLOYD (J. Soc. Leather Trades Chem., 1935, **19**, 336– 344).—A review. D. W.

**Recent theories of the chemistry of tanning**. (MISS) D. J. LLOYD (J. Soc. Leather Trades Chem., 1935, **19**, 345—349).—The suppression of the negativelycharged active centres in the collagen mols. by the tanning process must cause the groups of mols. held by the polypeptide backbone linkings to move apart, thus altering the spacing of the salt linkings. This theory has been confirmed by Astbury's X-ray photographs. Tanning causes the disappearance of the small inner rings of the X-ray picture. Loss of definition in other rings may be due to bad preservation of the raw material, bad beamhouse work, or bad tanning. D. W.

Absorption of and dehydration by salt in the different layers of raw hide in curing. F. STATHER and H. HERFELD (Collegium, 1935, 333—344).—NaCl was absorbed by raw hide most quickly when the latter was brined; the max. was attained in 12 hr. The NaCl absorbed increased with the [NaCl] and temp., but not in direct proportion; it was independent of the  $p_{\rm H}$  of

the aq. NaCl and absorption by flesh and grain sides was equal. The different layers of the hide absorbed equal amounts of NaCl from a dil. aq. NaCl, but the outer layers at first absorbed more NaCl than did the inner layers from a conc. aq. NaCl; after prolonged immersion the amounts were nearly equalised. Hides were swollen by 2% and 4% aq. NaCl and dehydrated by aq. NaCl > 8%. The dehydration of the hide increased with the [NaCl] to a max. with 32% NaCl after 48 hrs.' treatment. Max. NaCl absorption and complete penetration of the raw hide after 24 hr. were obtained by sprinkling fine or coarse NaCl on the flesh side. The NaCl absorption was unaffected by additions of NaHSO3 or Na2CO3 to the NaCl. The hide was dehydrated more by solid than by aq. NaCl. No difference in the [NaCl] in the different layers of the hide was observed in the two methods of curing, but the curing effect was not so good with more dil. aq. NaCl. D. W.

Oiling leather with petrolatum. D. BAZAROVA (Kozh. Obuvn. Prom., 1933, 12, 613).—Satisfactory penetration was obtained by drumming with a hot mixture of petrolatum 45, mineral oil 40, and degras 15%. CH. ABS. (e)

Preparing chamois leather of natural colour from rejected kid skins. V. KULIKOV (Kozh. Obuvn. Prom., 1933, 12, 609—610).—Details of the treatment are given. CH. Abs. (e)

Utilising rejected leather for shoes. V. GOLD-SCHTEIN (Kozh. Obuvn. Prom., 1933, 12, 608—609).— Rejected skins, after the usual preliminary treatment, are dyed with 3% nigrosine in presence of 0.5% aq. NH<sub>3</sub>, sand-papered, freed from leather dust, and coated with blood loading containing 3% of casein-black and 4—5% of alizarin oil (calc. on the amount of casein pigment in the paste). The product is further dried, covered with a glaze, again loaded, dried, and pressed at 80—90°/20— 30 atm. CH. ABS. (e)

**Preparation of the leather substitute " prima."** B. PRIGOSHIN (Kozh. Obuvn. Prom., 1933, 12, 510—514).— A rubber dispersion is prepared by use of fat and resin soaps and clay is added. After incorporation with chrome-tanned leather fibre the rubber is coagulated by alum at  $p_{\rm H}$  7—8, a protective colloid (casein, dextrin) being used. The leather fibre is further tanned and neutralised with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, the pptd. S being utilised in subsequent vulcanisation. Fat-liquoring is carried out with sulphonated oils. Other fibres (hemp, flax) may also be used in the prep. Vulcanisation of the material does not affect its properties. CH. ABS. (p)

Leather substitute for [boot] uppers. D. GRAFOV (Kozh. Obuvn. Prom., 1933, 12, 572-573).—Cotton fabric is coated with a mixture of leather dust, ZnO, lampblack, S, and "thiuram" incorporated in a 10% solution of rubber in petroleum with a filler.

Сн. Авз. (р)

Judging the quality of leather. H. HERFELD (Angew. Chem., 1935, 48, 521-527).—The following limits are given:  $H_2O$ , 14; fat: sole leather  $\gg$  3, belting, hand-stuffed 8, drum-stuffed 15, impregnated 22, waterproof shoe uppers 10-30%. Ash and  $H_2O$ -sol. matter: pit-tanned sole leather,  $\gg$  1, 9.0; modern

tannages, > 2.5, 15.0; belting and dyed leathers, ---, 6.0%. Deliberate filling may be suspected if the leather contains > 2.5% of mineral matter, 4.0% of  $MgSO_4,7H_2O, 18.0\%$  of total  $H_2O$ -sol. matter, or 2.0% of sugary matter. Vegetable-tanned leather containing >0.05-0.10% Fe<sub>2</sub>O<sub>3</sub> is liable to become brittle. The difference in the Fe<sub>2</sub>O<sub>3</sub> contents of the dyed and undyed portions of a vegetable-tanned leather blacked with Fe salts should be > 0.075%. The neutral salt content of Cr-tanned and Al-tanned leathers respectively should be > 1.5 and 10.0% and the S in two-bath Cr-tanned leather is 0.3-1.5%. The hide substance in noncurried vegetable tanned leathers is 30-45 and the fixed tan 23-40%. The degree of tannage in pit-tanned sole leathers is 50-70 and in drum-tanned <90%. A val. >100 is abnormal and indicative of some type of filling. The  $p_{\rm H}$  val. of the aq. extract of a vegetable-tanned leather (2 g. in 100 c.c. of H<sub>o</sub>O) is normally  $3 \cdot 8 - 5 \cdot 5$ . The apparent d of vegetable-tanned leather is 0.8-1.0, of sole leather < 1.2, and chrome leather 0.6-0.85. The true d is 1.3-1.5, independent of the tannage or dressing. The tensile strength of noncurried vegetable-tanned leather is 2.5 kg./mm.<sup>2</sup> and stretch 30%. For Cr leather these vals. are increased 11-2 times; they are also increased by currying. Semichrome leather has less strength and stretch than have vegetable-tanned leathers. Other physical D. W. properties are also reviewed.

Recent work on the physical testing of leather. C. H. SPIERS (J. Soc. Leather Trades Chem., 1935, 19, 293-306).—A review. D. W.

Adhesion of curried [belting] leather [to pullies]. L. HOUBEN (J. Soc. Leather Trades Chem., 1935, 19, 314—335).—Grease is necessary in belting leather if the coeff. of friction is to be > the rate of slipping. Leather should be curried with suitable mixtures. Fish oils, degras, and tallow are best because they combine with the leather fibres. Wool grease, rubber, and petroleum jelly, mineral oils of high  $\eta$ , and similar greases which retain their oiliness indefinitely are also suitable ingredients for belting leather. Greases which are solid at room temp. but liquid at high temp. do not give good adhesion. The adhesion of the belt to the pulley can be increased by the application of a special grease to the side running in contact with it. D. W.

Increasing the water-resistance of casein pigments [on leather] by means of chrome alum. V. TORSUEV (Kozh. Obuvn. Prom., 1933, 12, 491).— Leather dyed with casein pigments is treated with a solution of chrome alum (I) (25%) and petroleum sulphonic acids (0.3%). It is suspended from trestles for drying (1 hr.), excess of (I) being then washed off. The product has a high and stable gloss. CH. ABS, (e)

Action of nitrous acid on collagen. Properties of deaminised collagen. E. MEUNIER and E. SCHWEIKERT (J. Soc. Leather Trades Chem., 1935, 19, 350-364).— The capacity of collagen (I) to swell is diminished by the action of  $HNO_2$  and therefore it must be a tanning action., The latter is increased by subsequent treatment of (I) with NaHSO<sub>3</sub>. Deaminised (I) absorbs less  $H_2O$ , is less readily gelatinised, swells less, and combines with less acid dyestuff but more basic dyestuff than doe<sup>s</sup> untreated (I). D. W.

Mordanting and dyeing processes. XXVII. Action of metal compounds on gelatin. E. ELÖD and T. SCHACHOWSKOV (Kolloid-Z., 1935, 72, 221–229; cf. B., 1935, 818).—Compounds of Ni<sup>H</sup>, Zn<sup>H</sup>, Mn<sup>H</sup>, or Co<sup>H</sup> have no tanning effect. The effect of other compounds examined decreases in the order:  $Cr^{H} = Co^{H} > Al^{H} = Fe^{H} > Sn^{H} > Th^{H} > U^{H}$ . The tanning effect depends on the  $p_{\rm H}$  of the system; the max. does not occur at the isoelectric point of gelatin, but occurs at the  $p_{\rm H}$  at which the metal hydroxide is pptd. The mechanism is discussed. E. S. H.

Vegetable glues for painting and papering work. F. Ohl (Kunstdünger u. Leim, 1934, 31, 267-271; Chem. Zentr., 1935, i, 1128).—Descriptive, R. N. C.

Dyeing tannins.—See VI. Joining rubber to leather.—See XIV.

#### PATENTS.

Unhairing bath and process for treating hides. N. D. HARVEY, JUN., Assr. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,976,881, 16.10.34. Appl., 15.3.32).—S, physically or chemically combined with an aliphatic amine  $[e.g., (CH_2 \cdot NH_2)_2]$ , mixed with an alkaline inorg. compound (CaO) is claimed. D. W.

Soaking of [raw] hides. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 430,767, 21.3.34. Addn. to B.P. 341,851; B., 1931, 359).—Hides are soaked in a solution containing a salt or other product of H<sub>2</sub>SO<sub>3</sub> and a hydroxyalkylamine. D. W.

Tanning of hides and skins. K. E. BELL and M. C. McDONALD, Assrs. to A. C. LAWRENCE LEATHER Co. (U.S.P. 1,977,226, 16.10.34. Appl., 29.3.33).— Hides or skins are treated with basic Cr liquors, struck out on the flesh side, and while in the extended condition treated or swabbed with dil. aq. NaHCO<sub>3</sub> to complete the tannage. D. W.

Manufacture of glue bases and glues [from starch]. PERKINS GLUE Co. (B.P. 427,880, 27.7.33. U.S., 4.8.32 and 8.7.33).—Starch glue bases, stable on storage, are made by mixing raw cassava or other starch with 3-20% of urea and 0.3-2.5% of BaO<sub>2</sub> or other stable peroxide, with or without small quantities of whiting and soda ash. Liquid glues for plywood etc. are prepared by heating these bases with 1.3-2.25 times their wt. of H<sub>2</sub>O, usually with addition of 2-3% of NaOH. J. H. L.

Vegetable protein glue. R. G. PETERSON, Assr. to G. H. OSGOOD (U.S.P. 1,977,404, 16.10.34. Appl., 29.7.31).—An aq. mixture of a protein-bearing meal (flour) and viscose containing an excess of alkali is claimed. D. W.

Rapid drying of gelatinous material. D. ABER-NETHY (U.S.P. 1,980,898, 13.11.34. Appl., 29.9.31).— Glue or the like in the form of a stiff jelly is violently agitated, preferably by injection of air, and the broken up or aërated mass is extruded under high pressure into a zone of much lower pressure to form catkin-like masses which may be rapidly dried. B. M. V.

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Adhesive. I. F. LAUCKS, ASST. to I. F. LAUCKS, INC. (U.S.P. 1,977,445, 16.10.34. Appl., 21.1.33).—A dry salt of cellulosexanthic acid is treated with aq. NaOH and Na<sub>2</sub>CS<sub>3</sub>, Na<sub>2</sub>CO<sub>3</sub>, or Na<sub>2</sub>S. D. W.

Treatment of hides, skins, or leather. TANNING PROCESS CO., Assees. of J. H. CONNOR and M. M. MERRITT (B.P. 432,634, 20.1.34. U.S., 16.3.33).—See U.S.P. 1,954,798 ; B., 1935, 115.

## XVI.—AGRICULTURE.

Soil survey of Sierra Leone. F. J. MARTIN and H. C. DOYNE (Sierra Leone Dept. Agric., 1932, 35 pp.).— The dominant soil type is lateritic  $(p_{\rm H} 4-6)$ . In general, the N content  $\infty$  the proportion of org. matter, and K vals. are low. Analytical data are given for numerous samples. CH. Abs. (p)

Deep cultivation [of soils] in the light of seven years' investigation. J. APSITS (Z. Pflanz. Düng., 1935, 39, 326—349).—Improvements in  $H_2O$  relationships of soils, in aëration, and in crop yields were not produced by ploughing > 25 cm. deep and the beneficial effect of exceeding 10 cm., where apparent at all, was very small. The practical bearing of these results is discussed. A. G. P.

Direct determination of  $p_{\rm H}$  of soil in its natural state by the quinhydrone method. I. Determination of  $p_{\rm H}$  in paddy-field soil. A. ITANO and Y. TSUJI (Ber. Ohara Inst. landw. Forsch., 1935, 6, 587— 606).—The soil around the rice plant stubs is less acid than elsewhere. Vals. obtained by the use of the quinhydrone electrode as described were < those obtained by previous methods, viz., 0.21—0.56 for fresh soil and 0.03—0.27 for air-dried soil. P. G. M.

Saturation condition of soils. I. F. RADU (Landw. Versuchs-Stat., 1935, 123, 159—179).—For the characterisation of the activity of the colloidal complex of soils the vals. S and T - S form a better basis than does V. The applicability of Kappen's method for determining S in carbonate-rich soils, and of Daikuhara's method for exchange and hydrolytic acidities, are examined. The last-named val. is the most serviceable for calculating the CaO requirement of soils. Relationships between soil type and the properties of the exchange complex are discussed. A. G. P.

[Report on] biochemistry [of soils and sugar cane]. N. CRAIG (Mauritius Dept. Agric., 4th Ann. Rept. Sugar Cane Res. Sta. [1933], 1934, 23-35).— The exchangeable K in Mauritius soils decreases with rising annual rainfall. The  $SiO_2: Al_2O_3$  ratio of the clay fraction of various soil types is examined. Vals. for whole soils are not greatly different from those of the clay fractions, except in dark brown soils in which the %  $SiO_2$  was relatively high and that of  $Al_2O_3$  low. The P-fixing power of these soils is high. Samples in which none of the native P is sol. in 1% aq. AcOH fix 98-99% of P added in fertilisers.

No distinct correlation was apparent between the % of available P, exchangeable K' or Ca'' in soils, and the proportion of these elements in the juice of the sugar cane.

Increased yields of cane are obtained by use of K

fertilisers only if the soil contains < 0.02-0.03% of exchangeable K<sub>2</sub>O. CH. ABS. (p)

Agronomic importance of calcium. W. P. KELLEY (Soil Sci., 1935, 40, 103–109).—The rôle of Ca in relation to the formation and structure of soils, to their acidity, and to the mineral nutrition of plants is discussed. A. G. P.

