

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

DEC. 22 and 29, 1933.\*

### I.—GENERAL; PLANT; MACHINERY.

**Heat transmission in rotary kilns.** V. The drying zone. W. GILBERT (Cement, 1933, 6, 327—338; cf. B., 1933, 847).—The length of a kiln has been divided to correspond with the stages of the process and the heat balance was rearranged to show the heat which would be required in each stage. From these figures the length of kiln in the various stages has been calc. C. A. K.

**The heat-transfer problem.** W. B. HAWES (Trans. Inst. Chem. Eng., 1933, 10, 161—167).—The temp.-velocity distribution curves are given for H<sub>2</sub>O flowing through a coiled pipe which formed part of an a.-c. circuit heating the H<sub>2</sub>O. For Reynolds nos. of 30—6000 (approx.) the flow follows two helices symmetrically disposed with regard to the diam. at right angles to the axis of the coil. The film coeff. of heat transfer on the outside of the curve is > that on the inside. For coiled pipes it is > for straight pipes at the same Reynolds no. D. K. M.

**Melting process of chemically homogeneous masses.** E. HERLINGER and P. BECK (Sprechaal Keram., 1933, 66, 161—162; Chem. Zentr., 1933, i, 3483).

**Comparison of methanol and other anti-freeze agents.** T. C. ALBIN (Chem. & Met. Eng., 1933, 40, 526—527).—A specification of anti-freeze MeOH is proposed. The f.p., b.p., and composition of the vapour at the b.p. for aq. EtOH, aq. MeOH, and aq. COMe<sub>2</sub>, and the f.p. of aq. MeOAc and of aq. MeOH and COMe<sub>2</sub> are given. D. K. M.

**Fractional degassing of hot and cold waters in the Claude-Boucherot process.** P. CHAMBADAL (Compt. rend., 1933, 197, 825—827; cf. B., 1933, 1).—If  $n$  be the no. of preliminary degasifiers used to remove the dissolved gases the % available power used in the process is  $0.0119 + [0.024/(0.444 + n)]$ , showing that it is advantageous to use  $n = 2$ , and but little so when  $n$  is > 2. C. A. S.

**[Fractional degassing of hot and cold waters in the Claude-Boucherot process.]** G. CLAUDE (Compt. rend., 1933, 197, 827; cf. preceding abstract).—The use of preliminary degasifiers also reduces the amount of H<sub>2</sub>O vapour carried away by the gases removed at a pressure of about 0.01 atm. C. A. S.

**Apparatus for controlling the sugar content of boiler feed-water.** H. WITTE (Deut. Zuckerind., 1933, 58, 406—407).—A 10% solution of  $\alpha$ -C<sub>10</sub>H<sub>7</sub>·OH and H<sub>2</sub>SO<sub>4</sub> drops continuously into a reaction vessel which empties at the end of a predetermined interval by siphon action, the formation of a violet colour being

indicated by a light-sensitive cell, so as to cause the operation of a signalling device. J. P. O.

**Sedimentation of fine particles in liquids.** R. F. STEWART and E. J. ROBERTS (Inst. Chem. Eng., Oct., 1933. Advance proof, 14 pp.).—Amongst factors modifying the behaviour of a falling particle is the presence of other particles > a certain concn. In a dispersed pulp there may be a different effective viscosity for every size of particle. Flocculent pulps which behave as plastic solids exhibit very complicated behaviour. The effective  $d$  of the medium is also modified by the presence of many particles finer than that considered, and entrainment is also important. Classification is best carried out on a dispersed pulp and depends on opposing sedimentation by an upward stream of liquid (hydraulic classifiers), or by its restriction by the use of a settling tank and overflow (non-hydraulic classifiers). The latter type may or may not have mechanical agitation. Designs and applications of each type are considered. Thickening or clarification is an operation calling for a flocculent sediment, which can often be improved by modification of agitation, of  $p_H$ , etc. Settling occurs in 2 zones, viz., (A) those in which free settling occurs and (B) those where compression of the flocs is conceived to occur, the dilution at the change being a crit. point. Capacity in the zone A is governed by area, but in zone B it is affected also by depth. Slow stirring of a pulp in compression assists settling. Pulps too dil. to show slime settling depend on the settling rate of the slowest particles, and pretreatment is important if a clear overflow is required. C. I.

**Effect of tube diameter in cyclonic dust collectors.** E. ANDERSON (Chem. Met. Eng., 1933, 40, 525—526).—The efficiency of the removal of cement dust and fuller's earth from gases is plotted against draught loss for different tube diam. The efficiency increases with increased draught loss and is greatest with tubes of smallest diam. D. K. M.

**Construction and proof of nomogram for reduction of gas volumes to standard temperature and pressure.** D. G. BAGG (Trans. Inst. Chem. Eng., 1933, 10, 172—173).—Mathematical. D. K. M.

**Speedy methods of determining moisture.** S. STANWORTH (Trans. Ceram. Soc., 1933, 32, 443—454).—Rapid (e.g., 2—4 min.) determinations of the H<sub>2</sub>O content of ceramic and other materials are obtained by means of the "Speedy Moisture Tester" (described), in which a given wt. of the wet substance is mixed with a CaC<sub>2</sub> compound, the resulting rise of pressure, due to evolved C<sub>2</sub>H<sub>2</sub>, being measured on a gauge calibrated in % H<sub>2</sub>O.

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



H<sub>2</sub>O determinations by this and by the oven-drying method on china clay, soil, brick shale, and fireclay showed good agreement. Methods of moisture determination in coal and in flour are discussed. A. L. R.

**Balance-plastometer; simple apparatus for measurement of plasticity and recovery of soft materials [including rubber].** J. HOEKSTRA (Kautschuk, 1933, 9, 150—152).—The apparatus consists essentially of a balance with unequal arms to the longer of which is attached a round rod (which can be loaded) resting on the test-piece. Plasticity is measured as in van Rossem's modification of the Williams plastometer. The construction ensures sensitiveness and accuracy. D. F. T.

**Gloss and its measurement.** A. KLUGHARDT (Papier-Fabr., 1933, 31, 521—524).—The total amount of light reflected from a real surface does not consist of a simple mixture of reflected and diffused light owing to modifications due to diffraction at the smallest particles and polarisation of the regular reflected light, making impossible an exact theoretical interpretation of gloss. Methods are described of obtaining the gloss of a surface by measuring photometrically the amount of light of standard spectral composition reflected from it at varying angles of incidence from 0° to 22.5° and comparing with a standard BaO surface. An attempt is made to differentiate between gloss and optical smoothness. D. A. C.

**Fineness of powdered coal (etc.).**—See II. Kilns.—See IX. Alloys for furnaces.—See X. Evaporation of viscous solutions. Viscosity coeffs. of viscous liquids.—See XVII.

See also A., Nov., 1115, Rate of settling of suspensions. 1134, Thermo-regulators. Tyndallmetric examination of filtered liquors. 1135, Measurement of viscosity.

## PATENTS.

**Vapour condenser.** O. A. LINDEMANN, Assr. to SCHUTTE & KOERTING Co. (U.S.P. 1,894,696, 17.1.33. Appl., 11.11.30).—In a condenser suitable for removing vapours from permanent gas, the spraying Venturi is of the same angle as the larger Venturi for the mixture of liquid and gases, and is so situated that the spray from the one completely fills the cone of the other. B. M. V.

**Gas conditioner.** C. G. HAWLEY, Assr. to CENTRIFIX CORP. (U.S.P. 1,894,744, 17.1.33. Appl., 6.12.27).—A form of apparatus in which a gas is passed in turn through liquid sprays, a moisture eliminator, and, if desired, a reheater is described. B. M. V.

**Fractionation of hydrocarbon oils.**—See II.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Degradation of coal.** F. HOPKINSON (J. Inst. Fuel, 1933, 7, 41—43).—A method is suggested for calculating an index figure representing the "overall" or the "unit" degradation of a coal, after mechanical treatment, from the initial and final sieve analyses. The effects of size degradation are briefly discussed. A. B. M.

**Determining the dustiness of coal and coke.** A. R. POWELL and C. C. RUSSELL (Ind. Eng. Chem.

[Anal.], 1933, 5, 340—341).—A weighed quantity of fuel is dropped into a closed container and the dust is allowed to settle on a polished metal plate so that it can be removed and weighed. E. S. H.