**Determination of aluminium in soil analysis.** R. RINNE (Z. Pflanz. Düng., 1935, **39**, 278—286).—In the determination of small amounts of Al in soil extracts by pptn. with aq. NH<sub>3</sub> errors are caused through incomplete pptn. from dil. solutions and by losses during the washing of the ppt. The use of very rapid filter-papers may necessitate very prolonged washing. To the Al solution, which, after removal of SiO<sub>2</sub>, should be  $\geq 100$  c.c., are added 10 c.c. of saturated aq. NH<sub>4</sub>Cl (3 g.) prior to pptn. of Al(OH)<sub>3</sub>. After washing 2—3 times the ppt. is redissolved in HCl and repptd. in the usual way except that 10 c.c. of 60% aq. NH<sub>4</sub>NO<sub>3</sub> are now substituted for the NH<sub>4</sub>Cl. A. G. P.

Phosphate availability in calcareous soils : a function of carbon dioxide [content] and  $p_{\rm H}$ . W. T. McGEORGE, T. F. BUEHRER, and J. F. BREAZEALE (J. Amer. Soc. Agron., 1935, 27, 330–335).—Solid CaCO<sub>3</sub> in soils reduces the solubility of PO<sub>4</sub><sup>'''</sup> in carbonato-apatite because of the common-ion effects of Ca<sup>''</sup> and CO<sub>3</sub><sup>''</sup>. The effect of OH' is to lower the absorption of P by plants and also to modify the normal ionisation of orthophosphates in such a manner that  $H_2PO_4'$ , which is desirable if not essential for plants, occurs only in small proportions. Only  $H_2O$ -sol. P fertilisers are suitable for these soils. A. G. P.

Solubility of soil phosphorus as affected by moistening and drying basic soils. T. J. DUNNE-WALD (J. Amer. Soc. Agron., 1935, 27, 325–329).— Certain arid and irrigated soils showed a marked decline in  $PO_4^{\prime\prime\prime}$  solubility when air-dried and stored in the dark. Original vals. were regained after moistening and re-drying overnight. Horizons containing org. matter were notably affected. Subsoils having high CaCO<sub>3</sub> contents reacted differently. A. G. P.

Comparison of Truog's extraction method and dialysis for [determining] phosphate in soils. L. SMOLFK (Vestn. czechoslov. Akad. Zemed., 1934, 10, 602—606; Chem. Zentr., 1935, i, 1293).—Electrodialysis of soil for 24 hr. removes the same amount of  $PO_4^{\prime\prime\prime}$  as is extracted by  $0.002N-H_2SO_4$  in 30 min., except in acid soils for which the latter, and in alkaline soils for which dialysis, gives the higher vals. A. G. P.

Conversion of soil potash from non-replaceable into the replaceable form. F. A. E. ABEL and O. C. MAGISTAD (J. Amer. Soc. Agron., 1935, 27, 437-445).— The transition of non-replaceable to replaceable K in individual soils occurred to a somewhat similar extent whether these were fallowed or cropped to legumes or non-legumes, and was intensified by liming. The transformation took place as readily in soils having initially a high, as in those having a low, replaceable K content. A. G. P.

Determination of the potassium requirement of soils, with special reference to Dirks' method.

b

L. GISIGER (Landw. Versuchs-Stat., 1935, 123, 209– 225).—The K content of aq.  $Ca(HCO_3)_2$  extracts of soil is always > that of  $H_2O$  extracts. The influence of soil reaction on the available K recorded by Dirks', Neubauer's, and Mitscherlich's methods is examined. The use of graded limiting vals. for Dirks' method is suggested. A. G. P.

Modified procedure in nitrogen-transformation studies in forest soils. H. A. LUNT (J. Amer. Soc. Agron., 1935, 27, 346-355).-Sampling and incubation methods are described. In mull types nitrification is considerable. Little NH<sub>3</sub> is formed. Accumulation of NH<sub>3</sub> is most marked in thick duff. CaO intensified nitrification in white-pine but not in red-pine plantation soils. In the case of the latter, growth is directly correlated with the nitrifying capacity of the soil. The initial acidity of soils is related inversely to NH2 accumulation and directly to NO3' accumulation. During incubation soils in which ammonification dominated showed a decrease, and those in which nitrification was greatest an increase, in' acidity. No relationship was apparent between N transformations and the amount of other soil constituents present. A. G. P.

Reactions of liming materials in fertiliser mixtures. K. C. BEESON (Amer. Fertiliser, 1934, 81, No. 10, 5–7, 24, 26; cf. B., 1934, 977).—Admixture of limestone with superphosphate or  $\rm NH_4H_2PO_4$  (I) must be limited to equimol. proportions to avoid formation of  $\rm Ca_3(PO_4)_2$  in the former or loss of  $\rm NH_3$  from the latter. Mixtures of superphosphate and (I) in the proportion of 1 mol. of  $\rm Ca(H_2PO_4)_2$  to  $\gg 1$  mol. of (I) may be treated with limestone in any proportion without loss of  $\rm NH_3$ . Dolomite reacts with superphosphate, evolving CO<sub>2</sub> but causing no decline in citric-sol.  $\rm PO_4'''$ . Interaction of dolomite and (I) yields CO<sub>2</sub> and MgNH<sub>4</sub>PO<sub>4</sub>.

Сн. Авз. (р)

Solubilisation of phosphates in organic fertilisers. J. ALVARINO and A. BONAZZI (Proc. Cuban Sugar Tech. Assoc., 1933, 7, 51—55).—On red ferruginous soils P fertilisers are rendered insol. A fertiliser is prepared by fermenting a mixture of ground bones and molasses. The product contains considerable amounts of  $H_2O$ -sol.  $PO_4^{\prime\prime\prime}$ . CH. ABS. (p)

Efficiency of phosphate fertilisers as affected by distribution in the soil. S. D. CONNER (Amer. Fertiliser, 1934, 81, No. 10, 9, 20, 22).—Sol. phosphates are more effective when drilled in the row or spread near the ridge. Insol. phosphates are preferably mixed with soil. Sol. phosphates may also be mixed with soil if the amount supplied exceeds the fixing power of the soil. CaO usually increases their efficiency.

Сн. Авз. (р)

Crop-improving action of mixtures of superphosphate and calcium cyanamide. M. GERLACH (Z. Pflanz. Düng., 1935, 39, 349—356).—Recorded trials show that the efficiency of a 1:1 mixture of superphosphate and CaCN<sub>2</sub> was comparable with that of ammoniated superphosphate. A. G. P.

Iron and aluminium phosphates as sources of phosphorus for plants. D. L. ASKINAZI and D. M. KHEIFETZ (Phosphorsäure, 1934, 4, 705-741).—Phosphates of Al and Fe prepared under conditions simulating those of fertiliser manufacture are examined. Those pptd. from  $H_3PO_4$  solution by aq.  $NH_3$  were equally sol. in 2% citric acid solution and in  $H_2O$ . The solubility in Petermann's reagent was > 90%. With any of the customary fertiliser solvents, solubility of the phosphates decreased as the  $p_H$  of pptn. increased. Phosphates pptd. by Ca(OH)<sub>2</sub> were less sol. in  $H_2O$ than in 2% citric acid and the solubility increased with increasing  $p_H$  of pptn. The  $H_2O$ -solubility of the latter group of phosphates was much < that of the former. In pot cultures crop yields obtained with Fe and Al phosphates averaged 75% of those with sol.  $PO_4'''$ .

Сн. Авз. (р)

Influence of some fertilisers on the reaction of soils. I. F. RADU (Landw. Versuchs-Stat., 1935, 123, 181–203).—Reaction changes in soil following application of fertilisers are much more satisfactorily recorded by  $p_{\rm H}({\rm KCl})$  than by  $p_{\rm H}({\rm H_2O})$ . Variations in the condition of saturation of soils associated with manuring, cropping, etc. are examined on acid, red-brown, forest soils. A. G. P.

Determination of potassium in mixed fertilisers. K. SCHARRER and H. SCHORSTEIN (Landw. Versuchs-Stat., 1935, 123, 227—234).—The method is based on the preliminary destruction of  $NH_4$  salts by boiling with NaOH, pptn. of K as cobaltinitrite, dissolution of the ppt. in HCl, and the final determination of K as KClO<sub>4</sub>. A. G. P.

Dependence of nitrification [in soil] on the nature and rate of decomposition of organic materials. F. SCHEFFER and Y. M. KARAPURKAR (Kühn-Archiv, 1934, 37, 143—172; Chem. Zentr., 1935, i, 1110).— The rate of nitrification in soils is retarded if the C: N ratio of the materials is > 20: 1, and is influenced by the composition of the org. matter, the rate at which it is decomposed, and by the nature and solubility of the N source. Hemicellulose and cellulose retard and lignin facilitates nitrification. Crop yields are reduced by straw-manuring unless the N supply is such as to maintain a C: N ratio of  $\gg 20$ . Leguminous crops are not reduced by straw. A. G. P.

[Nitrate in soils.] Report of Cotton Experiment Station, Barberton, S. Africa. O. V. S. HEATH (Empire Cotton Growing Corp. Rept. Exp. Sta. [1932– 33], 1934, 50–83).—Data are given showing differences in NO<sub>3</sub>' content of surface (9-in.) soils under various crops and in fallow soils. CH. ABS. (p)

Utilisation of ammonia liquor as a source of nitrogen for vegetables. A. P. KASCHAEV and V. V. SINELSCH (Lenin Acad. Agric. Sci. Ged. Inst. Fert., 1934, No. 3, 166–174).—The liquor was as effective as  $(NH_4)_2SO_4$  for tomatoes and onions. CH. ABS. (p)

Modern treatment of stall manure. WIESSMANN (Landw. Versuchs-Stat., 1935, 123, 24-41).—The val., use, and methods of handling stall manure are discussed. A. G. P.

Comparison of organic manures in relation to the maintenance and improvement of soil productivity : stall manure, artificial manure, strawand green-manuring. W. MACHWIRTH (Kühn-Archiv, 1934, 37, 269–316; Chem. Zentr., 1935, i, 1108).— The influence of the various manures on the biological

activity of soils fluctuated widely. In general, artificial manure and green-manuring produced better crop yields than did stall manure. Straw-manuring gave positive or negative results according to whether it was supplemented with N or not. A. G. P.

Influence of the soil on the growth of the plant. H. LUNDEGÅRDH (Soil Sci., 1935, 40, 89—101).—Relationships between the concn. of nutrient ions in the soil and in plant juices are discussed with reference to growth rates of plants. A. G. P.

Influence of various cations, of chlorides, and of sulphates in nutrient solutions on the growth of plants. A. G. SCHESTAKOV and V. G. SCHVUINDENKOV (Lenin Acad. Agric. Sci. Ged. Inst. Fert., 1934, No. 3, 40-67).—In sand cultures of flax, sunflower, and potato, high [Cl'] in the nutrient tended to reduce total yields > did high [SO<sub>4</sub>"]. Grain yields, however, were higher with high [Cl'], irrespective of the cations present. Of the cations Na was more injurious than Mg. Plants accumulated Cl' faster than SO<sub>4</sub>". Cl' tended to lower the PO<sub>4</sub>"" intake of plants. The presence in the nutrient of MgSO<sub>4</sub> decreased, and that of MgCl<sub>2</sub> increased, the intake of K and Ca. CH. ABS. (p)

Influence of nutrient supplies on plant growth in highly acid soils. K. SCHARRER and W. SCHROPP (Z. Pflanz. Düng., 1935, 39, 265–278).—Crop increases due to application of fertilisers to acid soils were in the order barley and rape > flax and wheat > buckwheat. Acid-sensitive plants produce excellent growth in soils of high [H] provided the nutrient supply is at optimum level. A. G. P.

Importance of micro-elements and the Ca/Mg ratio for plant growth in relation to the liming of acid soils. G. A. TALIBI (Z. Pflanz. Düng., 1935, 39, 257–264).—Heavy application of  $CaCO_3$  and/or MgCO<sub>3</sub> to soil caused an increase in the base intake of plants (flax) and prevented the assimilation of B and the satisfactory growth of the crop. The injurious action was not affected by the relative proportions of Ca and Mg applied, but was corr. by additions of B. The rôle of B in the mineral nutrition of plants is discussed.

## A. G. P.

Plants and polar sorption of soils : physiological removal of sorbed calcium ions. I. S. D. DI DELUPIS (Z. Pflanz. Düng., 1935, 39, 295–300).—The growth of plants in soil causes an exchange of H for Ca<sup> $\circ$ </sup> in the colloid complex and subsequent pptn. of the released Ca as CaCO<sub>3</sub>. The ability of crop plants to cause exhaustion of adsorbed Ca varies with their dry matter production, but on a comparative basis is in the order lucerne > sugar beet > wheat. A considerable proportion of the Ca thus pptd. is probably redissolved and subsequently re-enters the exchange complex. The energy exchanges in this cycle of reactions are discussed. A. G. P.

Intake of calcium-bound phosphoric acid by plants. W. -U. BEHRENS (Z. Pflanz. Düng., 1935, 39, 301-309).—Comparison is made of the efficiency of basic slag and other sources of P in sand cultures of oats. When the materials were not uniformly mixed with the sand, poor yields occurred if the fertiliser remained in close contact with CaCO<sub>3</sub> and also if Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> were used as the source of P. The latter effect is related to the very low solubility of the P of  $Ca_3(PO_4)_2$  in buffered solutions containing Ca<sup>\*\*</sup>. Customary pot-culture trials of fertilisers, in which the material is intimately mixed with the substrate, do not satisfactorily reflect the efficiency of the fertilisers in the field. A. G. P.

Influence of fertilisers containing large quantities of chlorine on the quality and quantity of crop yields. A. G. SCHESTAKOV and V. G. SCHVUINDENKOV (Lenin Acad. Agric. Sci. Ged. Inst. Fert., 1934, No. 3, 3-40).-A new fertiliser, "potazot" (obtained by treating carnallite with NH3 and CO2, forming KCl and NH4Cl), is examined. Its high [Cl'] causes lowering of the starch content of potatoes and of the chlorophyll content of the leaves. The burning quality of tobacco is also impaired. Flax and cabbage showed slightly lowered chlorophyll. Sugar beet, rye, and oats were not adversely affected. On podsols CaO should be used in conjunction with the fertiliser. Rock phosphate is as effective as pptd. phosphate when applied with " potazot." CH. ABS. (p)

Relative growth and dry-weight production of plant tissue under Mazda, neon, sodium, and mercury-vapour lamps. J. M. ARTHUR and W. D. STEWART (Contr. Boyce Thompson Inst., 1935, 7, 119— 130).—On the basis of dry matter production of buckwheat plants the order of efficiency of the lamps was Ne > Mazda > Na > Hg. Vals, calc, for equal amounts of energy in the visible region placed the lamps in the order Na > Ne > Mazda > Hg. No relationship was apparent between the emission bands of the lamps, the absorption bands of chlorophyll pigments, and the efficiency of dry matter production. Gaseous-discharge lamps produced greener leaves and a lower stem : leaf ratio than did Mazda lamps. A. G. P.

Growth, yield, and composition of certain tropical fodders. D. D. PATERSON (J. Agric. Sci., 1935, 25, 369—396).—In trials with Elephant grass, Uba cane, and Guatemala grass, increasing the period between cutting from 45 to 180 days resulted in progressive increases in dry matter yields and decreases in the % of protein. Max. protein yields were obtained with 90-day intervals (cf. A., 1933, 1341). A. G. P.