**Laws governing the fineness of powdered coal.** P. ROSIN and E. RAMMLER (J. Inst. Fuel, 1933, 7, 29—36).—A series of grinding tests with bituminous coal in a tube mill without air separation have given the relation  $L = cR^p$  ( $p > 0$ ), where  $L$  is the throughput of the mill (tons/hr.) and  $R$  is the % residue on a test sieve, the same type of relation holding for all test sieves with mesh apertures between 60 and 150  $\mu$ . From this has been deduced the relationship  $R = 100e^{-bx^n}$ , between  $R$  and particle size  $x$ . Examination of a wide range of types of coal and other powdered materials (e.g., cement, magnesite, clay, ground glass, ores, etc.) and methods of grinding has shown this relationship to be a universal law of size distribution, valid for all powders, provided that pulverisation has been carried beyond a certain stage. The laws of distribution in the first stages of size reduction, such as are obtained in hammer mills or jaw crushers, are more complicated. For tube mills without air-sweeping, the exponent  $n$  was independent of degree of fineness for the same coal (e.g., 1.25 for Westphalian lean coal, 1.34 for Ruhr coking coal or brown coal semi-coke), whilst the parameter  $b$  increased rapidly with increasing fineness; for a triple-ring roll mill  $n$  increased with increasing fineness. By referring the results to a unit particle size, e.g., the mean particle size (cf. Heywood, B., 1933, 416), a non-dimensional representation of the fineness characteristic curve is obtained; i.e., when  $n$  is const. the curves remain identical with increasing fineness of coal. Other properties of the curves are briefly discussed. A. B. M.

**Composition of coal. Action of solvents.** J. E. ASHMORE and R. V. WHEELER (J.C.S., 1933, 1405—1408).—Anomalous figures obtained for the amount of extract obtainable from coal are due to catalytic interaction of the solvent with the constituents of the coal. Cockshead coal extracted with xylene-amyl alcohol affords an extract in spite of the fact that the coal loses no wt. Similarly, anthracite calcined at 1000° is "extracted" to the extent of 18.2%. Extraction of coal by C<sub>5</sub>H<sub>5</sub>N followed by CHCl<sub>3</sub> affords dependable results. J. L. D.

**Flotation cleaning of fine coal.** C. BERTHELOT (Chim. et Ind., 1933, 30, 770—786).—Results are given for various collieries working the "Minerals Separation" froth-flotation process for fine coal (< 2.5 mm.). The Kleinbentink process, in use in Holland and France, requires only 2.7 h.p. per ton of treated coal and employs creosote oil for froth-forming; all the coal is converted into washed fines ( $\geq 10\%$  of ash). It has been shown in America that pyrites can be separated from the coal by the addition of FeSO<sub>4</sub> or Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, by maintaining the  $p_{\text{H}}$  at 4.5—6.9, or by dilution and decantation. The apparatus used in the above two processes, together with the Elmore process of the Fuel Research Board, is described. The American Trent process employs fine-ground coal (100-mesh) and a wt. of oil 30—40% of that of the coal. The oil and cleaned coal sink as a paste which can be carbonised to a semi-



coke for household purposes. Vac. filtration will not reduce the  $H_2O$  content to  $< 21\%$  and is costly. The Elmore centrifugal machine, used in Holland, gives a product containing  $8.8\% H_2O$ . C. I.

**Action of pyridine vapour on peat.** J. DUBOIS (Przemysł Chem., 1933, 17, 223—225).—Peat (I) adsorbs small quantities of  $CHCl_3$ ,  $Et_2O$ ,  $EtOH$ ,  $C_6H_6$ ,  $CCl_4$ ,  $C_5H_{11}OH$ ,  $EtOAc$ , and  $C_5H_{11}OAc$  vapours, equilibrium being attained within 24 hr. Considerable quantities of  $C_5H_5N$  (II) vapour are absorbed by (I) ( $40\%$  of its wt. after 15 days), which in this respect behaves similarly to coal. Absorption of (II) increases with the age of (I), but appears not to be connected with the content of bituminous substances. R. T.

**Determination of sulphur in fuels.** Survey of the most important papers which have appeared since 1929. E. DITTRICH (Brennstoff-Chem., 1933, 14, 383—388).—Recent developments in the methods of determining S in solid, liquid, and gaseous fuels are briefly summarised. A full bibliography is appended. A. B. M.

**Pitchless coal briquette.** H. MATSUMAI and T. MORIMASA (J. Fuel Soc. Japan, 1933, 12, 110—111).—The finely-ground coal is heated to its softening temp., previously determined by means of the Lessing coking apparatus, and compressed in an Fe mould under a pressure of 50 kg./sq. cm. The strength of the briquette is the greater the finer is the coal and the longer the duration of compression. A. B. M.

**Combustion of brown coal high in ash.** P. ROSIN, E. RAMMLER, and F. DOERFFEL (Braunkohle, 1933, 32, 209—214; Chem. Zentr., 1933, i, 3388).—Drying and speed of ignition are not markedly influenced by ash content (I) ( $5$ — $24\%$ ).  $1\%$  increase in (I) diminished the steam-raising power by about  $2.6\%$ . Variations in amount and  $CO_2$  content of waste gaseous products are recorded. A. A. E.

**Pyrogenation of bituminous schists of the Franch-Comté Jura.** J. BARLOT (Compt. rend., 1933, 197, 850—852).—Optimum yields ( $50\%$  of the total org. matter) of oil (I) are obtained from the bituminous schists (II) of Creveney by rapid distillation at  $500^\circ$ /vac. and washing of the evolved gases.  $30$ — $40\%$  of (I) has b.p.  $< 205^\circ$ , and much has b.p.  $33$ — $90^\circ$ . Four rocks gave 130—190 litres of (I) per cu. m. The composition of (II) varies: org. matter 13—37, CaO 35—45, MgO 0.5—2,  $SiO_2$  35—40,  $Fe_2O_3 + Al_2O_3$  13—18, and FeS 1.5—3%. The val. of (II) as a source of combustibles is indicated. R. S. C.

**Recovery of benzol [from coal distillation].** H. EGELING (Chem.-Ztg., 1933, 57, 821—822).—The operation of the benzol-recovery plant in a gasworks is shown in a series of tables giving the amount absorbed per cu. m. of gas, the loss in calorific val. of the gas by the scrubbing operation, the composition of the light oil (I) recovered from the scrubber (II), and the steam consumption in expelling (I) from (II) and in distilling (I). A. R. P.

**Increase in benzol extraction in the Ruhr district.** RETTENMAIER (Gas- u. Wasserfach, 1933, 76, 791—792).—Statistics covering the last 5 years show that the % benzol extracted per ton of coal carbonised has in-

creased. This is due to milder treatment of crude benzol, more efficient carbonisation, and better methods of extraction, e.g., low-temp. cooling and adsorption by active C. R. N. B.

**Constituents of brown coal gas-benzine.** H. E. BROMME (Braunkohlenarch., 1933, 39, 1—44; Chem. Zentr., 1933, i, 3838—3839).—Basic constituents ( $1\%$ ) contained  $C_5H_5N$  and probably  $\beta$ -picoline; the creosote (chiefly PhOH and cresols) content was  $7.5\%$ . Oxidation with  $KMnO_4$  and  $O_3$  indicates the content of unsaturated (I) and aromatic (II) hydrocarbons, but the I val. is not a measure of (I) owing to interaction of (II). A. A. E.

**Photoelectric study of sedimentation of active carbons, fresh, exhausted, and regenerated.** V. D. NETUKA (Chim. et Ind., 1933, 29, Spec. No., 1160—1163).—Measurements of light transmission through  $0.2\%$  suspensions of Norit and Carboraffin over a period of 50 min. indicated almost equal rates of subsidence for fresh and regenerated Norit in distilled  $H_2O$  and only slightly higher rates for both in  $0.02N$ -KCl or  $-Na_2SO_4$  solution. Fresh or regenerated Carboraffin subsided rather more slowly than Norit in these salt solutions, and considerably more slowly in distilled  $H_2O$ , probably because it contains less sol. salts than Norit. Both carbons, after being exhausted by treatment of molasses solutions and then washed with  $H_2O$  until the washings were colourless, showed no perceptible subsidence in distilled  $H_2O$  within 50 min. and only very slow subsidence in  $0.02N$ -salt solutions. J. H. L.

**[Gas-]purification materials.** K. BUNTE, H. BRÜCKNER, W. LUDEWIG, and O. RUNGE (Gas- u. Wasserfach, 1933, 76, 785—789).—The activity of Fe oxide for gas purification depends on its  $H_2O$  content. Natural ores, bog ore, luxmasse, and pptd.  $Fe(OH)_3$  have been examined. Moisture is determined by loss in wt. on exposure in air at room temp. or by cold extraction with EtOH and measurement of its solution temp. with light petroleum. Chemically combined  $H_2O$  is removed by extraction with boiling  $C_2H_4Cl_2$  or by drying at  $160^\circ$ . Total  $H_2O$  may be determined directly by heating to redness in a stream of  $N_2$  and absorbing the  $H_2O$  in  $CaCl_2$ . Humic acid content is measured by oxidation with aq.  $KMnO_4$ . The velocity of adsorption of  $H_2S$  is obtained by a static method involving measurement of pressure drop. The above properties for 9 different oxides are tabulated. R. N. B.