Mineral content of fodder crops. R. R. FOLLETT-SMITH (Divisional Repts. Dept. Agric. Brit. Guiana [1933], 1934, 97–98).—Analytical data show fodder crops from front-land clays and pasture grasses from intermediate savannahs to be deficient in Ca and P. CH. ABS. (p)

Yield and chemical composition of certain species of grass. J. G. ARCHIBALD and E. BENNETT (J. Agric. Res., 1935, 50, 711—715).—Analyses of feeding val. and mineral constituents of 6 species of grasses and of wild Dutch clover are recorded. A. G. P.

Effect of manuring with stall manure and with potassic fertilisers on the yield and starch content of potato tubers in relation to the chlorine content of the soil. A. NĚMEC (Vestn. Czechoslov. Akad. Zemed., 1934, 10, 571-576; Chem. Zentr., 1935, i, 1291).—The starch content of tubers is reduced by Cl added in cattle manure or artificial fertilisers. The

natural Cl content of the soil is an important factor regulating the intensity of these effects. A. G. P.

Shortening the rest period of potato tubers. F. E. DENNY and L. P. MILLER (Contr. Boyce Thompson Inst., 1935, 7, 157—181; cf. B., 1928, 583).—Germination of cut dormant tubers was hastened by soaking for 1 hr. in 1% aq. NaCNS. Non-dormant tubers were not injured by this treatment. Similar results were obtained by exposure of tubers to vapour of CH<sub>2</sub>Cl·CH<sub>2</sub>·OH (I) for 7 days. Considerable variations in temp. during treatment, storage, and planting did not prevent positive results. V.-p. curves for (I) and its const.-boiling mixture are given. The tubers absorb considerable amounts of (I) during treatment. Much of this is decomposed within the tissue. A method is described for determining (I) in treated tubers, involving recovery by distillation and decomp. with Ba(OH)<sub>2</sub>.

À. G. P

Effect of soil conditions and treatment on yields of tubers and sugar from the American artichoke (Helianthus tuberosus). H. B. SPRAGUE, N. F. FARRIS, and W. G. COLBY (J. Amer. Soc. Agron., 1935, 27, 392—399).—Soil texture affects yields as a result of H<sub>2</sub>O relationships during the crit. period of tuber formation. The hexose (chiefly fructose) content of tubers varied between  $14 \cdot 6\%$  in moist seasons to  $19 \cdot 3\%$  in dry seasons. On an acid soil ( $p_4 5 \cdot 9$ ) CaO markedly increased sugar yields. Fertiliser requirements are examined.

Padi-soil investigations. R. R. FOLLETT-SMITH (Div. Repts. Dept. Agric. Brit. Guiana [1932], 1934, 107).—No relationship was apparent between the yield of padi and any of the customary nutrient characteristics of the soils. CH. ABS. (p)

Analysis of variance of maize yields obtained in crop-rotation experiments. R. J. GARBER and T. C. MCLIVAINE (J. Amer. Soc. Agron., 1935, 27, 480-483).—The application of statistical methods to the interpretation of cropping trials is discussed. A systematic arrangement of plots is described. A. G. P.

Sugar-cane soils. R. R. FOLLETT-SMITH (Div. Repts. Dept. Agric. Brit. Guiana [1932], 1934, 96—103). —Analyses and profile characteristics of these soils are recorded and their formation is discussed. The effects of drainage are examined. CH. ABS. (p)

Salinity of cane soils. R. R. FOLLETT-SMITH (Div. Repts. Dept. Agric. Brit. Guiana [1932], 1934, 104).— Sol. salt contents of 0.16-0.31% (surface) and 0.16-0.41% (subsoil) are recorded. Flooding with H<sub>2</sub>O lowered the vals. and improved the yield of cane. CH. ABS. (p)

Soil salinity and magnesium toxicity. R. R. FOLLETT-SMITH (Div. Repts. Dept. Agric. Brit. Guiana [1933], 1934, 90—92).—The disease is not associated with accumulation of Mg in sugar-cane plants or with an unfavourable MgO: CaO ratio. It results from high concns. of sol. salts (probably Na and Mg chlorides and sulphates) in soil and subsoil. The ash of diseased plants has high Cl' and K' contents. CH. ABS. (p)

Changes occurring in cane soils of British Guiana, R. R. FOLLETT-SMITH (Div. Repts. Dept. Agric. Brit. Guiana [1932], 1934, 103).—Improved sugar yields over a period of years are associated with the effects of drainage and cultivation in removing Na from soils and consequent lowering of the  $p_{\rm H}$  from > 7.0 to approx. 5.0. CH. ABS. (p)

Flooding of sugar-cane soils. R. R. FOLLETT-SMITH (Div. Repts. Dept. Agric. Brit. Guiana [1932], 1934, 105).—Soils containing 0.7—5.9 mg.-equiv.-% of exchangeable Na were completely flocculated in 24 hr. by shaking with aq. Ca(OH)<sub>2</sub> (0.027% CaO). Flocculation was maintained in nearly all cases by replacement of 50% of the supernatant liquid with a dil. solution (0.0027% CaO). CH. ABS. (p)

Comparative values of commercial fertilisers. M. TREMOLS (Proc. Cuban Sugar Tech. Assoc., 1933, 7, 41-45).—For acid sugar-cane soils  $NO_3'$  is a better source of N than is  $NH_4$ . Fertilisers should also include org. N (dried blood, tankage, urea, or  $CaCN_2$ ) and  $NH_4$  phosphates. For soils of high Fe content  $NH_4$ phosphates are more efficient than is superphosphate.

Сн. Авз. (р)

Methods adopted in St. Kitts for fertilising the sugar-cane crop. [Composition of soils.] ANOX. (Rept. Agric. Dept. St. Kitts-Nevis [1933], 1934, 23-27).—Analyses of these volcanic soils are recorded and results of fertiliser treatment are described.

Сн. Авз. (р)

Influence of excessive quantities of fertilisers on the composition of sugar beets. B. BUSZCZYNSKI (Bull. Assoc. Chim. Sucr., 1935, 52, 345—348).—On 7 plots, effects of normal and very excessive doses of K, N, and P fertilisers were compared. Excessive doses increased the ash and N contents and lowered the sugar content of the roots. J. H. L.

Effect of ammonia and nitrates on the yield of sugar beet in relation to anion components and the reaction of the medium. A. V. VLADIMIROV (Lenin Acad. Agric. Sci. Ged. Inst. Fert., 1934, No. 3, 104-129).-In sand cultures with NH<sub>4</sub> media yields of sugar beet were higher in the presence of Cl' than in that of SO<sub>4</sub>". With NO<sub>3</sub>' media, SO<sub>4</sub>" favoured higher yields. With increasing [NH<sub>4</sub>], the N content of beet increased in SO<sub>4</sub>" and Cl' media of  $p_{\rm H}$  4.5 and 6.5 but decreased in those of  $p_{\rm H}$  8.2. The sol. N content of beet was increased by KCl but decreased by K2SO4 in the media. With increasing [K'] either as CI' or SO4", the total N of the beet was increased in nutrients of  $p_{\rm H}$  4.5 and 8.2 but was not affected in those of  $p_{\rm H}$ 6.5. Sugar contents were similarly affected. With  $NO_3'$  media the % of N in beet was higher and the % of sugar lower in the presence of Cl' than in that of  $SO_4''$ . K increased the chlorophyll content of leaves in NH. media at all  $p_{\rm H}$  vals. Ash contents of leaves in Cl' media were > those in SO<sub>4</sub>" media. Acid nutrients favoured absorption of K, and alkaline nutrients that of Ca and Mg. In soil cultures Cl' was more injurious than  $SO_4''$ . CH. ABS. (p)

Manurial experiments on oil palms. W. N. C. BELGRAVE (Malay. Agric. J., 1935, 23, 321-335). On the soils examined, mature palms did not respond to N or K fertilisers. Where P fertilisers were effective rock phosphate gave as good results as did basic slag.

A. G. P.

A. G. P.

Ovicidal action of winter washes. M. D. AUSTIN, S. G. JARY, and H. MARTIN (J. South-East. Agric. Coll. Wye, 1935, No. 36, 86—94).—Petroleum oils having  $\eta$  126—800 sec. (Redwood I) at 21·1° and unsulphonatable residue 60—100 vol.-% were equally effective against eggs of *L. pabulinus*. Bu<sub>2</sub> phthalate, methylcyclohexyl stearate, and diamyl tartrate were less efficient than petroleum fractions of similar boiling ranges. The val. of emulsions containing 4% of strained anthracene oil and 6% of semi-refined petroleum oil is confirmed. A. G. P.

Action of Nettolin in preventing scab in potatoes. H. RHEINWALD (Landw. Versuchs-Stat., 1935, 123, 205-207).—Scab was most abundant in unmanured soils, less on those receiving Nettolin, Huminol, and mineral manures, and least where farmyard manure was applied. A. G. P.

Bordeaux mixture-nicotine combinations against Aphis and apple scab. M. D. AUSTIN, S. G. JARY, and H. MARTIN (J. South-East Agric. Coll. Wye, 1935, No. 36, 95—99).—Bordeaux-sulphite lye and -cottonseed oil showed similar fungicidal efficiencies to that of simple Bordeaux mixture. Oil-Bordeaux caused less foliage injury and fruit russeting. When combined with nicotine (I) the modified Bordeaux washes gave better control of Aphis than did Bordeaux-(I). Two pre-blossom applications of the combined washes were not sufficiently effective to eliminate the use of tar distillate winter washes. A. G. P.

Control of apple scab : Allington pippin and Newton. Wonder. W. GOODWIN, N. H. PIZER, E. S. SALMON, and W. M. WARE (J. South-East. Agric. Coll. Wye, 1935, No. 36, 55—61).—The efficiency of Bordeaux mixture was ≮ that of a cottonseed oil-Bordeaux prep.

A. G. P.

Spraying trials against the raspberry beetle. R. A. H. GRAY and H. E. BROOKS (J. Roy. Hort. Soc., 1935, 60, 339—341).—Best results were obtained by two applications (bud-opening and petal-fall) of derris or  $BaSiF_6$  sprays. The former was more uniform in action. Use of dusts for the first treatment proved less successful. A. G. P.

Influence of season and advancing lactation on butterfat content of Jersey milk. R. B. BECKER and P. T. D. ARNOLD (J. Dairy Sci., 1935, 18, 389–399).— The % of fat in the milk varied inversely with temp. within certain limits. Vals. declined somewhat during the first 2 months of lactation and subsequently increased steadily until the twelfth month. Advancing lactation was of greater influence than season. A. G. P.

Maturity of beets.—See XVII. Vetch seeds.— See XX.

#### PATENTS.

Insecticide. D. H. GRANT, ASST. to STANCO, INC. (U.S.P. 1,976,780, 16.10.34. Appl., 11.6.31).—A wax (paraffin, beeswax, etc.) is dissolved in a refined white oil to produce a gel of m.p.  $> 32^{\circ}$ . An emulsifying agent (soaps, sulphonated vegetable oils, casein, triethanolamine, etc.) is also incorporated. The wax prevents penetration of oil into leaf tissues when the prep. is utilised as an oil emulsion insecticide. A. G. P. Insecticide and fertiliser. W. THORDARSON (U.S.P. 1,976,905, 16.10.34. Appl., 4.2.33).—Sulphite waste liquor is neutralised and mixed with a toxic alkalimetal salt (arsenite, arsenate, silicofluoride, etc.), and a solution of an appropriate salt (Fe, Cu, Zn, Ca, etc.) is added to produce a ppt. of a ligninsulphonate-metal salt complex. This is filtered, dried, and ground, and may be suspended in  $H_2O$  to form a spray. By use of sol. phosphates a corresponding phosphate complex is obtained and may be used as a fertiliser. A. G. P.

Superphosphate.-See VII.

### XVII.—SUGARS; STARCHES; GUMS.

Maturity and manufacturing value of [sugar] beets. F. GUILBERT (Bull. Assoc. Chim. Sucr., 1935, 52, 467—478).—The purchase of beets in France being based on d of juice, the author urges the necessity of a criterion of maturity which will indicate in course of growth the industrial val. of the roots from Sept. onwards. The ratio of reducing sugars to sucrose, which is a min. at maturity, appears suitable. At a rate which varies with alternations of sun and rain, this ratio gradually falls as the purity of the juice rises, and it provides a safe basis for estimating when a field will be ready for pulling, provided the beets are of fairly uniform size, a condition which recent methods of culture will ensure. J. H. L.

Phosphorus in sugar beets, fodder beets, and their hybrids. E. BOUGY (Bull. Assoc. Chim. Sucr., 1935, 52, 551-558).—The dry substance of decorticated sugar beets contained 0.29-0.44% P<sub>2</sub>O<sub>5</sub>, compared with 0.63-0.94% in fodder beets and mangolds; in hybrids of the two classes the % P<sub>2</sub>O<sub>5</sub> varied inversely with the sugar content. The quantity of P<sub>2</sub>O<sub>5</sub> insol. in hot dil. AcOH, in the decorticated and pulped roots, was much the same for both classes, about 0.05-0.07%on dry matter. The ratio of P and N insol. in dil. AcOH was the same (7:100) for all types of beets. J. H. L.

Flocculation of the pectin substances [of sugar beets] by alkaline-earth bases. A. CHAUDUN (Bull. Assoc. Chim. Sucr., 1935, 52, 425-432) .- The beet pectin in a solution having an angular rotation of  $+1.5^{\circ}$ in a 200-mm. tube was completely pptd. as a gel by excess of lime-water. Ca(OH)<sub>2</sub> at concns. of 0.003-0.02N produced flocculates after periods from 12 hr. to 15 sec. Sucrose at 5% concn. retarded flocculation and rendered the flocculates less dense. KCl retarded and Na citrate retarded or prevented flocculation. Na<sub>2</sub>CO<sub>3</sub> at moderate concn. produced partial flocculation, but NH<sub>3</sub> none. Ba(OH)<sub>2</sub> was rather more effective than Ca(OH)<sub>2</sub>. Beet pectin is precipitable completely by basic Pb acetate but only incompletely by the neutral salt even in excess. J. H. L.

Hyposulphites and sulphoxylates in [beet] sugar manufacture. R. BROQUET (Bull. Assoc. Chim. Sucr., 1935, 52, 385—388; cf. B., 1934, 343, 938).—Treatment of raw juice of average purity  $83 \cdot 5\%$  with 80 g. of  $ZnS_2O_4$  per ton of roots not only decolorised the juice and improved the filtration after first carbonatation, but also raised the syrup purity by 1—2%. Difficult pan boilings were improved by introduction of 2 kg. of Na sulphoxylate, which has a powerful decolorising effect at high temp. and lowers viscosity. J. H. L.