**Causes of the presence of hydrogen sulphide in gas after purification with hydrated ferric oxide.** G. BIANCHETTI (Acqua e Gas, 1933, 22, 13—15; Chem. Zentr., 1933, i, 3388).—Small quantities of  $H_2S$  are formed by decomp. of  $CS_2$ , COS, and sulphurised hydrocarbons in presence of small quantities of  $H_2O$  and in contact with Fe pipes. S-free gas is obtained by heating at  $90$ — $100^\circ$ , cooling, and again purifying with hydrated  $Fe_2O_3$ . A. A. E.

**Evaluation of the benzol content of gas, and the economic importance of benzol washing.** R. MEZGER (Gas- u. Wasserfach, 1933, 76, 775—777; cf. B., 1930, 749).—Removal of benzol (I) should be profitable for any gasworks producing  $< 10^6$  cu. m. of gas per year. The thermal val. of the (I) is replaced by



addition of water-gas; a relationship between the val. of the (I) and the cost of producing coal gas and water-gas is given. (I) recovery increases the output of a works without a great increase in capital costs. Figures are given for the amount and cost of the extra coal carbonised. R. N. B.

**Gas as fuel for a calcining oven.** H. MANSKOPF (Gas- u. Wasserfach, 1933, 76, 772—775).—The installation of regenerative gas firing in place of coke firing in a crematorium is described; it resulted in a great saving in fuel costs. R. N. B.

**Primary tar of olive sansa [exhausted husks].** A. MANGINI and F. LABELLARTE (Annali Chim. Appl., 1933, 23, 373—391).—The tar described previously (B., 1932, 1111) appears to contain 1:2-dimethyl- and 2-ethyl-1:2:3:4-tetrahydro-, 4-ethyl-, 3-ethyliso-, and 3-methyl-2-ethyl-quinoline, and an unidentified base  $C_{11}H_{11}N$  (picrate, decomp. 200°, not melted at 220°);  $HCO_2H$ , an acid  $C_6H_{10}O_2$  (? hexoic), and octadecanecarboxylic acid. The phenol fraction (34.52%) contains complex phenols of pyrocatechol character, and the neutral oil (23.53%) five paraffins from  $C_{25}H_{52}$  to  $C_{29}H_{60}$ . T. H. P.

**Tar emulsions and their applications.** J. BOJANOWSKI (Przemysl Chem., 1933, 17, 225—230).—Tar emulsions for road construction are stabilised by 1:1 mixtures of a 1% solution of soft soap, pitch, and caseinogen with saturated aq. humic acid, or with a solution containing 3% of  $Na_2SiO_3$  and of certain by-products of PhOH chlorination. R. T.

**Road-tar consistency conversion chart.** E. O. RHODES (Eng. News-Rec., 1933, 111, 474—475).—The chart refers to road tars prepared from an average coke-oven tar. It covers interconversion of the following units at various temp.: degrees Engler, float test, Saybolt Furol, modified Redwood, Hutchinson (jun.) tar-tester, Saybolt Universal, and softening points by the Kraemer and Sarnow, ring-and-ball, and cube-in- $H_2O$  methods. R. N. B.

**Chemistry of pitch.** F. HOFMANN and L. BOENTE (Brennstoff-Chem., 1933, 14, 381—382; cf. B., 1933, 210).—The bases have been extracted from a pitch distillate and fractionated, partly under atm. and partly under reduced pressure. The presence in the fractions of the following compounds has been established:  $NH_2Ph$  (traces), dimethylpyridine,  $o$ - $C_6H_4Me \cdot NH_2$ , quinoline, isoquinoline, quinaldine,  $\beta$ - $C_{10}H_7 \cdot NH_2$ , acridine, and some higher-boiling bases of unknown constitution but probably of the higher condensed-ring type corresponding to pyrene. A. B. M.

**Sterlitamak crude oil.** A. S. VELKOVSKI and S. N. PAVLOV (Neft. Choz., 1932, 24, 231—235).—Characteristics of the crude oils and composition of various fractions are recorded. Gasoline and kerosene fractions are high in S. CH. ABS.

**Oil refining in the Far East.** E. V. FROLOV (Neft. Choz., 1932, 24, 204—208).—Products to be obtained at the Khabarovsk (Siberia) refinery are described. CH. ABS.

**Utilisation of petroleum oil refining by-products in the lacquer pigment industry.** G. ZILBERMAN,

A. BOLOTIN, and S. YAKUBOVICH (Lakokras. Ind., 1933, No. 1, 37—40).—A residual product of the cracking of oil had  $d$  0.933, I val. 177, acid val. 5, sap. val. 20; it reduces the oil-absorption capacity of pigments and can be substituted for linseed oil to the extent of 75%. CH. ABS.

**Theoretical comparison of petroleum distillation systems.** P. MEYER (J. Inst. Petroleum Tech., 1933, 19, 819—834).—Methods are derived mathematically for calculating the vol. of liquid boiling > the cut-point temp. present in the distillate for a hypothetical binary liquid for flash-vaporisation (I), continuous and batch distillation. Published results of (I) are used to check the method of calculation. The possibility of comparing the efficiencies of different distillation systems by means of an "overlap coeff." is discussed. D. K. M.

**Conversion of fatty and waxy substances into petroleum hydrocarbons.** W. F. SEYER (J. Inst. Petroleum Tech., 1933, 19, 773—783).—Theoretical. Petroleum is considered to arise from fatty acids by slow cracking at high pressures and comparatively low temp. The absence of  $H_2$  and unsaturated hydrocarbons in the more volatile fractions is attributed to the high pressure, and possibly to catalysis increasing the speed of rehydrogenation and polymerisation, since these are second-order reactions. The various types of petroleum are accounted for on the basis of their relative ages, allowance being made for differing environments. A. A. L.

**Destructive hydrogenation of Grozni paraffinic petroleum products.** L. A. ALEKSANDROV (Neft. Choz., 1932, 24, 301—307).—Hydrogenation experiments at 150—200 atm. original  $H_2$  pressure in presence of Fe and Ni catalysts are described. The gasolines had high stability and low gum content. CH. ABS.

**Examination and evaluation of carburetting oils.** P. SCHLÄPFER and S. SCHAFFHAUSER (Monatsbull. Schweiz. Ver. Gas- u. Wasserfachmänn., 1933, 13, 125—143, 159—168, 193—204; Gas J., 1933, 203, 676).—Laboratory cracking experiments have been carried out with gas oils at 650—790° with and without a  $H_2$  atm. The following properties of the oils and of 10° fractions were determined:  $d$ , distillation curve, dispersive power, ultimate composition and chemical nature of the hydrocarbons. These were correlated with the yield, calorific val., and composition of the gas produced. The temp. and rate of oil feed govern the production of the max. no. of B.Th.U. per lb. of oil. The nature of the oil affects the yield but not the composition of the gas. If  $H_2$  is present the calorific val. is increased by 90—225 B.Th.U./cu. ft., with a consequent reduction in vol. Oils containing > 50% of hydrocarbons boiling below 300° are unsuitable. The val. of an oil depends on its chemical composition, which may be expressed by the formula  $KH/G$ , where  $K$  is the average b.p. at 10 mm. pressure,  $H$  the H content, and  $G$  the  $d$ . A suitable oil should have a carburetting val. < 2600. R. N. B.

**Thermal aspects of carburation, with special reference to the vaporisation of ethyl alcohol.** J. SMALL (Phil. Mag., 1933, [vii], 16, 641—656).—



Theoretical. Total heat-fuel content and total heat-entropy charts for EtOH have been constructed, and are discussed. The action of the strangler valve and factors controlling rate of evaporation are considered.

H. J. E.

**Degree of purity of benzene, and dehydration by means of hetero-azeotropic distillation.** W. SWIENTOSLAWSKI and J. USAKIEWICZ (Rocz. Chem., 1933, 13, 495—500).—The  $H_2O$  content of  $C_6H_6$  is reduced to 0.0001% by a triple distillation, rejecting the lower-boiling fractions, which contain the hetero-azeotrope, b.p.  $69.25^\circ$ . The dry  $C_6H_6$  thus obtained boils at  $80.110^\circ$ , and its vapour condenses at  $80.106^\circ$ . EtOAc can readily be eliminated from  $C_6H_6$  by a similar procedure. Thiophen forms zeotropic mixtures with  $C_6H_6$ .

R. T.

**Smoke point of kerosene.** SUB-COMMEE. OF INST. OF PETROLEUM TECHNOLOGISTS (J. Inst. Petroleum Tech., 1933, 19, 812—814).—Standard conditions and apparatus for determining the max. height of a non-smoky flame obtainable with kerosene are described.

A. A. L.

**Determination of ethyl alcohol in gasoline-alcohol mixtures, using a Zeiss immersion refractometer.** F. S. MORTIMER and E. H. GIESE (Ind. Eng. Chem. [Anal.], 1933, 5, 356).—50 c.c. of the gasoline-EtOH mixture are shaken with 25 c.c. of  $H_2O$  to equilibrium. The  $n$  of the  $H_2O$  layer is then determined.

E. C. S.

**Ozone, knock-inducer extraordinary.** D. B. BROOKS (J. Inst. Petroleum Tech., 1933, 19, 835—844).— $O_3$  in the air intake of an internal-combustion engine reduces the  $C_8H_{18}$  no. of the fuel. Different amounts of  $O_3$  are required to counteract an equal improvement in the anti-knock val. of a fuel obtained by different knock inhibitors. In the concns. tried ( $> 0.012$  vol.-%  $O_3$  in air intake)  $O_3$  had no effect on power, economy, or optimum spark advance.

D. K. M.

**New distillation unit increases yield of lubricating oils.** P. TRUESDELL (Chem. Met. Eng., 1933, 40, 517—519).—A distillation unit at the plant of the Bradford (U.S.) Oil Refining Co. is described.

D. K. M.

**Evaluation of motor lubricating oils.** M. FREUND and S. THAMM (Petroleum, 1933, 29, No. 40, 1—14; No. 41, 1—13).—For a series of lubricating oils having the same viscosity ( $\eta$ ) at  $50^\circ$  the volatility (determined by fractionation under a pressure of 0.1 mm. Hg), the slope of the  $\eta$ -temp. curve, and, in general, the sludge formed on oxidation, increased with increasing  $d$ , whilst the C residue (Conradson), in general, decreased. The  $\eta$ -temp. and the boiling-range curves of an oil belonging to this series seem to be definitely determined by its  $d$ , whether the oil is a straight fraction from one type of crude or a mixture of different types. The stability of the oils on heating and their behaviour on coking, on the other hand, seem partly dependent on the amount of asphaltic substances in the oil. The C residue, the hard asphalt content, and the depth of colour of oils of the same  $d$  and approx. the same  $\eta$  seem to be related. For lubricating oils prepared in the same manner from the same crude the  $d$ , slope of

the  $\eta$ -temp. curve, C residue, depth of colour, and reactivity towards oxidation increased with increasing  $\eta$ ; for such oils, however, the  $d$ - $\eta$  const. has a characteristic const. val. (cf. B., 1928, 592). For the complete specification of a normal refined lubricating oil the val. of  $\eta$  at a standard temp., e.g.,  $50^\circ$ , of  $d$ , e.g., at  $20^\circ$ , of the setting point, and a suitable index of the asphalt content, e.g., the Conradson val. or the depth of colour, should be adequate.

A. B. M.

**Determination of absolute viscosity.** M. LOUIS and M. PEYROT (Ann. Office nat. Combust. liq., 1932, 7, 1055—1061; Chem. Zentr., 1933, i, 3656).—Flow methods for oils are discussed.

A. A. E.

**Determining moisture.**—See I. **Explosive decomp. of  $C_2H_2$ .** **Extracting phenols from phenoxide solutions.**—See III. **Effect of tar acids on wood.**—See IX. **Coal-tar coatings. Lacquer [films] and liquid fuels.**—See XIII. **Oil-field  $H_2O$  analysis.**—See XXIII.

See also A., Nov., 1103, **Dielectric losses of insulating oils.** 1126, **Nature of activated carbons.** 1180, **Determination of  $C_5H_5N$  bases in presence of  $NH_3$ .**

#### PATENTS.

**Furnaces for distilling or coking fuel.** O. HELLMANN (B.P. 399,053, 30.12.32. Ger., 30.12.31 and 20.6.32).—The charge of fuel to be carbonised is fed in a thin layer on to a rotating platform ( $P$ ) below which (and rotating therewith) are the heating flues and regenerators or recuperators for preheating the air for combustion and, if desired, the fuel gas also.  $P$  is divided by means of partitions into flat sectors. Pressure sections ( $S$ ), which can be lowered on to the charge when the latter reaches the plastic stage, are provided in apertures in the furnace roof, which is stationary; the undersides of  $S$  are ribbed so that while compressing the material they divide it into strips or cubes. A scraper, movable radially, discharges the carbonised material into a container wherein it is cooled, e.g., by the introduction of steam.

A. B. M.

**Coking retort oven.** J. BECKER, ASST. to KOPPERS Co. (U.S.P. 1,904,191, 18.4.33. Appl., 10.4.24).—In an oven having the cross-over principle of flow of the heating gases a series of independent horizontal-flow ducts (e.g., an interior pair, a mediate pair, and a side pair) are provided in the upper part of each heating wall ( $W$ ) and the vertical flues are divided into groups, the upper ends of the flues of each group communicating with the corresponding flow duct. A cross-over duct connects each of the flow ducts of one  $W$  with the corresponding flow duct of an adjacent  $W$ . The regenerators below the heating walls and coking chambers are so arranged that those for passing combustible gas are separated from those passing waste gas by regenerators for passing air.

A. B. M.

**By-product coke oven operation.** S. P. MILLER, ASST. to BARRETT Co. (U.S.P. 1,904,161, 18.4.33. Appl., 18.4.29).—The hot gases from a coke oven or retort are cleaned, e.g., by scrubbing with tar or by electrical pptn., in order to remove suspended tar and pitch, the treatment being carried out at a sufficiently high temp. to maintain a proportion of tar oils in the form of vapour.



The resulting gases and vapours are then brought into direct contact with a light tar or tarry oil, and cooled, whereby clean oils are condensed from the gas and blend with the tar to give a tarry oil product, suitable for use as a creosoting composition. A. B. M.

**[Laboratory] carbon-combustion furnace.** C. B. FRANCIS (U.S.P. 1,903,036, 28.3.33. Appl., 14.7.31).—The furnace (*F*) comprises a hollow tubular body lined with refractory material and end-walls of similar material. Each of the latter has a central opening for the combustion tube, surrounded by a no. of regularly spaced apertures through which project the ends of resistor elements, in the form of rigid bars, which extend through *F*. The ends of the resistor elements are connected to suitable terminals to which the heating current is supplied. A. B. M.

**Production of carbonaceous material.** A. OBERLE (U.S.P. 1,903,834, 18.4.33. Appl., 11.8.28).—Carbonaceous materials of mineral oil origin, *e.g.*, petroleum coke, pitch, or other residues, are converted into activated C by heating to 100–2000°, preferably under pressure (50–500 lb./sq. in.), in presence of steam, activating gases or salts, or mineral acids, etc., the volatile reaction products being withdrawn during the treatment. A. B. M.

**Carbonisation of coal.** T. E. W. SCHUMANN, Assr. to COMBUSTION UTILITIES CORP. (U.S.P. 1,901,476, 14.3.33. Appl., 7.3.29).—Coal is carbonised in an externally-heated retort so designed that coking takes place progressively in the direction away from the heated sides and a plastic zone is formed between the coked and uncoked fuel. Air or other gas containing O<sub>2</sub> is supplied through one or more central pipes and passes to the inner side of the plastic zone where it sets up a limited amount of combustion. The local heating so effected accelerates the coking process without adversely affecting the yields and quality of the products. A. B. M.

**(A) Production of carbon black. (B) Re-forming of natural gas.** J. A. YUNKER, Assr. to NATURAL GAS HYDROGEN CORP. (U.S.P. 1,902,746–7, 21.3.33. Appl., 8.7.30).—(A) A bed of coke is air-blasted (3 min.) and purged with steam ( $\frac{1}{2}$  min.), and a hydrocarbon gas, *e.g.*, natural gas, is then passed through the incandescent material (8–10 min.). The cycle of operations is repeated. The C black formed is recovered from the gases produced. (B) The gases produced during about the last  $\frac{3}{4}$  min. of the air blast, during the steam purge, and during the run with natural gas are purified and mixed. The mixture is blended with natural gas to give town's gas. A. B. M.