Crystallisation of sugar and formation of molasses. I, II. P. M. SILINE (Bull. Assoc. Chim. Sucr., 1935, 52, 265-275, 364-373) .- Modifying the theory of Noves and Whitney in view of Marc's observation that with increasing agitation the rate of crystallisation tends to become  $\infty$  the square of the concn. of the solution in excess of saturation (A., 1909, ii, 798, 983; 1910, ii, 834 ; 1911, ii, 193), the author assumes on the crystal face an immobile film of syrup, of thickness rand concn.  $c_1$ , < the concn., C, of the surrounding syrup but > the saturation concn. c. The rate of diffusion of sugar from the surrounding syrup to the crystal face is assumed to be  $k_1 (C - c_1)/r$ , and the rate of crystallisation at the face  $k_2 (c_1-c)^2$ . Equating these rates and transforming certain terms, an expression is derived for K, the rate of crystallisation per unit area of crystal face, in terms of T,  $\eta$ , and the supersaturation of the syrup (C-c). With suitable consts. this expression gives vals. of K in accord with Kucharenko's experimental vals. for pure sucrose syrups. At const. supersaturation K is approx. doubled by a rise of temp. of 10°. Other factors being const.,  $K \propto 1/\eta^{2.5}$ , but this does not apply to low-grade syrups the  $\gamma$  of which is partly structural, i.e., due to complex colloids which have little effect on diffusion. Determinations of Kwere made experimentally on beet sugar syrups and molasses at 42° under different conditions. Other factors being const., K was  $\infty$  the supersaturation (C-c), and independent of the size of the crystals (0.06-0.49 g.), the quantity of crystals present (16.7- $41 \cdot 1\%$ ), and the rate of stirring ( $1 \cdot 5 - 4$  r.p.m.). At the same degree of supersaturation, syrups of purity quotients (sugar % of total solids) 100, 79.5, 64.4, and 55.5%, having viscosities in the ratios 126:355:1527:33,650, gave vals. of K in the ratios 963:443:107:15respectively. J. H. L.

Working of [beet sugar] massecuites with rapidcooling Werkspoor crystallisers. L. XHIGNESSE (Bull. Assoc. Chim. Sucr., 1935, 52, 276-281).-In the two-massecuite system, the second massecuite purity should be  $\Rightarrow$  70-71%, and to obtain run-offs of this purity the first massecuite must be boiled at about 85% purity when ordinary crystallisers are used, and this involves boiling low-grade syrups with the first massecuite. With Werkspoor crystallisers run-offs of 70-71% purity can be easily obtained from massecuites of 88-89% purity. Data for such working, at the Santerre factory, are given. The massecuites are thinned in the crystallisers with syrup of about 71% purity and cooled to 40° without any formation of fine grain or difficulty in centrifuging. J. H. L.

Colouring substances of cane sugar. II. Anthocyanin and tannin. III. Colouring substances having the nature of humus bodies. IV. Colouring substances from a Formosan molasses. I. SAKUMA and I. MOMOSE (J. Soc. Chem. Ind., Japan, 1935, 38, 224—225 B, 225—227 B, 227—229 B; cf. B., 1935, 603). —II. An anthocyanin has been isolated from the outer rind of a Formosan cane and tannin from the cane tops. III. Limin and escenaratin have been isolated by the

III. Lignin and saccharetin have been isolated by the

alkali method from the bagasse during sugar manufacture. The extinction coeffs. of humic substances obtained by boiling sucrose, glucose, and fructose with 3% HCl or with 0.1% aq. Ca(OH)<sub>2</sub> have been measured.

IV. The extinction coeffs. of fractions separated by treating molasses with EtOH or AcOH have been determined. F. R. S.

Viscosity of concentrated sugar solutions. E. LANDT (Z. Wirts. Zuckerind., 1935, 85, 394–404; cf. B., 1931, 510).—Determinations on pure sucrose syrups of 60–73% concn., at 20–80°, with an improved Höppler viscosimeter (B., 1935, 79) having a precision of  $\pm 0.5\%$ , gave  $\eta$  vals. in most cases several % < those of Bingham and Jackson (A., 1919, ii, 268) and Bennett and Nees (B., 1930, 260). J. H. L.

History of density tables for sucrose solutions. D. SIDERSKY (Bull. Assoc. Chim. Sucr., 1935, 52, 432– 448).—A table is given relating sucrose contents to d20°/4° and d 28°/4°. Sucrose contents from 5 to 25% are given as g./100 g. (° Brix) and g./100 c.c., but from 26 to 70% only as g./100 g. They are graded by 0.01% from 0 to 2%, by 0.02 from 2 to 5%, by 0.05 from 5 to 10%, by 0.1 from 10 to 25%, and by 1% from 26 to 70%. Temp. correction tables cover the ranges 20° ± 5° and 28° ± 5°. All d are given to 5 decimal places. The tables are based on those of the 1898 German Imperial Commission (Plato's tables).

J. H. L.

Calcium saccharates. E. ANGELESCU and C. VLADESCU (Bull. Assoc. Chim. Sucr., 1935, 52, 374-384).-In solutions containing CaO and sucrose (I) in a given ratio, the temp. at which pptn. begins shows a well-defined min. val. at a certain dilution (A., 1932, 229). Such a min. is also observed if the solutions contain electrolytes or gelatin, but the temp. at which pptn. begins is higher in presence than in absence of these substances at all dilutions of the CaO-(I) mixture. The rise in temp. is probably due to a virtual increase in the concn. of CaO and (I), due to withdrawal of part of the H<sub>2</sub>O by hydration of the electrolytes or gelatin; it is greater with Ca salts than with Na salts at equiv. concn. Electrolytes increase the degree of dispersion of the ppt. produced, which at high concns. may be a gel. In the pptn. of Ca<sub>3</sub> saccharate from impure sugar solutions, dilution to a certain point lowers the temp. at which pptn. occurs and improves the filtering quality of the ppt., but beyond that point free CaO will separate with the saccharate. J. H. L.

Construction and working of potato flake factories. A. SCHWIETER (Z. Spiritusind., 1935, 58, 235-236, 238-239).—The dimensions and design of each element of the plant are discussed, as are yields and costs of manufacture. T. H. P.

Yield and yield tables [in potato-starch manufacture]. B. HOSPES (Z. Spiritusind., 1935, 58, 237-238). —Yield tables differ owing to their varying basis, some referring to total starch produced and others only to starch of high grade. The losses involved in various ways prior to the production operations and during each stage of the manufacture are discussed. According to Saare 100 kg. of potatoes containing 18% of anhyd. starch give 18 kg. of commercial dry starch under the most efficient working, or 12.6 kg. with poor working; according to Sprockhoff the yields are 24 and 18.0 kg. respectively. T. H. P.

Sugar beets and cane. Sugar content of American artichoke.—See XVI.

#### PATENTS.

Decolorisation of sugar juices. DEUTS. GOLD- U. SILBER-SCHEIDEANSTALT VORM. ROESSLER (B.P. 428,740, 10.7.34. Ger., 10.7.33).—Glucose syrups, beet syrups, etc. are treated at 60—90° with  $H_2O_2$  and "surfaceactive materials" (bone char, active C). Addition of 0.1-0.15% of 30%  $H_2O_2$  to 70% beet-sugar syrups, just before treatment with active C, greatly enhances the decolorising effect of the latter. The quantity of  $H_2O_2$  added should be such that none remains undecomposed after the C filtration. J. H. L.

Manufacture of non-mottling and non-hardening maple sugar [blocks]. L. SKAZIN and J. F. SNELL (U.S.P. 1,970,870, 21.8.34. Appl., 31.3.32).—Maple sap or syrup is boiled in an open vessel until the temp. reaches 125°, then allowed to cool, and continuously stirred until cold. The cryst. mass obtained, containing about 2% of H<sub>2</sub>O, is pressed into blocks occupying  $30-31\cdot4$  cu. in. per lb. J. H. L.

Isolation of lævulose as lime [calcium] lævulosate. R. M. HIXON, J. H. MCGLUMPHY, J. W. EICH-INGER, JUN., and J. H. BUCHANAN, ASSTS. to IOWA STATE COLL. OF AGRIC. & MECHANIC ARTS (U.S.P. 1,970,605, 21.8.34. Appl., 30.7.31).—A fructose solution and a CaO suspension are introduced together slowly, in the correct proportions and at room temp., into a vessel containing a large quantity of Ca fructosate seed, and continuously stirred. The product is granular and can be easily filtered and washed. Apparatus for carrying out the process continuously is described. J. H. L.

[Maize] starch-making system. INTERNAT. PATENTS DEVELOPMENT CO. (B.P. 425,936, 4.4.34. U.S., 26.4.33).—In a modification of the process of B.P. 419,286 (B., 1935, 120) the mill starch, or a portion of it, is treated in settling vessels to separate not only part of its  $H_2O$ , which is returned to working (*ibid.*), but also part of its starch in a substantially gluten-free condition requiring only washing without tabling. The rest of the mill starch is tabled, and the whole of the resulting gluten- $H_2O$  is returned to the maize steep. J. H. L.

Conversion of starch [in flours]. H. F. BAUER, Assr. to STEIN, HALL MANUFG. Co. (U.S.P. 1,969,347, 7.8.34. Appl., 24.7.31).—In the manufacture of dextrinised products from sensibly dry flour, interference of gluten, fat, etc. with the conversion is overcome by addition of  $SO_2$ , sulphites, urea, or other substances which inhibit gel formation by these constituents. Alternative methods are to use a dry converting agent (AlCl<sub>3</sub> or HCl gas), or a sensibly dry mixture of a converting agent with a carrier starch as starch, which can be intimately mixed with the flour, or a converting agent dissolved in a non-aq. solvent (AlCl<sub>3</sub> in CCl<sub>4</sub> or HCl in EtOH). J. H. L.

Insert for diffusers for extraction of sugar from sugar-beet cossettes. H. SCHIEBEL (B.P. 426,820, 10.11.33. Ger., 11.11.32).

Glues from starch.—See XV.

## XVIII.—FERMENTATION INDUSTRIES.

Barley varieties for brewing. L. R. BISHOP (J. Inst. Brew., 1935, 41, 329—334).—The relevant analytical characters of numerous barley samples are described and discussed. Barleys having characters which appear to agree best with particular requirements are listed. I. A. P.

Influence of biological acidification on the composition of wort and beer. P. KOLBACH, G. HAUSS-MANN, and G. WILHARM (Woch. Brau., 1935, 52, 233-238, 241-246).-Lactic acid (I) (0-1 kg. to 10 cwt. of malt) was added either to the mash and/or to the wort, suitable proportions of a mash soured with B. Delbrücki being added to give the desired concn. of (I). Detailed analytical figures are quoted for cast worts and beers thus obtained by different mashing systems from different malts under brewery conditions, the results being discussed. In general, acidification increased the buffer action of wort or beer, while causing decreases in  $p_{\rm H}$ , acid formed during yeast fermentation, colour, and bitter principles. Changes in N relationships were somewhat irregular ; sol. N and formol-N usually increased with fall in  $p_{\rm H}$  although not with any regularity when the wort alone was acidified. I. A. P.

Oils to prevent frothing [in beet distilleries]. P. BERTI (Bull. Assoc. Chim. Sucr., 1935, 52, 286–288; cf. Modiano, B., 1934, 422).—An effective agent is made by adding 1 vol. of  $H_2SO_4$ ,  $d \cdot 1 \cdot 84$ , to 3 vols. of olive oil and stirring. The mixture becomes warm, darkens, froths copiously, and evolves some  $SO_2$ . After a few min. it may be added in small quantities to the fermenting beet wort, as required, but should not be used after 5–6 hr. J. H. L.

Filtration of draught beers. R. B. ULLMAN. G. M. PARSONS (J. Inst. Brew., 1935, 41, 319-328).—A debate. I. A. P.

Presence of sorbitol in pure grape wines. E. VOGT (Z. Unters. Lebensm., 1935, 69, 587—591).—Of 44 wines examined only 5, all Ruländer, contained sorbitol. The amount was too small to justify suspicion of adulteration with fruit wine. E. C. S.

Determination of hydroxymethylfurfuraldehyde and lævulosin in port wine and other sweet wines. C. I. KRUISHEER, N. J. M. VORSTMAN, and L. C. E. KNIPHORST (Z. Unters. Lebensm., 1935, 69, 570-582).-A method for the determination of hydroxymethylfurfuraldehyde (I) in sweet wines by extraction with Et<sub>2</sub>O and pptn. as phloroglucide is described. Other aldehydes interfere, and must be removed. Failure to do this renders von Fellenberg's method (A., 1934, 1241) untrustworthy. Oxidation of (I) with atm. O2 must be avoided. Sweet wines, such as pure port wine, free from conc. must or caramel, contain no (I), or only traces, but if conc. must or caramel has been added 100-1000 mg. of (I) per litre may be found. Adulteration of port wine with certain other sweet wines may be detected by this means. Lævulosin (cf. B., 1930, 1043) is also present in small quantities in some sweet wines. E. C. S.

Chemical criteria for brandy. G. BÜTTNER (Z. Unters. Lebensm., 1935, 69, 463-467).-The possibility

of using the % of higher alcohols, lauric acid, and total esters as criteria for the evaluation of brandy is discussed. E. C. S.

Testing of [potable] spirits with the quartz [ultra-violet] lamp. H. BARSCH (Z. anal. Chem., 1935, 101, 388—392).—When spirits are subjected to the Micko distillation using a special rectifier to obtain 7 fractions the 4th or the 5th or both fractions of a true brandy give(s) a strong blue fluorescence under ultraviolet radiation. Since some varieties of rum also provide fractions giving a similar fluorescence the test does not preclude the possibility of adulteration. A. R. P.

Titrimetric determination of alcohol with dichromate. H. KREIPE (Z. Spiritusind., 1935, 58, 228— 229).—Using a modification of the micro-method of Widmark (A., 1922, ii, 789), EtOH can be determined in 0.5 c.c. of suitably diluted alcoholic liquids (*e.g.*, liquors obtained during vinegar manufacture, wines). As modified, the method is more convenient in practice than, and gives results agreeing closely with, the pyknometric method. I. A. P.

Influence of solvents on butyl alcohol-acetone fermentation. L. I. TSCHEKAN (Microbiology, U.S.S.R., 1934, 3, 266–273).—In the fermentation of maize meal with *Cl. acetobutylicum* the amount of  $COMe_2$  usually formed (0.5-0.7%) does not affect the fermentation. 1% of BuOH lowers gas production by 50% and 1.5%of BuOH arrests fermentation. CH. ABS. (p)

**Preparation of lactic acid by direct fermentation** of barley. V. KULIKOV and V. KOLOGRIVOVA (Microbiology, U.S.S.R., 1933, 2, 353—359).—Barley contains sufficient amylase to effect saccharification of the starch without the aid of malt. Any residual starch is saccharified during lactic fermentation (48—49°). Yields exceed those obtained with the aid of malt. CH. Abs. (p)

Conditions for the semi-plant production of citric acid. S. KOSTUITSCHEV and V. BERG (Bull. State Inst. Agric. Microbiol. U.S.S.R., 1933, 5, 8—27).— After 2 days' growth at 30—32° of young cultures of A. niger, the nutrient beneath the fungus is replaced by a 20% sugar solution without minerals or N. Production of citric acid is complete in 4 days. Yield 64% of the sugar fermented. The fungus layer should be 1 cm. thick. The yield is reduced if evaporation is permitted during fermentation. CH. ABS. (p)

Oxidisability and iodine values for differentiating fermentation and distillation vinegar. A. SCHMIDT (Z. Unters. Lebensm., 1935, 69, 472–478).—The oxidisability vals. of fermentation and distillation vinegar were 3.5-8 and 1-1.5 c.c. of 0.1N-KMnO<sub>4</sub> respectively, and the I vals. 30-60 c.c. and < 20 c.c. of 0.01N-Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. Samples must be adjusted for comparison to  $\geq 3\%$  of AcOH. E. C. S.