**Production of carbon black.** (A) D. J. BEAVER, (B) S. P. BURKE, (C) T. P. KELLER, Assrs. to GEN. ATLAS CARBON CO. (U.S.P. 1,902,753, 1,902,797, and 1,904,469, [A, B] 21.3.33, [C] 18.4.33. Appl., [A] 27.1.31, [B] 10.9.27, [C] 19.2.31).—(A) Hydrocarbon gases (I) are burned at a series of narrow, flattened burners (*B*) mounted in the lower part of a heat-insulated furnace chamber (*C*) in such a manner, *e.g.*, in a row with their longer sides parallel, that the flames radiate mainly to each other. They are surrounded

by air ducts wider than *B*. The air supply amounts to 40–75% of that required for the complete combustion of (I), and the size of *C* is such that the time of contact of the gases in the hot zone of *C* is preferably 0.5–1.0 sec. The C black is recovered from the gases leaving the stack at the top of *C*. (B) A stream of (I) is passed in stream-line flow within a stream of highly-heated gas in a suitable chamber so that decomp. of the hydrocarbons to form C black occurs in the gaseous phase and not on a solid surface. The heated gas is conveniently produced by partial combustion of the (I), *i.e.*, by supplying a regulated stream of air around the stream of gas. (C) (I) is discharged upwardly through a series of narrow ducts mounted in parallel, vertical planes in an unobstructed *C*, and a regulated supply of air is introduced into *C* near the lower end of the gas ducts. Partial combustion of (I) with formation of C black takes place. Deposition of C on the ends of the gas ducts is prevented by bevelling the edges thereof and so facilitating stream-line flow of (I) and air at these points. A. B. M.

**Manufacture of water-gas.** HUMPHREYS & GLASGOW, LTD., and A. R. GRIGGS (B.P. 399,119, 20.4.33).—The fuel bed (*B*) of the generator (*G*) is fed from a supply shaft (*S*) the lower part of the wall of which extends downwardly into *G* so as to form an annular chamber (*C*) surrounding the lower end of *S*. The floor of *C* is formed by the exposed surface of *B*. The blast gases are burned with secondary air in *C*, and during the water-gas supply period, if and when carburetting is required, oil, oil residuum, tar, etc. is vaporised and cracked therein, the gases so produced being "fixed" by passage through part of *B*. A. B. M.

**Treatment of tar and like oils.** W. HIGBURN and P. C. REILLY (U.S.P. 1,901,872, 21.3.33. Appl., 12.1.26).—The tar is distilled and the vapour passed through a heated tube, *e.g.*, at 510–750°, and then again condensed. The sulphonation residue, *i.e.*, the % of constituents insol. in H<sub>2</sub>SO<sub>4</sub>, is reduced and the *d* increased. A. B. M.

**Manufacture of low-boiling tar acids.** S. CAPLAN, Assr. to COMBUSTION UTILITIES CORP. (U.S.P. 1,904,269, 18.4.33. Appl., 29.9.26).—High-boiling tar acids are vaporised, mixed with an excess of steam, and passed through a reaction tube at 600–800°. No catalyst is used and the reaction is carried out in the absence of any material which reacts with steam to form H<sub>2</sub>. A. B. M.

**Production of asphalt.** L. KIRSCHBRAUN, Assr. to FLINTKOTE CORP. (U.S.P. 1,902,305, 21.3.33. Appl., 14.5.30. Cf. U.S.P. 1,715,069; B., 1929, 633).—Apparatus for making asphalt from petroleum residuum is described. A. B. M.

**Recovery of drip oil from certain residuums.** G. N. HARCOURT, Assr. to ALCO PRODUCTS, INC. (U.S.P. 1,901,238, 14.3.33. Appl., 2.4.30).—Water-gas tar emulsions are heated in stages to above 100°, and are then sprayed into a vaporising and separating chamber (*C*), from which unvaporised tar and H<sub>2</sub>O are withdrawn continuously from separate points. The H<sub>2</sub>O is utilised to preheat the ingoing emulsion. The vapours from



*C* are condensed and passed to a second separator, from which drip oil and H<sub>2</sub>O are separately withdrawn.

A. B. M.

**Gasifying of heavy oils through partial combustion.** SOC. DES PROC. CHILOWSKY, and C. CHILOWSKY (B.P. 398,640, 5.9.32).—An apparatus is described in which heavy oil is atomised directly into an unobstructed zone (*A*), by means of an amount of preheated air insufficient for supporting complete combustion, in such manner as to obtain in *A* a free jet of hot products in which a partial combustion of the heavy oil is being performed. The hot products are transferred to a second zone, separated from *A* by a heat-radiating body (*B*), and before leaving it they are caused to yield heat to the centre part of *B*.

H. S. G.

**Production of highly combustible gaseous fuel from hydrocarbons.** C. A. ROSIER (B.P. 398,037, 6.10.32).—Liquid hydrocarbons are subjected to the action of a catalytic agent while being heated for vaporising, expanding, and gasifying, and the gaseous constituents (hydrocarbons, air, or steam) are separately preheated in the same device, the intensity of the heat being adjusted to bring both to the desired temp.; the gasified hydrocarbons are subsequently injected into the gaseous constituent.

H. S. G.

**Treatment [cracking] of hydrocarbon oil.** UNIVERSAL OIL PRODUCTS CO., ASSEES. OF J. DE L. SEGUY (B.P. 398,566—7, Appl., 7.4.32. U.S., 7.4.31).—(A) The oil under pressure is subjected to a cracking temp. in a heating zone (*H*) and is delivered to a reaction zone (*R*) from which the products are removed separately or as commingled liquid and vapour. The unvaporised liquid is separated and subjected to cooling with or without accompanying vaporisation by reduction of pressure. The vapours are subjected to reflux condensation and the condensate is returned for further treatment. The conditions of operation, *i.e.*, cracking temp., pressure, and ratio of reflux condensate (returned to *H*) to the raw oil treated, are so correlated as to establish an operating index (*O. I.*) in accordance with the formula: [average of *H* outlet temp. and middle of *R* temp. (° F.)/30] + (pressure on *R* in lb. per sq. in./100) + reflux ratio. The *O. I.* is maintained at 36—38 so that a distillate is produced which represents a % of the raw oil treated equal to  $\leq 1\frac{1}{2} \times$  the A.P.I. gravity of the raw oil treated, or  $\leq$  the yield calc. from min. gasoline yield/gravity of charging stock = 3 — (gravity of charging stock/32). (B) The process is operated so as to establish *O. I.* = 39—43 in accordance with a similar formula (given).

H. S. G.

**Fractional distillation of [hydrocarbon] oils.** FOSTER WHEELER, LTD. FROM FOSTER WHEELER CORP. (B.P. 398,099, 17.2.33).—A fractionating tower equipped with bubble trays (*T*) is divided by a gastight partition into upper (*A*) and lower (*B*) chambers. Oil and vapour are introduced into *A* where they are separated, the liquid being led at substantially the same temp. into *B*, which is maintained under a total pressure lower than that in *A* by a thermo-compressor (*D*) supplied with steam and having its suction inlet connected to *B* at a point above the *T* therein. The steam from *D* and the vapours from *B* are discharged into *A* at a point

below the *T* therein, to reduce the partial pressure of the oil.

H. S. G.

**Hydrocarbon oil refining.** W. M. MALISOFF, ASSR. TO ATLANTIC REFINING CO. (U.S.P. 1,899,042, 28.2.33. Appl., 10.12.30).—Desulphurisation is effected by repeated washing with a solution of an alkaline sulphide and an alkaline hydroxide in at least one solvent from the group MeOH, EtOH, Pr<sup>n</sup>OH, Pr<sup>β</sup>OH, glycol, glycerin, COMe<sub>2</sub>, COMeEt, COEt<sub>2</sub>, and epichlorohydrin, such solution containing sufficient H<sub>2</sub>O to inhibit any mutual solubility of the oil and solution; the solution is separated from the oil after each wash.

H. S. G.

**Purification of petroleum and hydrocarbon compounds.** T. W. CULMER, ASSR. TO LINCOLN OIL REFINING CO. (U.S.P. 1,899,314, 28.2.32. Appl., 3.11.30).—Low-b.p. hydrocarbon oils are desulphurised by treatment with a Cd salt-NH<sub>3</sub> solution containing free NH<sub>3</sub>. The oil is filtered and the residue is dried, oxidised, and the Cd recovered as CdO, Cd(NO<sub>3</sub>)<sub>2</sub>, or CdCl<sub>2</sub> and returned to the process with additional NH<sub>3</sub>.

H. S. G.

**Treatment of sludge acid.** CHEM. CONSTRUCTION CORP., ASSEES. OF I. HECHENBLEIKNER (B.P. 398,023, 26.8.32. U.S., 10.10.31).—Sludges obtained by the H<sub>2</sub>SO<sub>4</sub> treatment of petroleum products are continuously passed through three zones (*A—C*) in order of rising temp. countercurrent to a stream of hot combustion gases containing 1—10% (2%) of O<sub>2</sub>. Zone *A* is maintained at a temp. high enough to evaporate H<sub>2</sub>O and to distil light hydrocarbons; zone *B*, from which SO<sub>2</sub> is removed, at a temp. at which the SO<sub>4</sub> content is rapidly reduced to SO<sub>2</sub>, but below that at which H<sub>2</sub>S is formed or at which SO<sub>3</sub> distils (*e.g.*,  $\gt 215^\circ$ ); and zone *C* at a temp. sufficiently high to transform the carbonaceous material of the sludge into a granulatable fuel.

H. S. G.

**Production of lubricating oils, kerosene, and other improved hydrocarbons from petroleum stocks by treatment with aluminium chloride.** GULF REFINING CO., ASSEES. OF E. AYRES and H. G. SMITH (B.P. 398,032, 30.9.32. U.S., 10.3.32).—The petroleum stocks (which advantageously may be mixed with a lighter petroleum fraction or a hydrocarbon of lower mol. wt.) are treated with AlCl<sub>3</sub> at  $\gt 150^\circ$  in presence of an excess of Cl<sub>2</sub> or HCl introduced into the stock either prior to or during the treatment. The AlCl<sub>3</sub> sludge is removed by settling and decanting off the resulting sour oil (I). Traces of suspended AlCl<sub>3</sub> remaining may be decomposed by heating to 230—285°. Alternatively, (I) may be agitated with dil. H<sub>2</sub>SO<sub>4</sub> and the acid separated therefrom, or an excess of Ca(OH)<sub>2</sub> and a little clay may be added and the oil filtered.

H. S. G.

**Production of lubricating oils.** EDELEANU GES.M.B.H. (B.P. 399,030, 31.10.32. Ger., 5.11.31).—A lubricating oil of high viscosity is obtained by subjecting the stock oil to an extraction according to the Edeleanu process at  $\gt 25^\circ$ .

H. S. G.

**Purification of viscous lubricating oils by liquid sulphur dioxide.** EDELEANU GES.M.B.H. (B.P. 398,992, 29.7.32. Ger., 30.12.31).—In an Edeleanu process, the viscosity of a highly viscous lubricating oil is reduced by mixing a substantial amount (10—40%) of SO<sub>2</sub> with



the oil prior to its reaching the countercurrent mixer and without effecting any separation into extract and raffinate, the amount of  $\text{SO}_2$  added being  $\ll$  the approx. limit of its solubility in the oil. H. S. G.

**Lubricants.** KODAK, LTD., Assees. of K. C. D. HICKMAN (B.P. 398,936, 18.4.32. U.S., 17.4.31).—A cellulose ether or ester is soaked in castor or sperm oil, then heated until dissolved, and, after cooling, the solution masticated with a heavy mineral lubricating oil constituting 25–50% of the finished lubricant.

H. S. G.

**Concn. of olefines.**—See III. Road surfacing.—See IX.

### III.—ORGANIC INTERMEDIATES.

**Polymerisation and explosive decomposition of ethylene under pressure.** G. EGLOFF and R. E. SCHAAD (J. Inst. Petroleum Tech., 1933, 19, 800–811).— $\text{C}_2\text{H}_4$  under pressure (50 atm.) in presence or absence of  $\text{Al}_2\text{O}_3$  requires a temp. of  $420^\circ$  in a continuous process to produce the same yield of oil as at  $380^\circ$  in a stationary autoclave. Polymerisation begins in the latter at  $325$ – $340^\circ$ . Paraffins up to  $\text{C}_9$ , and butadiene were detected in the products.  $\text{C}_2\text{H}_4$  at similar pressures in presence of a  $\text{NiO-Al}_2\text{O}_3\text{-CuO}$  catalyst explodes at  $330^\circ$ , the products being  $\text{C}$ ,  $\text{H}_2$ , and  $\text{CH}_4$ . A. A. L.

**Analysis of technical solvents.** IV. Colour reactions for trichloroethylene, carbon tetrachloride, and other chlorinated aliphatic hydrocarbons. H. H. WEBER (Chem.-Ztg., 1933, 57, 836; cf. B., 1923, 215).—Colour reactions for the detection in petrol and  $\text{C}_6\text{H}_6$  of the following compounds with (A) a 2% solution of  $\alpha\text{-C}_{10}\text{H}_7\text{-OH}$  in cyclohexanol, (B) cyclopentanol, and (C) 2% phenolphthalein in cyclohexanol are tabulated: (1)  $\text{CH}_2\text{Cl}_2$ , (2)  $\text{C}_2\text{H}_2\text{Cl}_2$ , (3)  $\text{CHCl}_3$ , (4)  $\text{CCl}_4$ , (5)  $\text{C}_2\text{H}_4\text{Cl}_2$ , (6)  $\text{C}_2\text{HCl}_3$ , (7)  $\text{C}_2\text{Cl}_4$ , (8)  $\text{C}_2\text{H}_2\text{Cl}_4$ , and (9)  $\text{C}_2\text{HCl}_5$ . The tests are made by boiling a drop of the liquid with 2 c.c. of A, B, or C, and a small piece of NaOH, then cooling (a) and adding an equal vol. of 85%  $\text{H}_2\text{SO}_4$  (b) or glacial AcOH (c) to form a lower layer, and shaking after 1 min.; (1) is yellow with Ac, (2) reddish-violet with Ab, (3) violet with Ab, (4) orange-yellow with Ac, (5) red with Ac, (6) lilac with Cc, (7) green with Bc, (8) green with Ab and yellow with Bc, (9) grey-green with Ab and yellow with Ac. A. R. P.

**Aliphatic hydrocarbons in "Lorol."** J. R. RUHOFF (J. Amer. Chem. Soc., 1933, 55, 3889–3890).—"Lorol" (a mixture of aliphatic alcohols formed by high-pressure hydrogenation of coconut oil) contains small amounts of *n*-undecane and *n*-tridecane. H. B.

**Catalytic high-pressure hydrogenation of aromatic hydrocarbons, and catalytic high-pressure destruction of the corresponding hydrogenated products.** II. Mesitylene. III. Hexamethylbenzene. A. DROS, A. J. TULLENERS, and H. I. WATERMAN (J. Inst. Petroleum Tech., 1933, 19, 784–799; cf. B., 1932, 791).—II. Mesitylene (improved prep.) [3 fractions (I), b.p.  $164.5$ – $164.9^\circ/769.4$  mm. (least stable, absorbs  $\text{O}_2$ ), (II), b.p.  $164.9$ – $165.6^\circ/769.4$  mm., and (III), b.p.  $165.6$ – $168.1^\circ/769.4$  mm., solidifying point  $-56^\circ$  (most stable)] is completely hydrogenated at lower temp. the greater is the pressure, the product being a mixture of *cis*- and *trans*-trimethylcyclohexane, which at  $450$ –

$460^\circ/180$  kg./sq. cm. for 1 hr. gave  $\text{H}_2$ ,  $\text{CH}_4$ , and aromatic hydrocarbons.

III.  $\text{C}_6\text{Me}_6$  (prepared by a continuous process) is removed from its hydrocarbon solutions with conc.  $\text{H}_2\text{SO}_4$ , and is completely hydrogenated at  $< 319^\circ$  with high pressures of  $\text{H}_2$ . Hexamethylcyclohexane at  $472^\circ$  with  $\text{H}_2$  is partly unchanged. A. A. L.

**Extraction of phenols from phenoxide solutions.** K. DREES and G. KOWALSKI (Brennstoff-Chem., 1933, 14, 382–383).—Mixtures of  $\text{C}_5\text{H}_5\text{N}$  with  $\text{Et}_2\text{O}$  or with  $\text{C}_6\text{H}_6$  are about 3 times as efficient as is  $\text{Et}_2\text{O}$  alone for this purpose. Ketones also appear to be good extraction solvents, but whereas  $\text{COMe}_2$  is itself too sol. in the aq. phenoxide the higher ketones are too costly for use in this process. A. B. M.

**Detection of *p*-phenylenediamine.** C. GRIEBEL (Z. Unters. Lebensm., 1933, 66, 253; cf. B., 1933, 582).—The methods of separation and identification of *p*- $\text{C}_6\text{H}_4(\text{NH}_2)_2$  etc. previously outlined (*loc. cit.*) are not practicable in the presence of 2 : 5-diaminoanisole.

E. C. S.

**Anti-freeze agents.**—See I. [Bases in] pitch.—See II. Determining NHPH<sub>2</sub>.—See XXII.

See also A., Nov., 1127, Electro-reduction of aromatic ketones. Oxidation of  $\text{NH}_2$ -acids. 1139, Prep. of  $\text{Pr}^\beta\text{Br}$ . Analysis of liquid org. substances. 1141, Prep. of  $\text{Bu}^+$  and amyl nitrites, aliphatic mercaptans and sulphonic acids of high mol. wt., and anhyd. AcOH. 1144, Prep. of  $\text{Ac}_2\text{O}$ . 1166, Prep. of tetrahydropyrene. 6-Aldehyde-4-methyl- $\alpha$ -naphthapyrone. 1172, Tetrazine. 1204, Prep. of lactic acid. Transforming  $\text{CH}_2\text{Ac-CHO}$  [into  $\beta$ -butylene glycol]. Prep. of phosphoglyceric acid. Synthesis of alanine.