Detection of fusel oils.—See III. Determination of free acid.—See XIX.

#### PATENT.

Clarifying and preserving liquids.-See XIX.

### XIX.—FOODS.

Detection of rye and wheaten flour in mixed flour by means of the trifructosan content. H. WERNER and H. VOLGER (Z. Unters. Lebensm., 1935, **69**, 555— 562; cf. B., 1932, 815).—The trifructosan (I) content of wheaten flour is approx. const. (reduction val.  $\equiv 29$  mg. CuO per g. of dry matter), but that of rye flour varies (59—111 mg. CuO per g.). The composition of a mixture can, therefore, be determined only when the (I) content of the rye flour is known. During baking (I) is lost, so that the composition of the flour cannot be determined. E. C. S.

Micro-methods for determining the quality of wheat: a new micro-method: H. ENGELKE (J. Landw., 1935, 83, 89—106).—Comparative vals. of various standard tests are discussed. The gluten-swelling test is adversely criticised. Micro-apparatus for examination of dough fermentation is described. A. G. P.

Tallowiness and acidity of wheat flour. H. Y. CHANG and Y. S. CHAO (J. Chinese Chem. Soc., 1935, 173—175).—Tallowiness of flour developed during storage is due to oxidation of  $Et_2O$ -extractable oil; too low a  $H_2O$  content makes the flour tallowy and too high  $H_2O$  content renders it musty. F. R. S.

Oxidation-reduction potentials of flours. P. POTEL and R. CHAMINADE (Compt. rend., 1935, 200, 2215—2217).—Oxidation-reduction potentials were measured at 30° with a Pt electrode on a mixture of 1 pt. of flour and 1.5 pts. of a buffer solution of  $p_{\rm H}$  6.2 made in a N<sub>2</sub> atm., PhMe being added to prevent fermentation, and the whole covered with a layer of vaseline. Characteristic potentials were observed, which were correlated with extensimeter measurements.

H. J. E.

Gluten hydrolysis and preparation of d-glutamic acid hydrochloride. C. L. TSENG and M. HU (J. Chinese Chem. Soc., 1935, 154—172).—A review of the literature. F. R. S.

Milk of a typical herd of shorthorn cows. II. J. GOLDING, J. MACKINTOSH, and E. C. V. MATTICK (J. Dairy Res., 1935, 6, 6-25; cf. B., 1933, 362).—The % fat in the morning milk declined in spring when cows were on young grass. During a drought period there was a marked decrease in solids-not-fat in both morning and evening milk. This is associated with low P vals. Other seasonal variations are discussed. A. G. P.

Seasonal variation of the percentage butter fat content of milk : results of individual cow tests. C. D. OXLEY (J. Dairy Res., 1935, 6, 1-5).—Standard deviations of vals. for morning and afternoon milkings throughout the year are examined. Sources of error due to the nature of the sample and seasonal variations are discussed. A. G. P.

Seasonal variations in the physical properties and nutritive value of cow's milk-serum. F. E. STIRN, C. A. ELVEHJEM, and E. B. HART (J. Dairy Sci., 1935, 18, 333–336).—The winter product was clarified with greater difficulty and had a lower nutritive val. (notably less vitamin-B). A. G. P.

Statistical examination of the interrelationship and variability of plate count, presumptive coliform content, and keeping quality of raw milk. H. BARKWORTH (J. Dairy Res., 1935, 6, 26–48).—The variabilities of the three factors are too great to permit the prediction of any one from vals. for the other two. A. G. P.

Oxidised flavour in milk. I. Probable relation of lecithin to the flavour. L. M. THURSTON, W. C. BROWN, and R. B. DUSTMAN (J. Dairy Sci., 1935, 18, 301-306).—The "oxidised" flavour of milk probably differs from the "tallowy" flavour of oxidised butter fat, and is associated with changes in lecithin rather than in fatty constituents. A. G. P.

Rational pasteurising of milk. K. GORINI (Arch. Mikrobiol., 1935, 6, 1–8).—Desirable limits of bacterial elimination are discussed. A. G. P.

Steam pressure in milk-heating and the evaporation of whey. O. T. KORITNIG (Milch. Zentr., 1935, 64, 255–257).—The advantages of low-pressure systems are discussed. A. G. P.

Effect of heating milk on the time during which curds remain in the abomasum of calves. F. N. MORTENSON, D. L. ESFE, and C. Y. CANNON (J. Dairy Sci., 1935, 18, 229–238).—Boiled and autoclaved milks coagulate more slowly in, but are evacuated more quickly from, the stomach than is raw milk. Heating lowers the curd tension of milk and facilitates the breaking up of the mass in the stomach. A. G. P.

Germicidal efficiency of lye and chlorine solutions for sterilising milk machines and cream separators. A. C. FAY, W. J. CAULFIELD, and W. H. RIDDELL (J. Dairy Sci., 1935, 18, 239–245).—Solutions containing 100 p.p.m. of available Cl or 0.3% NaOH are effective for tubing and teat-cups provided the rack method is used. In immersion methods Cl<sub>2</sub> solutions are the less satisfactory. For rinsing separators Cl<sub>2</sub> solutions are preferable and should contain  $\leq 200$  p.p.m. of Cl. Lye tends to corrode separator discs. The bacterial efficiency of solutions containing 0.5-1.0% of NaOH is practically the same. A. G. P.

Relation of proteolytic enzyme activity to the proteolytic organisms found in [milk-]separator slime. G. SPITZER and E. H. PARFITT (J. Dairy Sci., 1935, 18, 267—272).—A relationship is established between the nos. of proteolytic bacteria in separator slime and its proteolytic activity. The latter is greater in summer than in winter and is largely destroyed (70%) by heating at 63° for 30 min. or 74° for 10 min.A. G. P.

Heat-stability of evaporated milk made from hard- and soft-curd milks and milk from mastitisinfected udders. R. C. WELCH and F. J. DOAN (J. Dairy Sci., 1935, 18, 287–294).—Hard- and soft-curd milks show similar heat-stability when conc. Mastitismilk is less stable and is affected abnormally by pretreatment and by chemical treatments. A. G. P.

Physical effects of freezing on milk and cream. B. H. WEBB and S. A. HALL (J. Dairy Sci., 1935, 18, 275-286).—Slow freezing of milk or cream caused a gradual pptn. of the caseinate system and an intermediate destruction of the fat emulsion. The effect on the fat in cream is diminished by addition of sucrose or solids-not-fat prior to freezing. Homogenisation retarded the separation of fat occurring in frozen lowfat creams. Freezing destroyed fat-clumps formed in homogenised cream and restored its initial heat-stability. Freezing of skim milk produced a gradual increase in the size of the casein aggregates. Fresh whole milk may be pasteurised, evaporated to  $\frac{1}{3}$  of its wt., and frozen without detriment, and is indistinguishable from fresh milk when reconstituted. When frozen homogenised cream is thawed at a temp.  $\leq$  the m.p. of the fat, clear serum may be drained off and the fat-casein mixture is utilisable for the prep. of normal casein or for increasing the protein content of ice-cream mixtures. A. G. P.

(A) Coagulation process [in milk]. (B) Effect of ultra-short waves on Streptococcus cremoris. H. KÖRBER (Oesterr. Chem.-Ztg., 1935, 38, 122–123, 123–124).—(A) The effects of short-wave (4 and 15 $\mu$ ) irradiation on the coagulation time of raw and pasteurised milks are examined (cf. A., 1934, 1032). Dialysis accelerates coagulation without appreciably changing the  $p_{\rm H}$  of the milk. The mechanism of the process of coagulation is discussed.

(B) The increased titratable acidity of milk following irradiation varied with the  $\lambda$  used (4 to > 15  $\mu$ ).

A. G. P. Titratable acidity of milk. I. Influence of various buffers on the titration curves of fresh and sour milk. II. "Buffer curves" of milk. W. J. WILEY (J. Dairy Res., 1935, 6, 72—85, 86—90).—I. The influence of  $PO_4^{\prime\prime\prime}$ , Ca, casein, and citrate on titration curves (phenolphthalein) is examined in the range  $p_{\rm H}$   $4\cdot 8$ — $8\cdot 0$ . Each constituent tends to change the form of the curves but the nature of the change cannot be predicted from the titration curves of the individual constituents.

II. The pronounced max. in the titration curve of milk is related to the dissolution and pptn. of Ca phosphate. The difference between the curves for milk and acid whey is attributable to the influence of casein on the Ca phosphate equilibrium. A. G. P.

Determination of [milk] curd tension by use of hydrochloric acid-pepsin coagulant. D. MILLER (J. Dairy Sci., 1935, 18, 259–265).—Excess of CaCl<sub>2</sub> in Hill's coagulant retards coagulation and produces a subnormal curd tension. Pepsin (0.45 g.) in 0.4% HCl (100 c.c.) gives truer vals. and records more definitely the softening effect, on curds, of brief boiling. On the other hand the CaCl<sub>2</sub> coagulant indicates differences in treated whole milks which were not apparent when pepsin-HCl was used. A. G. P.

Accuracy of determination of fat [in milk]. W. WILSMANN (Milch. Zentr., 1935, 64, 271-273).-A method of checking the accuracy of methods of determination of fat is described. E. C. S.

Detection of added water in milk. B. DAVIES (J. Dairy Res., 1935, 6, 56—71).—Methods of utilising the n, electrical conductivity,  $\kappa$ , and rotation of the plane of polarisation (P) of milks in detecting added H<sub>2</sub>O are examined. The product  $P \times \kappa \times (n_{\text{milk}} - n_{\text{H2O}})$  tends to become a const. for a given herd. The ratio of the product for a standard pure milk to that for the test sample may serve as an index of watering. A. G. P.

Simple titrimetric determination of milk phosphates. G. T. PVNE (Sci. Proc. Roy. Dublin Soc., 1935, 21, 223–229).—To determine sol. phosphate, 1 drop of saturated aq.  $K_2C_2O_4$  is mixed with 10 c.c. of the milk containing 5 drops of 0.5% phenolphthalein and after 1—2 min. the mixture is titrated with 0.1N-NaOH to the colour of 10 c.c. of milk containing 1 drop of 0.01% alcoholic magenta. 2.0 c.c. of 0.1N-NaOH are added, followed by 1 c.c. of *M*-CaCl<sub>2</sub>, and the liquid is titrated after 1—2 min, with 0.1N-acid (*a* c.c.) to the standard tint. 2.0 - a c.c. represents the acidity developed by the formation of Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>, *i.e.*, 2K<sub>2</sub>HPO<sub>4</sub> + 3CaCl<sub>2</sub>  $\rightarrow$  Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> + 4KCl + 2HCl, and is equiv. to sol. PO<sub>4</sub>. 1 c.c. 0.1N-acid  $\equiv 3.1$  mg. P. For total phosphate, 1 c.c. of aq. K<sub>2</sub>C<sub>2</sub>O<sub>4</sub> is added to the re-neutralised milk, and after 5—6 min. titration with 0.1N-acid is continued. HCl (*b* c.c.) + K<sub>3</sub>PO<sub>4</sub> [from the action of K<sub>2</sub>C<sub>2</sub>O<sub>4</sub> on Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>]  $\rightarrow$  KCl + K<sub>2</sub>HPO<sub>4</sub>. *b*  $\equiv$  total inorg. PO<sub>4</sub>''' (1 c.c.  $\equiv 3.1$  mg. P). The results are in good agreement with recognised methods.

Detection of formaldehyde in milk by the methylene-blue reduction test. A. C. FAY (J. Dairy Sci., 1935, 18, 327–331).—Milks showing a reduction of < 1 hr. may be suspected to contain CH<sub>2</sub>O. The range of dilutions of CH<sub>2</sub>O which effectively retard bacterial growth and delay coagulation time without affecting flavour lies between 1 : 15,000 and 1 : 25,000.

A. G. P.

Determination of dirt in milk. W. PLÜCKER (Z. Unters. Lebensm., 1935, 70, 96—107).—The insol. matter in milk is collected on a standard silk filter, suspended in  $H_2O$ , and centrifuged in a leucocyte tube. Sol. fæces can be detected only by bacteriological examination. To inhibit the growth of *B. coli* etc. in pasteurised milk a pure culture of lactic acid bacteria must be added. E. C. S.

Composition of media for bacteriological analysis of milk. C. S. BOWERS and G. J. HUCKER (New York State Agric. Exp. Sta., Tech. Bull., 1935, No. 228, 42 pp.).—Addition of 0.5% of skim milk to the standard agar medium increased bacterial counts, especially in pasteurised milk. A tryptone–glucose–skim milk agar increased the no. and size of colonies in high-count milks and in samples containing mastitis organisms and heat-resistant types. The nos. from low-count milks were much less affected. A. G. P.

Determination of ammonium salts in milk as a method of hygienic examination. A. I. BURSTEIN and F. S. FRUM (Z. Unters. Lebensm., 1935, 69, 421— 431).—A rapid method of determination of NH<sub>3</sub> in milk based on the exchange reaction with permutit is described. Fresh milk contains 0.10-0.12 mg. of NH<sub>3</sub> per 100 c.c., increasing to > 0.23 mg. in 24 hr. when kept at  $10-12^{\circ}$  under clean conditions. > 0.23mg. of NH<sub>3</sub> per 100 c.c. indicates that milk is > 24 hr. old, or has been kept under insanitary conditions. Addition of Na<sub>2</sub>CO<sub>3</sub> increases the rate of NH<sub>3</sub> formation. Occasionally, after an initial rise, a fall in the % of NH<sub>3</sub> is observed. The % of NH<sub>3</sub> is a certain index of the extent of bacterial proteolysis. E. C. S.

Milk contamination and the methylene-blue reduction test. H. R. THORNTON *et al* (Canad. Publ. Health J., 1934, 25, 284—294).—The only factors which invariably caused reduction of methylene-blue within  $5 \cdot 5$  hr. were utensil contamination, inadequate cooling, and the presence of abnormal milk. CH. ABS. (*p*) Fireh Chemicel Abet+acta-B

Neutralisation of cream for butter making. W. J. WILEY (J. Dairy Res., 1935, 6, 91–102).—Pasteurisation has little effect on the  $p_{\rm H}$  or titratable acidity of cream "neutralised" by Ca(OH)<sub>2</sub>. The reduction in acidity due to addition of Ca(OH)<sub>2</sub> differs from that calc. from the NaOH titration to an extent which depends on the  $p_{\rm H}$  of the neutralised cream. When NaHCO<sub>3</sub> is used for neutralisation the loss of CO<sub>2</sub> during pasteurisation is the principal factor deciding the degree of neutralisation attained. A. G. P.

Factors influencing the separation of whey in ice-cream mixes containing vegetable stabilisers. I. A. GOULD and P. S. LUCAS (J. Dairy Sci., 1935, 18, 307-315).—Separation of whey depends on the milk constituents (probably protein) rather than on the stabiliser. A. G. P.

Factors affecting economical manufacture, uniformity in composition, and quality of butter. D. H. NELSON (J. Dairy Sci., 1935, 18, 207—211).— For most effective working the overrun should be so controlled that the fat content of the butter is 80—81%. The Kohman method of analysing each batch of butter is preferred. A. G. P.

Hydrogen-ion concentration of creamery waters and their relationship to washing butter. N. S. GOLDING (J. Dairy Sci., 1935, 18, 359—371).—Alkaline wash-H<sub>2</sub>O tends to remove slightly more protein from butter than does acidified H<sub>2</sub>O. A. G. P.

Detection of butter fat in confectionery. Changes in butter fat during baking, or due to admixture with egg substances. G. PRANGE (Z. Unters. Lebensm., 1935, 69, 548—555).—The baking of pastry prepared with butter causes an increase in n, acid val., and in all vals. for volatile acids except the A val. (the Polenske and B vals. being most affected), and a decrease in I val. Admixture with flour fat causes an increase in n and I val. Admixture with egg oil causes a decrease in all the above vals. except n, acid val., and I val., the effect of baking being less marked. For the determination of the % of butter fat in a mixture the A and B vals, are most useful. Adulteration can be detected after baking. E. C. S.

Distribution of fat in cheese with high fat content. F. KIEFERLE (Z. Unters. Lebensm., 1935, 70, 107—112).—The fat in 20 Brie cheeses from the same batch varied from 50 to 56%. In a single Roqueforttype cheese, the fat varied from  $57 \cdot 4\%$  in the outer region to  $64 \cdot 7\%$  in the inner. E. C. S.

Refractometric determination of fat in cheese. W. LEITHE (Z. Unters. Lebensm., 1935, 70, 91–96).— The method is essentially the same as that previously described for chocolate and milk (cf. B., 1935, 172, etc.).  $1-C_{10}H_7Br$  is used as extractant. E. C. S.

Mechanical control of the fat content of Swiss cheese. W. V. PRICE and G. C. NORTH (J. Dairy Sci., 1935, 18, 149—161).—Removal of > 10% of the total fat from milk or the establishment of casein : fat ratios > 0.81 tends to produce cheese having < the legal limit of fat. The necessity of thorough analytical control in processing is emphasised. A. G. P.

Bacteriology of Swiss cheese. II. Bacteriology of the cheese in the press. W. C. FRAZIER, L. A.

BURKEY, A. J. BOYER, G. P. SANDERS, and K. J. MATHE-SON (J. Dairy Sci., 1935, **18**, 373–387).—The rapid decline in  $p_{\rm H}$  of the cheese in the first 7—8 hr. is largely due to the activity of *Streptococcus thermophilus* at a stage when the buffer capacity of the cheese is small. *Lactobacillus* organisms then become active and tend further to lower the  $p_{\rm H}$  towards a min. of  $p_{\rm H}$  5·0. The acidity developed in the first 24 hr. is an important factor controlling the course of subsequent fermentation. A. G. P.

Vegetable casein from soya bean and peanut. K. C. CHANG and Y. S. CHAO (J. Chinese Chem. Soc., 1935, 3, 177–182).—Casein may be extracted with  $Et_2O$ from soya bean (22% yield) and peanut (12%). The composition differs from that of milk casein but the properties are very similar. F. R. S.

Chemistry of hen's egg. J. GROSSFELD (Z. Unters. Lebensm., 1935, 70, 82—91).—The composition of the egg and changes which take place during storage are reviewed. E. C. S.

Decomposition and conservation of hens' eggs. A. JANKE and L. JIRAK (Z. Unters. Lebensm., 1935, 69, 434-452).—The advantages and disadvantages of the various methods of storing eggs are discussed in relation to the refractometric method of examination (A., 1934, 1021). For cleaning and disinfecting the surface aq. NaOCl is recommended. Preservation by impregnation of the shell with a pasty emulsion resulted in the least deterioration. E. C. S.

Detection of coal-tar dyes in hens' eggs. J. GROSS-FELD and H. R. KANITZ (Z. Unters. Lebensm., 1935, 69, 582—584).—Capsanthin, carotene, and bixin are decolorised by HNO<sub>3</sub>, but the EtOH-Et<sub>2</sub>O extract of the yolks of eggs from hens fed on coal-tar dyes is not bleached when treated with HNO<sub>3</sub>. Wool may be dyed by dissolving the dye from the HNO<sub>3</sub>-treated extract in light petroleum, taking it up in EtOH, and pouring it into H<sub>2</sub>O, with which the wool is treated in the usual way. E. C. S.

Determination of egg volume. L. W. JIRAK (Z. Unters. Lebensm., 1935, 69, 431-434; cf. B., 1934, 523).—A method of determination is described. The approx. age of an egg may be calc. from its wt. and vol. E. C. S.

Arsenic storage in game fish. M. M. ELLIS (Copeia, 1934, No. 2, 97).—Fish from  $H_2O$  polluted with As ore contained appreciable proportions of As in the viscera. Larvæ of the stone fly, on which fish feed, also contained As. The possibility of As poisoning through eating such fish is discussed. CH. ABS. (p)

Occurrence of acetaldehyde in tropical fruits. A. STEINMANN (Z. Unters. Lebensm., 1935, 69, 479–481). —The examination of 50 varieties of tropical fruit confirms Griebel's generalisation (cf. B., 1924, 922) that MeCHO occurs in all fruits which possess a sharp taste before ripening and lose their sharpness during or after ripening, E. C. S.

**Pineapples.** R. R. FOLLETT-SMITH (Divisional Repts. Dept. Agric. Brit. Guiana [1933], 1934, 89–100).— The acidity and  $p_{\rm H}$  of the juice of local varieties recorded compared favourably with those of the same varieties grown elsewhere. CH. ABS. (p) **Botrytis** rot of grapes and its control during 1933—4. S. J. DU PLESSIS (Farming in S. Africa, 1934, 9, 395—397).—Partial control was obtained by dusting the coloured and maturing grapes in the field with  $CuSO_4$ . Development of the fungus during storage was not thus prevented. Cu–CaO preps. and  $Na_2S_2O_3$ kaolin were ineffective. Wrapping papers soaked in  $CuSO_4$ , KI, Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, or KMnO<sub>4</sub> did not control disease in storage. Addition of  $(NH_4)_2CO_3$  (0·2 g. per bunch) placed in wrappers controlled *Botrytis* without injury to fruit or deterioration of flavour. CH. ABS. (p)

Spray residues on fruit and vegetables. C. H. RICHARDSON (Rept. Iowa State Hort. Soc., 1933, 68, 73-82).—A review of developments relating to Pb, As, and F residues. CH. ABS. (p)

Composition of local [British Guiana] honey. R. R. FOLLETT-SMITH (Divisional Repts. Dept. Agric. Brit. Guiana [1933], 1934, 100).—Average vals. were; total sugars 75.8%, sucrose 8.67%, and  $p_{\rm H}$  4.23. CH. ABS. (p)

Microscopy of bee honey. H. THALER (Z. Unters. Lebensm., 1935, 69, 405–407).—The characters of an absorbing trichome of *Bromelia* observed in honey are described. E. C. S.

Colour and chlorogenic acid content of coffee beans during roasting. W. HOEPFNER (Z. Unters. Lebensm., 1935, 70, 112—117).—The changes in colour, and in % of H<sub>2</sub>O, chlorogenic acid, and extract, of coffee treated by the Lendrich process and of untreated coffee during roasting are compared. E. C. S.

Removal of caffeine from coffee. E. Bossing (Chem.-Ztg., 1935, 59, 684—685).—A review.

Rapid determination of arsenic and lead (insecticides) in foodstuffs. P. BERG and S. SCHMECHEL (Z. Unters. Lebensm., 1935, 70, 52—60).—The determination of As in apples by Sanger-Black and Smith's method (cf. A., 1932, 786) and of Pb in apples and various beverages by a colorimetric method with dithizone is described. E. C. S.

Determination of free acid in strongly coloured [alimentary] liquids. H. THALER and M. DE MINGO (Z. Unters. Lebensm., 1935, 69, 407–409).—Comparative results of titration of wines, coffee essences, etc. by the drop method and with the quinhydrone electrode are given. The results are concordant. E. C. S.

Digestibility and nutrient value of sweet-lupin seed for ruminants. E. MANGOLD and H. STOTZ (Landw. Versuchs-Stat., 1935, 123, 147—158).—Vals. obtained for yellow and blue sweet lupins in sheepfeeding trials are recorded. A. G. P.

Biological value of the protein of sweet-lupin seed in pig-feeding. W. LINTZEL and E. MANGOLD (Landw. Versuchs-Stat., 1935, 123, 133—146).— Feeding-trial data are recorded. The biological val. of sweet-lupin grain is increased by feeding in conjunction with steamed potatoes. A. G. P.

Ash-free plates for filtration in qualitative and quantitative work, especially for the determination of "sand" in feeding-stuffs. W. LEPPER (Landw. Versuchs-Stat., 1935, 123, 235-236).—The use of a

compressed filter-pad in conjunction with a perforated Ni disc is described. A. G. P.

Microscopical examination of feeding-stuffs after screening and chloroform treatment. F. BART-SCHAT (Angew. Chem., 48, 1935, 549-550).-A 10-g. sample is classified in a 1.0-mm, and 0.5-mm, double sieve. The % distribution between the fractions gives information as to the grain etc. used. Living mites, if present, are found in the middle fraction. The colour of the fine fraction is noted. The second fraction is treated with chloral hydrate (I) and examined microscopically. This is sufficient to detect adulteration of barley meal with rye. A similar examination of the third fraction detects moulds. A further portion is treated with CHCl<sub>a</sub>, when extraction with (I) enables ergot, rust spores, etc. to be seen. The ppt. from the CHCl<sub>3</sub> includes mineral matter such as CaCO<sub>3</sub> and also sugars. C. I.

Enamel of cooking vessels.-See VIII. Ac, in fats. Synthetic butter.—See XII.

#### PATENTS.

Manufacture of flakes and the like from plastic materials containing vegetable matter, especially cereals. A. U. SÄRNMARK (B.P. 431,504, 2.10.33. Swed., 3.10.32) .- Paste is squirted in an intermittent manner through nozzles, the blobs falling on a surface (S) in the form of flakes. S may be a conveyor belt and the flakes are dried (and roasted) in several stages, e.g., at (1) 50-100°, (2) 80-160°, (3) > 140°. [Stat. ref.] B. M. V.

Economic production of milk powder from whole milk. J. B. M. KNUTSEN (B.P. 432,075, 2.10.34. Austral., 14.10.33).—A complete scheme is outlined by which milk, after condensing, is spray-dried in an atm. of H<sub>2</sub>. Producer gas is employed both as the source of the  $H_2$  and also for developing the necessary power for the plant. The H<sub>2</sub> used in the drying chamber is re-circulated, after suitable drying, and losses are made up from the main source of supply. E. B. H.

Preservation of perishable products, especially milk and the like, by freezing. J. DE L. HENNESEY, N. A. SAHLIN, and R. NILSSEN (B.P. 431,868, 17.1.34).-Treatment at temp.  $< -70^{\circ}/5-8$  atm., obtained by the use of liquid air, destroys micro-organisms in perishable foodstuffs. If such products are then stored at  $< 0^{\circ}$  (-7° to -10°), the valuable nutritional properties are retained. E. B. H

Preservation of meat, pork, fish, or the like. K. A. JOHANSSON and J. A. WALLSTRÖM (B.P. 431,994, 27.11.34).-Meat or fish, packed in cases with salt, is placed in a cylindrical metallic vessel and submitted first to the pressure of compressed air, which forces the salt into the flesh, and then to evacuation, which withdraws residual liquids and blood. The processes may be applied in the reverse order, and the cases may be heated. E. B. H.

Preparation and packing of prunes. C. B. PAPE, Assr. to California Prune & Apricot Growers' Assoc. (U.S.P. 1,976,961, 16.10.34. Appl., 22,8.32).—Dried prunes are washed with hot  $H_2O$ , treated with boiling  $H_2O$ , and packed while still hot ( $<74^\circ$ ). E. B. H.

Preparation of products containing pectin. R. C. MITHOFF, Assr. to Standard Oil Co. of California (U.S.P. 1,976,741, 16.10.34. Appl., 16.1.32).-Cellulosic pectin-bearing materials (pomace etc.) yield highergrade and purer pectin if previously extracted for 2 hr. at 60-90° with a 30% solution of BuyOH, which removes colour, flavour, and odour and renders pectin more easily extractable. E. B. H.

Clarifying and preserving [fruit juices and alcoholic] liquids containing colloids. KATADYN A.-G. (B.P. 431,872, 19.1.34. Ger., 19.1.33).-Since colloids impair the bactericidal action of Ag and Cu by forming flocculent ppts. which fix a large portion of the active metal, enough of the latter is added to provide a surplus sufficient to sterilise and preserve the liquid. The active metal may be recovered from the pptd. colloids. C. J.

Converting starch.-See XVII.

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

Commercial balsam of Peru: its assay. P. BEAUGEARD (Bull. Sci. pharmacol., 1934, 41, 209-219; Chem. Zentr., 1935, i, 1270).-The light-petroleum and CCl<sub>2</sub>·CHO, H<sub>2</sub>O tests are not of themselves sufficient proof of the genuineness of balsam of Peru, nor is determination of the cinnamein (I) content, since synthetic (I) can be added to artificial balsam up to the required val.; the I and sap. vals. are therefore not significant by themselves. (I) is determined by extracting an alkaline solution of the balsam with Et<sub>2</sub>O, repeatedly shaking the Et<sub>2</sub>O with H<sub>2</sub>O containing phenolphthalein, removal of Et<sub>2</sub>O, drying, and weighing. R. N. C.

Analysis of a genuine Salvador balsam (balsam of Peru). M. M. JANOT (Bull. Sci. pharmacol., 1934, 41, 219-224; Chem. Zentr., 1935, i, 1270).-The cinnamein content of a genuine balsam is 52-54%, the sap. val. 243-246, and the I val. 35.8-36.7.

R. N. C.

Toxicity and fluorescence of vetch seeds. P. W. DANCKWORTT (Z. Unters. Lebensm., 1935, 69, 458-463). -Cyanogenetic glucosides were present in Vicia sativa, angustifolia, and villosa, but were absent from V. Ervilia, tetrasperma, pannonica, hirsuta, Cracca, dumetorum, Faba, and three specimens of Villosa. The ultra-violet fluorescence varied in colour, and was not correlated with toxicity. E. C. S.