### PATENTS.

**Conversion of methane into other hydrocarbons [ethane and ethylene].** H. DREYFUS (B.P. 399,526, 26.2.32).— $\text{CH}_4$  is heated in contact with solid or liquid Ni, Co, Cu, or Zn at  $350$ – $850^\circ$ , *e.g.*,  $600^\circ$ , to give  $\text{C}_2\text{H}_6$  and  $\text{C}_2\text{H}_4$ . C. H.

**Concentration of olefines in a mixture containing them.** R. M. DEANESLY, Assr. to SHELL DEVELOPMENT Co. (U.S.P. 1,866,800, 12.7.32. Appl., 5.6.29).—Liquid  $\text{NH}_3$  is added to the mixture, *e.g.*, of  $\text{C}_4\text{H}_{10}$  and  $\text{C}_4\text{H}_8$ , and the whole is distilled under pressure;  $\text{C}_4\text{H}_8$  predominates in the residue,  $\text{C}_4\text{H}_{10}$  in the distillate. C. H.

**Detection of methyl chloride [and other gases containing chlorine].** A. B. LAMB (U.S.P. 1,864,544, 28.6.32. Appl., 1.10.29).—Portable apparatus is described by which a sample of gas can be blown across a Cu spiral heated by a non-luminous flame. C. H.

**Chlorination of paraffin [aliphatic] and/or aromatic hydrocarbons.** N. V. DE BATAAFSCHE PETROLEUM MAATS., Assees. of R. M. DEANESLY (B.P. 399,991, 29.10.32. U.S., 11.11.31).—Chlorination is induced in absence of sunlight by simultaneous addition of  $\text{Cl}_2$  to olefines in substantial absence of free  $\text{O}_2$ .  $\text{C}_4\text{H}_{10}$  gives Cl- and  $\text{Cl}_2$ -derivatives at  $0^\circ$  in weak daylight in presence of 10% of  $\text{C}_4\text{H}_8$ . C. H.



**Manufacture of aliphatic [acetic] anhydrides.** H. DREYFUS (B.P. 399,792, 1.4.32).—Anhydriation is effected in presence of 5–25% (8–15%) of added H<sub>2</sub>O vapour, *e.g.*, at 500–700°. C. H.

**Manufacture or treatment of aliphatic [acetic] anhydrides.** BRIT. CELANESE, LTD., H. F. OXLEY, L. FALLOWS, and H. DREYFUS (B.P. 399,793, 1.4.32).—The (acetic) acid is anhydriated at >550° (*e.g.*, 550–700°) in presence of a catalyst (NaPO<sub>3</sub>, CaWO<sub>3</sub>) mixed with a non-reducible metal oxide, except WO<sub>3</sub>, as promoter, *e.g.*, NaPO<sub>3</sub> with Na<sub>3</sub>PO<sub>4</sub> (2%) and CaO (5%), or with SrO (7%), or CaWO<sub>3</sub> with CaO (10%), or Al<sub>2</sub>O<sub>3</sub> (5%), or V<sub>2</sub>O<sub>5</sub> (13%). C. H.

**Treatment [concentration] of aliphatic [acetic] acids.** BRIT. CELANESE, LTD., H. F. OXLEY, and W. H. GROOMBRIDGE (B.P. 399,529, 7.3.32).—The dil. acid is conc. to 60–70% by extraction with a volatile org. solvent (Et<sub>2</sub>O) of b.p. < that of the acid, and further H<sub>2</sub>O is removed as azeotropic mixture (preferably of b.p. > temp. of distillation of the solvent) with an org. liquid (C<sub>6</sub>H<sub>6</sub>, C<sub>2</sub>H<sub>4</sub>Cl<sub>2</sub>, C<sub>2</sub>H<sub>2</sub>Cl<sub>2</sub>, C<sub>2</sub>HCl<sub>3</sub>, EtOAc, BuOAc). C. H.

**Manufacture of water-soluble crystalline aluminium salts of lower fatty acids.** M. SCHWARZ, F. and E. ZSCHIMMER, R. and W. SCHWARZ (ZSCHIMMER & SCHWARZ CHEM. FABR. DÖLAU) (B.P. 399,737, 13.4.33. Ger., 18.4. and 15.8.32).—Aq. solutions of Al formate, acetate, etc. are mixed with the corresponding acid. On keeping for several days, with or without agitation, crystals separate. In the case of aq. Al formate of  $d \leq 1.100$  the HCO<sub>2</sub>H may be omitted. C. H.

**Polymerisation of vinyl derivatives.** W. E. LAWSON, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,867,014, 12.7.32. Appl., 22.9.28).—A solution of the vinyl compound or compounds (vinyl chloride and acetate, styrene), *e.g.*, in EtOAc, PhEt, etc., together with catalyst (Bz<sub>2</sub>O<sub>2</sub>) is forced through a heated Pb-lined tube at about 120°, the products being cooled and withdrawn through a regulated needle-valve. C. H.

**Manufacture of a preservative agent against moths and the like.** J. SIBILLE (B.P. 399,938, 21.6.32).—Cryst. quassin and quinine are treated with HBr and H<sub>2</sub>O<sub>2</sub> in EtOH with addition of a little glycerol (etc.) to prevent pptn.; or are treated with oleic acid and H<sub>2</sub>O<sub>2</sub> in petrol (etc.) with addition of a little heavy oil (cade oil, kerosene, etc.). C. H.

**Manufacture of higher alcohols from fats or fatty oils.** E. I. DU PONT DE NEMOURS & Co. (B.P. 399,848, 7.3.32. U.S., 5.3.31).—Glycerol esters of higher fatty acids are hydrogenated at 300–400°/100–205 atm. in presence of a Cd–Cu–Zn chromite catalyst. The resulting alcohols may be further hydrogenated in presence of a Ni catalyst to reduce olefinic linkings. Examples are the reduction of cottonseed, coconut, and castor oils. C. H.

**Preparation of heptaldehyde and undecylenic [undecenoic] acid [from castor oil].** H. A. BRUSON and J. D. ROBINSON, Assrs. to RESINOUS PRODUCTS & CHEM. Co., INC. (U.S.P. 1,889,348, 29.11.32. Appl., 9.5.31).—A mixture of castor oil and an acidic resin (rosin, ester gum) is vac.-distilled (at 300–340°). The

still-residues are fusible while hot, readily removed from the still, and are claimed to be of commercial val. H. A. P.

**[Preparation of] compound of hexamethylene-tetramine and [methylene]diphosphoric acid.** E. FREUND, Assr. to HEYL & Co., CHEM.-PHARM. FABR. A.-G. (U.S.P. 1,889,090, 29.11.32. Appl., 20.4.29. Ger., 20.4.28).—The compound, (CH<sub>2</sub>)<sub>6</sub>N<sub>4</sub>CH<sub>2</sub>(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub>, is prepared from hexamine and methylenediphosphoric acid in aq. or alcoholic solution. It is claimed that it liberates CH<sub>2</sub>O in urine (*in vivo*) even if the latter is alkaline. H. A. P.

**Manufacture of benzoic acid, metal benzoates, and alkyl benzoates.** A. GEORGE, Assr. to MONSANTO CHEM. WORKS (U.S.P. 1,866,849, 12.7.32. Appl., 22.4.25).—CPhCl<sub>3</sub> is treated with 1.8–1.9 mols. of H<sub>2</sub>O and catalyst (ZnCl<sub>2</sub>, AlCl<sub>3</sub>, Fe, Zn dust), and the BzCl is esterified, *e.g.*, with EtOH. The purified ester is converted into metal benzoates and BzOH. C. H.

**Manufacture of 6-bromo- [or -chloro-] 2:4-dinitroaniline.** I. G. FARBENIND. A.-G. (B.P. 399,769, 30.6.33. Ger., 2.7.32).—Br or Cl<sub>2</sub> is led gradually into an aq. suspension of 2:4-dinitroaniline, *e.g.*, through a distributing nozzle with rapid agitation, preferably at 40–60°. The 6-Br- (m.p. 151–152°) and 6-Cl- (m.p. 155°) -compounds are described. C. H.

**Manufacture of aromatic hydroxy-aldehydes.** F. HOFFMANN-LA ROCHE & Co. A.-G. (B.P. 399,723, 25.3.33. Ger., 18.5.32).—*o*- or *p*-Hydroxybenzyl alcohols are oxidised in aq. alkaline solution with a nitroarylsulphonic or -carboxylic acid (excluding 1-Cl-2-NO<sub>2</sub>-acids). Examples are: vanillin from vanillyl alcohol or 4-hydroxy-3-methoxyphenyltrichloromethylcarbinol; *p*- and *o*-hydroxybenzaldehyde from hydroxymethylphenols; 4-hydroxyvitaldehyde, m.p. 133°, from di(hydroxymethyl)resol. C. H.