Drug extraction. II. Effect of fineness of powder and of variation in solvents on the percolation of belladonna root. W. J. HUSA and C. L. HUYCK (J. Amer. Pharm. Assoc., 1935, 24, 446-450; cf. B., 1935, 252).-Measurement of the rate and degree of swelling of belladonna root with EtOH-H2O, glycerol-H2O and -EtOH mixtures and percolation tests with EtOH-H<sub>2</sub>O menstrua indicate that, within the limits of no. 20 and no. 80 powder, the fineness is of minor importance and that the best percolation menstrua are EtOH-H<sub>2</sub>O mixtures ranging from 5:1 to 1:1 (vol./vol.). E. H. S.

Cobalt nitrate as a reagent for pharmaceutical purine preparations. R. KLIMEK (Wiadom. farm., 1934, 61, 619-620; Chem. Zentr., 1935, i, 1270).-Salts of the common purine bases give characteristic

colours with 3% Co(NO<sub>3</sub>)<sub>2</sub>, which change to equally definite colours on addition of 15% NaOH. R. N. C.

Characteristics of tinctures produced by six different methods. S. VON BARI (Pharm. Ztg., 1935, 80, 852—856, 880—882).—Ash, H<sub>2</sub>O, alkaloid contents, and d (0·89—0·91) of the tinctures made by maceration (I), double maceration (II), digestion (III), hot digestion for 3 hr. (IV), percolation (V), and diacolation (VI), and the dry residues and ash contents of the tinctures of 24 drugs and mixtures are given. There are large differences in the dry residues, tinctures prepared by (II) having the lowest vals., followed by (I) and (III). (IV), (V), and (VI) all give higher but variable figures. The (III) process gives the tinctures with the lowest ash, and (V) and (VI) the highest. The % active principles and extraction from the drug, EtOH content and EtOH nos., and appearance of the tinctures are given.

### E. H. S.

Factors necessary for the stability of ergot preparations. M. BERNERÓWNA (Wiadom, farm., 1934, 61, 169—173; Chem. Zentr., 1935, i, 1269).—Liquid preps. of *Secale connutum* gradually lose their activity as a result of decomp. of the ergot alkaloids. These can be fractionally pptd. and stored as salts at different  $p_{\rm H}$  vals. The type and concn. of the acid used are involved in the stabilisation of the alkaloids. H<sub>3</sub>PO<sub>4</sub> is the only mineral acid that prevents their decomp., the most favourable reaction being  $p_{\rm H}$  2·99. The alkaloids are quite stable in tartaric and citric acids and H<sub>2</sub>C<sub>2</sub>O<sub>4</sub> at  $p_{\rm H}$  3–4, or mixtures of tartaric acid and H<sub>3</sub>PO<sub>4</sub> at  $p_{\rm H}$  3·58, no activity being lost in 9—12 months. R. N. C.

(A) Deterioration of digitalis. (B) Assay of digitalis. B. NUKI, M. TAMAKI, and T. MATSUO (Japan. J. Med. Sci., IV, 1934, 8, 145—146, 146—147).—(A) Rates of deterioration of 10% digitalis infusion, 0.1% aq. ouabain solution, and of digitamin are compared at 30° and at refrigerator temp.

(B) Comparison is made of various biological methods. CH. ABS. (p)

Colorimetric micro-determination of morphine in opium and preparations. J. A. SANCHEZ (Semana Med., 1935, I, 191—195).—The reagent contains Na<sub>2</sub>CO<sub>3</sub> 140 g., Na<sub>2</sub>HPO<sub>4</sub> 20 g., molybdic acid 70 g., in 500 c.c. of H<sub>2</sub>O to which are added 200 c.c. of HNO<sub>3</sub> and H<sub>2</sub>O to make 1 litre. To 10 c.c. of morphine (I) solution are added 1 drop of reagent and 1 drop of HNO<sub>3</sub> and, after 10 min., 20 drops of aq. NH<sub>3</sub>. The blue colour  $\infty$ the (I) present. (I) hydrochloride syrup may be used directly for examination. Opium preps. are extracted with Ca(OH)<sub>2</sub>, acidified, treated with excess of aq. NH<sub>3</sub>, and extracted with CHCl<sub>3</sub>-Pr<sup>g</sup>OH (4:1); after evaporation of the solvent the residue is dissolved in HCl and treated as above. CH. ABS. (p)

**Determination of diaminoacridine in euflavine.** F. REIMERS (Quart. J. Pharm., 1935, **8**, 218-230).— The accuracy of the methods of Gailliot (B., 1934, 555) and of Hall and Powell (B., 1935, 253) is questioned. Diaminoacridine hydrochloride may be determined by electrometric titration with NaOH in presence of Pr<sup>8</sup>OH, using a glass electrode with thymol-blue as indicator.

H. G. R.

Chemical and physical differences between strong and mild tobacco. P. KOENIG (Z. Unters. Lebensm., 1935, 70, 26—33).—The apparent strength of good-quality tobacco can vary widely with the manner of smoking. A required degree of mildness is best obtained by blending with naturally nicotine-free tobacco. E. C. S.

Testing galenical products by microsublimation. A. JURKOWSKI and L. SINIECKI (Bull. Soc. Amis Sci. Poznań, 1934, B 6, 14–23; Chem. Zentr., 1935, i, 1091).—Application of the method to a no. of preps. is described. A. G. P.

Evaluation of native plants. I. Peppermint leaves. K. H. BAUER [with S. LIMBACH and G. KÄPPLER] (Pharm. Zentr., 1935, 76, 501—504).—Micro-methods for the distillation (Clavenger's apparatus, B., 1928, 465) of the plant and the determination of menthol and its esters are discussed and the results of their application to German cultivated (Mitcham) and native peppermint plants are given. E. H. S.

Brazilian "cascarillas." F. W. FREISE (Pharm. Zentr., 1935, 76, 469-470).-Botanical and chemical characteristics of the bark of two plants similar to true cascarilla (Croton eluteria, L., Benn.) are described. Bark from C. echiniocarpus, Müll., Arg., contains essential oil 0.9 - 2.35%  $[d_4^{20} \ 0.915 - 0.932, \alpha_D \ -6^{\circ} \ 30'$  to -(?) 7° 10′, free acids 4%, isoeugenol  $\gg 0.8\%$ , thymol 11.2%, a terpene, b.p. 152-155°, 7.5%, two sesquiterpenes about 45%, a sesquiterpene alcohol, b.p. 295°, resin 0.75%], resins 11-13.5%, tannins 13.5-15.8%, mineral matter 4-6%, and at least two bitter substances 6-6.5%. The resin contains an acid and a neutral resin both of which are strong anthelmintics. 15% of bitter substance is possibly alkaloid; the remainder is glucosidic. The bark from C. floribundos, Spreng., contains essential oil > 4.25 %, tannins > 19%, bitter substance  $\gg 9\%$ , and resins 5-6%. E. H. S.

Japanese lemongrass oil. I. HEROLD (Deuts. Parfüm.-Ztg., 1934, 20, 339—340; Chem. Zentr., 1935, i, 1136).—Japanese lemongrass oil differs considerably from East Indian. It has  $d^{15} 0.916$ ,  $\alpha_{\rm D} + 0.88^{\circ}$ ,  $n_{20}^{20}$ 1.48452. It contains 60—70% of citral, with some terpineol and borneol, but scarcely any methylheptenone, terpenes, or sesquiterpenes. Three fractions obtained by vac. distillation are described. R. N. C.

Cod-liver oil.-See XII.

### PATENTS.

[Manufacture of germicidal] dressings, bandages, and the like. DEUTS. GOLD- & SILBER-SCHEIDEANSTALT VORM. ROESSLER (B.P. 431,656, 2.3.34. Ger., 7.11.33).— Cotton wool, bandages, etc. are impregnated with Ag and Mn oxides, preferably with  $Ag_2O_2MnO_2$ ; e.g., the material is soaked in aq.  $NH_3$ -AgNO<sub>3</sub>, then treated with aq. quinol, and finally, after washing, with aq. alkaline KMnO<sub>4</sub>. E. H. S.

Esters of 2-carboxy-5-aminodiphenyl. [Anæsthetics.] W. S. JONES and W. BRAKER, ASSTS. to E. R. SQUIBE & SONS (U.S.P. 1,976,940, 16.10.34. Appl., 12.9.32).—5-Amino-2-carboxydiphenyl (I) is prepared by the following stages: 5-nitro-2-amino-,  $\rightarrow$  5-nitro-2cyano-, m.p. 131—133°,  $\rightarrow$  5-nitro-2-carboxy-, which is then reduced. (I), Na salt, with the appropriate dialkylaminoalkyl chloride, yields, e.g., 5-amino-2-carbo- $\beta$ diethylaminoethoxy- and 5-amino-2-carbo- $\gamma$ -di-n-butylaminopropoxy-diphenyl. A. W. B.

Dialkylaminoalky lesters of (A) dialkylaminoalkoxy-3-carboxy-, (B) hydroxy-3-carboxy-, (C) alkoxycarboxy-diphenyls. [Anæsthetics.] W. G. CHRISTIANSEN and (A) W. BRAKER, (B) (C) A. W. HARVEY, Assrs. to E. R. SQUIBB & SONS (U.S.P. 1,976,921-2 and 1.976.924, 16.10.34. Appl., [A] 1.6.32, [B] 9.6.32, [c] 9.12.32).-(A) The Na2 salt of a hydroxy-3-carboxydiphenyl (I) is treated with a dialkylaminoalkyl halide (chloride). Examples include : 4- \Beta-diethylaminoethoxy-3-carbo-B-diethylaminoethoxy- and 4-y-di-n-butylaminopropoxy-3-carbo-y-di-n-butylaminopropoxy-diphenyl. (B) The Na salt of (I) is treated with an alkylene dibromide and the  $\omega$ -Br-ester treated with a sec.-amine. E.g., 4hydroxy-3- $\beta$ -bromo-  $\rightarrow$  4-hydroxy-3-carbo- $\beta$ -diethylaminoethoxy-diphenyl hydrochloride, m.p. 16716-8.5° (decomp.). (c) The Na, salt of a hydroxycarboxydiphenyl is etherified with, e.g., Et<sub>2</sub>SO<sub>4</sub>, BuBr, the ether-ester is hydrolysed to ether-acid, and this is treated as in (B). Examples are: 4-ethoxy-3-carbethoxy- $\rightarrow$  4-ethoxy-3-carboxy-(II)  $\rightarrow$ 4-ethoxy-3-carbo- $\beta$ -bromoethoxy-  $\rightarrow$  4-ethoxy-3-carbo- $\beta$ diethylaminoethoxy-, b.p. 195-205°/<1 mm.; (II)  $\rightarrow$  4ethoxy-3-carbo- $\gamma$ -bromopropoxy-  $\rightarrow$  4-ethoxy-3-carbo- $\gamma$ di-n-butylaminopropoxy-, b.p. 255-265°/1 mm.; 4-nbutoxy-3-carbo-n-butoxy-  $\rightarrow$  4-n-butoxy-3-carboxy-, m.p.  $83 \cdot 5 - 84 \cdot 5^{\circ}$ ,  $\rightarrow 4$ -n-butoxy-3-carbo- $\beta$ -bromoethoxy-,  $\rightarrow$ 4-n-butoxy-3-carbo-β-diethylaminoethoxy-, b.p. 210-220°/ 1 mm.; 2-ethoxy-3-carbethoxy-  $\rightarrow$  2-ethoxy-3-carboxy-, m.p. 97–98°,  $\rightarrow$  2-ethoxy-3-carbo- $\beta$ -bromoethoxy-  $\rightarrow$  2ethoxy-3-carbo-B-diethylaminoethoxy-diphenyl, b.p. 180-190°/1-1.5 mm. The products may be used in aq. solution as salts. A. W. B.

Aminoalkyl esters of the carboxyalkoxyaminodiphenyls. [Anæsthetics.] W. G. CHRISTIANSEN and A. W. HARVEY, ASSTS. to E. R. SQUIBB & SONS (U.S.P. 1,976,923, 16.10.34. Appl., 29.10.32).—The claims cover dialkylaminoalkyl esters of 4'-amino-3-carboxy-4-alkoxydiphenyls and their salts, and their application as anæsthetics. The following are the stages of the only detailed example : 4-ethoxy-3-carboxy- (see preceding abstract)  $\rightarrow$  (HNO<sub>3</sub>) 4'-nitro-4-ethoxy-3-carboxy-,  $\rightarrow$  4'nitro-4-ethoxy-3-carbo- $\beta$ -bromoethoxy-,  $\rightarrow$  4'-nitro-4ethoxy-3-carbo- $\beta$ -diethylaminoethoxy-,  $\rightarrow$  4'-amino-4ethoxy-3-carbo- $\beta$ -diethylaminoethoxy-diphenyl. A. W. B.

Production of pregnanolones [from pregnandiol]. SCHERING-KAHLBAUM A.-G. (B.P. 431,833, 24.12.34. Ger., 23.12.33 and 28.3.34).—A monoester (acetate, m.p. 170.5°) of pregnandiol (I), produced by partial esterification (Ac<sub>2</sub>O in C<sub>5</sub>H<sub>5</sub>N at room temp.) of (I) or cold alkaline hydrolysis (0.008N-KOH in MeOH) of the diester, is oxidised (CrO<sub>3</sub>-AcOH), and the pregnanolone ester (acetate, m.p. 142—142.5°) produced is hydrolysed. Alternatively, pregnandione is subjected to partial reduction (H<sub>2</sub>-Pt). Pregnanolone has m.p. 152° (semicarbazone, m.p. 203—204°). H. A. P.

Manufacture of adenosinephosphoric acid. P. E. OSTERN, ASST. to CHEM. & PHARM. FABR. DR. G. HENNING (U.S.P. 1,977,525, 16.10.34. Appl., 2.8.33. Ger., 2.8.32).—An acidified aq. extract of blood, muscle, etc. is made faintly alkaline and then re-acidified to ppt. albumin, inorg. phosphates are removed by addition of an alkaline-earth hydroxide,  $Pb(OAc)_2$  is added, and the ppt. is decomposed with H<sub>2</sub>S. The solution is neutralised with alkaline-earth hydroxide and the salts of inosic and adenosinephosphoric acids are separated by fractional crystallisation. E. H. S.

Reducing the nicotine content of tobacco. H. J. C. FORRESTER. From HALL TOBACCO CHEM. Co. (B.P. 431,596, 6.4.34).—Loose or packaged tobacco, containing  $\bigstar 8\%$  H<sub>2</sub>O, is treated with either gaseous or aq. alkylene oxides, *e.g.*, (CH<sub>2</sub>)<sub>2</sub>O. CO<sub>2</sub> may be used as diluent for the gas. E. H. S.

Separation of anethole from pine oil. I. W. HUMPHREY, Assr. to HERCULES POWDER Co. (U.S.P. 1,977,064, 16.10.34. Appl., 11.3.30).—Pine oil is heated with HI, e.g., with 0.4% of 50% HI at 190—225° for 5 hr., and the hydrocarbons formed (b.p. 170—185°) are distilled off and the anethole is separated from the residue by fractionation. E. H. S.

Manufacture of compounds [ureides] of therapeutic value. W. W. TRIGGS. From ABBOTT LABS. (B.P. 432,323, 1.8.34).—See U.S.P. 1,969,828; B., 1935, 749.

Reagents for carbonyl group. Phosphates of glycine alkyl esters. Diaryl sulphide derivatives. —See III.

# XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Silver iodide and sensitivity. T. S. PRICE (Phot. J., 1935, 75, 447—449).—The rôle of AgI in increasing the sensitivity of AgBr photographic emulsions is discussed. The incorporation of AgI in the AgBr lattice will set up strain and facilitate decomp. Reactions between solids are discussed; since AgI in emulsions is in a cubic lattice, it will be in a metastable and reactive state. It is probable that the AgI is the photosensitive substance. Alternatively, AgBr on decomp. would give free Br which, reacting with AgI (preferably to gelatin), would produce I, which would then be adsorbed on AgI. This acceptor action would increase the strain of the lattice. J. L.

Influence of cystine on supersensitive photographic emulsions. A. CHARRIOU and (MLLE.) S. VALETTE (Bull. Soc. Franç. Phot., 1935, 22, 136—139).— In the prep. of highly sensitive emulsions (about 1800 H. and D.), addition of very small amounts (0.0001 mol. per mol. of AgBr) of cystine has no effect; with increasing amounts, considerable desensitisation and reduction of fog are produced. Simultaneous addition of "hyposulphite" and/or ovalbumin causes greater desensitisation with, however, intense fogging. J. L.

Reduction of positives of mosaic tricolour [photographic] films. F. LAPEYRE (Bull. Soc. Franç. Phot., 1935, 22, 133–136).—Of various reducers tested, only that comprising a solution of  $K_2Cr_2O_7$  with  $H_2SO_4$  was found satisfactory, giving equal reduction in highlights and shadows. Various practical recommendations for development and reduction are given. J. L.

Fine-grain development with *p*-phenylenediamine. K. JACOBSOHN (Phot. Ind., 1934, 32, 1242—1244; Chem. Zentr., 1935, i, 1326).—The fine-grain developing properties of p-C<sub>6</sub>H<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub> are attributed to its low developing energy. H. J. E.

Influence of the thioacetyl derivatives of aromatic amines on [photographic] development. F. KALLAB and F. SPINNLER (Phot. Korr., 1934, 70, 182—184; Chem. Zentr., 1935, i, 1326).—Thioacet-anilide (0.05) or -naphthalide (0.04 g./litre) added to a metol-quinol developer reduced fog considerably, without changing the gradation. H. J. E.

Photography in wool research.-See V.

### PATENTS.

Manufacture of photographic bleaching-out layers. I. G. FARBENIND. A.-G. (B.P. 426,346, 2.10.33. Ger., 30.9.32).—The layer comprises a binding agent, sensitiser, acid, and a xantho-oxonium salt carrying a substituted or unsubstituted  $3-NH_2$ , and in position 6 OH or substituted OH, *e.g.*, the dye from *m*-acetamidophenol and CPhCl<sub>3</sub> or MeCHO. C. H.

Photographic bleaching-out layers. A. POLGÁR and C. HALMOS (B.P. 426,383, 9.8.34).—Dyes are selected by plotting bleaching-out against concn. of sensitiser, and using sensitiser at a concn. corresponding with the point of approach of the curves; substances (FeCl<sub>3</sub> or diazo compounds) giving blacks on development of unexposed portions are added. For process work the dyes may be very dil., producing pale shades bounded by black lines. *E.g.*, Safranin-GG and Flavindulin are used in conjunction with 10-bromo-1:1':3:3:3':3'hexamethylindocarbocyanine bromide. C. H.

Modification of the sensitivity of photographic emulsions. J. D. KENDALL (B.P. 431,142, 23.12.33).— The products of B.P. 431,187 (B., 1935, 842) are used as sensitisers or desensitisers. H. A. P.

Methine or polymethine compounds. Photosensitising dyes.—See IV.

### XXII.—EXPLOSIVES ; MATCHES.

Causes of explosions of solids. G. J. O. ZONNEN-BERG (Chem. Weekblad, 1934, 31, 722-726).—The tendency to explosion of dextrin is greater at 170° than at room temp., but spontaneous ignition does not occur below 540°;] on prolonged heating at 170° the reactivity passes through a max. An apparatus and method are described for studying the initiation of dust explosions by sparks resulting from a static charge. H. F. G.

Initiation of explosion in detonating explosives. A. J. DER WEDUWEN (Chem. Weekblad, 1934, 31, 699-700).—Methods of expressing the efficiency of a detonant are described, with particular reference to the work of Wöhler and Martin on the min. quantity of various fulminates and azides required to initiate the explosion of NO<sub>2</sub>-compounds. The merits of Hg fulminate, together with the influence of added PbNO<sub>3</sub> on the crit. velocity, are described. H. F. G.

Determination of diphenylamine in smokeless powders. S. G. Cook (Ind. Eng. Chem. [Anal.], 1935, 7, 250-255).—The NHPh<sub>2</sub> content of smokeless powders can be obtained by direct analysis using any of the following methods : gravimetric determination of (a) products of nitration (AcOH-HNO<sub>3</sub>), (b) products of bromination ( $\text{Et}_2\text{O}$ -Br); (c) extraction with  $\text{Et}_2\text{O}$  and determination as in (b); (d) as in (c) but using a volumetric method to determine % bromination. Special applications of the methods are indicated. F. N. W.

Determination of the rate of combustion of gunpowder with the aid of Thring's indicator. J. D. VAN ROON (Chem. Weekblad, 1934, 31, 732-733).— The Thring indicator for recording photographically the pressure changes during the combustion is described briefly; the instrument is but little sensitive to temp., the actual displacement is small, and the natural frequency is of the order of 10,000 per sec. H. F. G.

Determination of the combustion temperature of smokeless powder. P. F. M. DE PAUW (Chem. Weekblad, 1934, 31, 717—718).—Recent work on the determination of the temp. of combustion, and on the influence of admixed materials such as stabilisers, is noted. The heat of combustion of a given mixture may be calc. from  $100(H - \Sigma n \beta)/(100 + n)$ , where H is the val. for the basic explosive,  $\Sigma n$  the  $\frac{9}{6}$  admixture, and  $\beta$  a coeff. which is const. for each of the admixed substances. H. F. G.

**Prevention of "mouth flame" of ordnance.** G. DE BRUIN (Chem. Weekblad, 1934, **31**, 733—735).— Modern smokeless powders have the disadvantage that the gaseous products of combustion contain CO and  $H_2$ , which undergo secondary ignition when they mix with air after expulsion of the projectile. Methods of prevention based on (a) ensuring that combustion is complete some time before the projectile leaves the barrel, (b) using powder of comparatively low combustion temp., and (c) employing inhibitors of the secondary combustion, are discussed. H. F. G.

Explosions of coal dust, and of gases.-See II.

#### PATENTS.

**Explosive priming mixture.** WINCHESTER REFEAT-ING ARMS Co., Assees. of J. D. MCNUTT (B.P. 432,096, 15.3.35. U.S., 31.3.34).—The use of alkali and alkalineearth salts of dinitrophenyl azide is claimed. A suitable composition is the K salt 16, basic Pb salt of trinitroresorcinol 15,  $Ba(NO_3)_2$  40, and  $Sb_2S_3$  29%. W. J. W.

Blasting charge. J. TAYLOR, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 431,935-6, 15.12.33).-(A) A mixture of NaNO<sub>2</sub>, NH<sub>4</sub>Cl or (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, and 2% of alkaline material as stabiliser, which is capable of sustaining its own gas-producing decomp. when initiated, is enclosed in a rigid container together with means of initiation, e.g., blackpowder, the container being provided with a bursting disc. (B) The charge comprises a liquid medium capable, on being heated, of undergoing gas-evolving exothermic decomp. without explosion at ordinary pressure, e.g., an aq. solution of NH<sub>4</sub>NO<sub>3</sub> or NH<sub>4</sub>NO<sub>2</sub>, or of an alkali nitrate or nitrite, with or without other NH<sub>4</sub> salts and/or urea. The container consists of two parts, one containing the means of initiation and the other, vented to the atm., the charge. W. J. W.

Composition for fuse, igniter charges, and the like. H. E. NASH, Assr. to HERCULES POWDER Co.

(U.S.P. 1,960,591, 29.5.34. Appl., 22.3.32).—Mixtures of BaO<sub>2</sub> or  $Pb_3O_4$  with S, Se, or Te are claimed. A. R. P.

### XXIII.—SANITATION; WATER PURIFICATION.

Determination of very small amounts of carbon monoxide in air. II. L. W. WINKLER (Z. anal. Chem., 1935, 102, 99–101; cf. B., 1935, 526).—By employing 0.004N-KBrO<sub>3</sub> and 0.004N-NaAsO<sub>2</sub> and a 5-c.c. micro-burette the method described previously can be applied to the determination of 10–100 p.p.m. of CO in 250 c.c. of air. The test for CO using dil. aq. PdCl<sub>2</sub> is rendered more delicate by leaving the PdCl<sub>2</sub> in contact with the gas for 1 day and then adding a little (NH<sub>4</sub>)<sub>2</sub>MoO<sub>4</sub> solution, acidified by HCl. J. W. S.

Phenomena associated with the rôle of nitrogen in biological oxidation. F. R. O'SHAUGHNESSY and С. Н. НЕWITT (J.S.C.I., 1935, 54, 167-168 т).--In the absence of dissolved O<sub>2</sub> the avidity for O<sub>2</sub> of aërobic sludges from actively nitrifying activated-sludge plants or bacterial filters is such that nitrites present are rapidly decomposed with liberation of gaseous N2. In practice, the gas buoys up the particles of sludge in the vessel in which they are intended to settle and thus fouls an otherwise satisfactory effluent. The reaction is accompanied by a rise in  $p_{\rm H}$  val. and an increase in alkalinity, but it cannot be controlled by further artificial adjustments, e.g., with Ca(OH)2. The denitrifying action does not occur in the presence of non-nitrifying aërobic sludges and the N2 found in small amounts in the gases evolved during the anaërobic fermentation of sludge is considered to have been dissolved originally in the liquor. C. J.

Aëration of water by air diffusion. F. C. Roe (J. Amer. Water Works Assoc., 1935, 27, 897–904).— Small amounts of air (0.005-0.16 cu. ft. per gal.), when diffused through porous materials in fine bubbles, are effective in removing odours, CO<sub>2</sub>, and Fe from H<sub>2</sub>O and at the same time produce agitation for the mixing of coagulants without disintegrating the floc. The method is simple and flexible, and installation and operation costs are comparatively low. C. J.

Dilution and carbon treatment make salty water palatable. E. W. STEEL (Water Works Eng., 1934, 88, 138—140).— $H_2O$  containing 2600 p.p.m. of total solids is diluted with other  $H_2O$  having 550 p.p.m. of total solids and treated with activated C. CH. ABS. (p)

Removal of phenol from surface waters. A. LAGER-GREEN (Tekn. Tidskr., 1933, 63, 217).—Removal of approx. 75% of the PhOH results from treatment with  $Al_2(SO_4)_3$  and filtration through norit. CH. ABS. (p)

Control of the lime-soda water-softening process by a nomograph. F. W. STAFFELDT (Gas- u. Wasserfach, 1935, 78, 623).—Scales giving total hardness and phenolphthalein alkalinity are placed together vertically above the axis 0.0 on the left and a scale representing the Me-orange alkalinity vertically downwards at the right end of the axis. The intervening space is divided into four fields, the field in which the line joining the two points given by the titration cuts the axis indicating the correctness or otherwise of the softening process. C. I.

Water mains. IV. Corrosion : causes and prevention. F. J. TAYLOR (Contractors Rec., 1933, 467).--- The formation of incrustations begins with the appearance of small tubercles covering pits in metal which subsequently softens to a spongy condition. Treatment with CaO leads to a protective coating of  $CaCO_3$ 

CH. ABS. (p)New methods of water analysis. III. H. F. KUISEL (Helv. Chim. Acta, 1935, 18, 896—907; cf. B., 1935, 432).—K' is determined by pptn. with Na<sub>4</sub>Co(NO<sub>2</sub>)<sub>6</sub>, dissolving the ppt. in H<sub>2</sub>SO<sub>4</sub>, and titrating with KMnO<sub>4</sub>. The error is  $\pm 1.5\%$ . Fe<sup>---</sup> is determined colorimetrically as Fe(CNS)<sub>3</sub> after extraction by EtOAc. The error is about  $\pm 1\%$ . Mg<sup>--</sup> can be determined to  $\pm 1.5\%$  by pptn. with 8-hydroxyquinoline and titrating with 0.1N-KBrO<sub>3</sub>. E. S. H.

Rapid volumetric determination of sulphate in natural water and boiler blowdown using potassium palmitate. P. HAMER (J.S.C.I., 1935, 54, 250– 253 T; cf. Blacher, B., 1913, 158).—K palmitate may be used for the determination of  $SO_4^{\prime\prime}$  in H<sub>2</sub>O with results in good agreement with gravimetric determinations of S in natural waters. The agreement in the case of boiler blowdown is not as good as with natural H<sub>2</sub>O, but is sufficiently good for control of boiler feed-H<sub>2</sub>O conditioning and softening.

Turbidimetric determination of sulphate in water. Betz-Hellige method. R. T. SHEEN, H. L. KAHLER, and E. M. Ross (Ind. Eng. Chem. [Anal.], 1935, 7, 262-265).—The turbidimeter described compares a beam of light with the Tyndall effect produced from a lateral illumination of the specimen by the same light source. Results obtained with BaSO<sub>4</sub> suspensions compare favourably with those by the gravimetric method. E. S.H.

Creamery waters. Sterilising milk machines etc.—See XIX.

### PATENTS.

Cleaning agent. E. SCHNABEL (B.P. 432,022, 14.12.33).—A liquid or pasty solution of polyvinyl alcohol containing cosmetics, skin-preserving substances, *e.g.*, EtOH, glycerol, starchy substances, plant juices, *e.g.*, Carragheen moss (together with org. fat-solvents, if desired), is used for cleaning surfaces, particularly human skin, the cleanser in use rolling up into crumbs by which dirt is removed. S. S. W.

Sterilisation of liquids. KATADVN A.-G. (B.P. 432,101, 13.12.33. Ger., 23.2.33).—The liquid is treated with  $H_2O_2$  or other non-gaseous  $O_2$ -yielding substances other than halogen compounds and also with minute quantities of an oligodynamically active metal in such manner that the different reactions can be separately controlled. The active metals, in addition to their own sterilising action, also act as catalysts and by aiding the liberation of  $O_2$  accelerate the germicidal action of the  $H_2O_2$  (etc.).

Clarification of chlorinated solutions [sewage]. C. N. WINDECKER (U.S.P. 1,980,986, 13.11.34. Appl., 7.7.32).—Effluent from chlorinated sewage is treated with CaO to neutralise acid and produce a flocculent ppt. B. M. V.

Substituted phenols.—See III. Insecticides.—See VI and XVI.