**Purification of phthalide.** A. O. JAEGER, Assr. to SELDEN RESEARCH & ENG. CORP. (U.S.P. 1,889,961, 6.12.32. Appl., 10.11.30).—The odorous and coloured impurities in technical phthalide are removed by distillation in steam (slightly superheated). H. A. P.

**Preparation of organic acids and products [alkylated aromatic keto-acids].** H. A. BRUSON, Assr. to RESINOUS PRODUCTS & CHEM. Co. (U.S.P. 1,889,347, 29.11.32. Appl., 5.5.30. Cf. B.P. 365,038; B., 1932, 494).—Aromatic hydrocarbons or their derivatives are condensed with the anhydride of a dicarboxylic acid (phthalic anhydride) and an olefine or alkyl monohalide in presence of AlCl<sub>3</sub>, SnCl<sub>4</sub>, or FeCl<sub>3</sub>. A solvent or diluent may be used and the reaction carried out in one or two stages. *o*-Amylbenzoylbenzoic acid is specifically claimed. H. A. P.

**Production of primary aromatic amines of the benzene series by hydrogenation of their corresponding azo ethers.** R. P. PERKINS and P. S. PETRIE, Assrs. to DOW CHEM. Co. (U.S.P. 1,890,430, 6.12.32. Appl., 21.9.29).—Azo dyes containing > 1 alkoxy group and free from additional substituents other than alkyl groups are hydrogenated (Ni–Al<sub>2</sub>O<sub>3</sub>) under pressure (200–500 lb./sq. in.) in absence of a solvent and at



the m.p. of the dye to 200° (120—150°). The examples all refer to  $p\text{-NH}_2\cdot\text{C}_6\text{H}_4\cdot\text{OEt}$ . H. A. P.

**Separation of *N*-mono- and -di-alkyl aromatic amines of the benzene series.** E. C. BRITTON and R. D. HOLMES, Assrs. to DOW CHEM. CO. (U.S.P. 1,890,246, 6.12.32. Appl., 13.4.28).—The mixed amines are heated with a dicarboxylic acid (phthalic acid in absence of a solvent) at 100—200°, the (phthal)anilic acid thus formed from the *sec.*-amine is removed by treatment with aq. alkali, and the residual *tert.*-amine purified by distillation. The *sec.*-amine may be recovered by hydrolysis of the anilic acid with alkali or acid.

H. A. P.

**Manufacture of solid stable diazoazo salts.** I. G. FARBENIND. A.-G. (B.P. 399,753, 19.5.33. Ger., 19, and 20.5.32).—Diazotised aminoazo-*o*- or -*m*-toluene is treated with enough  $\text{H}_2\text{SO}_4$  to form the H sulphate, which is filtered off and mixed, if desired, with a diluent or dyeing assistant, e.g., borax, and partly dehydrated  $\text{Al}_2(\text{SO}_4)_3$ .

C. H.

**Separation of [2- and 4-]chlorodiphenyls.** E. C. BRITTON and W. C. STOEßER, Assrs. to DOW CHEM. CO. (U.S.P. 1,890,427, 6.12.32. Appl., 22.12.30).—The crude chlorination mixture after removal of free HCl is distilled, the fractions (I) b.p. 260—274°/1 atm. and (II) 274—284°/1 atm. being reserved. (I) is a mixture of  $\text{Ph}_2$  and  $2\text{-C}_6\text{H}_4\text{PhCl}$  (III), and is separated by cooling to a temp. slightly > the eutectic [19°; 31.6%  $\text{Ph}_2$ , 68.4% (III)], when  $\text{Ph}_2$  crystallises and is filtered off; the mother-liquors on redistillation give a fraction (b.p. > 260°) containing > 95% of (III) and fractions of lower b.p. similar in composition to (I). Fraction (II) is a mixture of (III) and  $4\text{-C}_6\text{H}_4\text{PhCl}$  (IV) and is similarly separated, the crystals that separate above the eutectic [13°; 70% (III) and 30% (IV)] being almost pure (IV).

H. A. P.

**Preparation of aminodiaryl ethers [4:4'-diaminodiphenyl ether].** E. F. GREYER, Assr. to DOW CHEM. CO. (U.S.P. 1,890,256, 6.12.32. Appl., 19.3.28).—Halogenodiaryl ethers are heated with (aq.)  $\text{NH}_3$  at 150—250° (170°) in presence of  $\text{Cu}^I$  derivatives ( $\text{Cu}_2\text{O}$ ).

H. A. P.

(A) Preparation, (B) purification, of aryl[am]ides of 2:3-hydroxynaphthoic acid. J. M. TINKER and L. SPIEGLER, Assrs. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,890,201—2, 6.12.32. Appl., 16.11.31).—(A) 2-Hydroxy-3-naphthoic acid is caused to interact with the hydrochloride of the arylamine in presence of a dehydrating agent ( $\text{PCl}_3$ ) in an inert solvent. It is claimed that the use of the hydrochloride instead of the free amine leads to a smoother reaction and purer products. (B) The arylamide (I) is milled with an aq. solution of a feebly alkaline substance (II) (e.g.,  $\text{NH}_3$ ,  $\text{NaHCO}_3$ , acetates, borates, phosphates), filtered off, and washed with  $\text{H}_2\text{O}$ . Solid, finely-divided mixtures of (I), (II), and wetting agent are claimed.

H. A. P.

**Manufacture of nuclear-substitution products of acenaphthalic [acenaphthene-5:6-dicarboxylic] acid and 1:4:5:8-naphthalenetetracarboxylic acid.** I. G. FARBENIND. A.-G. (B.P. 400,069, 2.3.33. Ger., 2.3.32).—Acenaphthene-5:6-dicarboxylic acid (I)

is sulphonated in position 3; the product is oxidised to the  $(\text{CO}_2\text{H})_4$ -acid, which by alkaline fusion yields  $\beta$ -naphthol-1:4:5:8-tetracarboxylic acid. (I) is converted by  $\text{HNO}_3$  in  $\text{H}_2\text{SO}_4$  into a dinitronaphthalene-1:4:5:8-tetracarboxylic acid, or by  $\text{POCl}_3$ ,  $\text{SO}_2\text{Cl}_2$ , and  $\text{PCl}_5$  into chloroacenaphthenedicarboxylic acid, m.p. 208—210° (anhydride, m.p. > 300°). C. H.

**[Manufacture of] anthraquinone derivatives.** IMPERIAL CHEM. INDUSTRIES, LTD., and F. LODGE (B.P. 399,528, 4.3. and 29.4.32).—A 1-halogenoanthraquinone-2-sulphonic acid free from further  $\text{SO}_3\text{H}$  groups is treated with a suitable amine with or without a Cu catalyst; an acid-absorber (alkali, or excess of amine) and a solvent ( $\text{H}_2\text{O}$ , EtOH) may be used, and further replaceable groups may also react. The 1-methylamino-, 1:4-di-*p*-toluidino-, and 1-anilino-2-sulphonic acids are described. C. H.

**Erratum.**—On p. 822, col. 1, lines 16 and 17, for  $\text{CuSO}_4$  read  $\text{CaSO}_4$ .

**Azo intermediates.**—See IV. Wetting agents.—See VI.

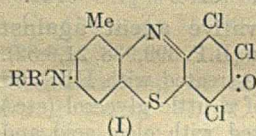
## IV.—DYESTUFFS.

**Fastness of coal-tar dyes.** H. HAASE (Farbe u. Lack, 1933, 484, 498).—The causes of discordance in light-resistance measurements are discussed. For useful data the dye should be tested only under practical conditions, i.e., dispersed in its medium, spread on a suitable surface, etc. S. M.

See also A., Nov., 1168, Yellow colouring matter of Khapli wheat. Dyes of yellow dahlias. Dyes from 6-aldehyde-4-methyl- $\alpha$ -naphthopyrone. 1169, Indigoid dyes. 1172, Dyes from phenanthraquinone. 1173, Blue Na salt of rhodamine-B and related substances. 1174, Azine dyes. 1185, Biological stains (nitro- and azo-dyes).

## PATENTS.

**Manufacture of dyes containing sulphur.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 399,583, 27.4.32).—Dyes (I) in which



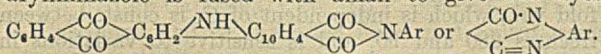
R = H, alkyl, or aralkyl, and R' = aryl carrying a 1 group R''-NH-SO<sub>2</sub> or R''-SO<sub>2</sub>-NH are prepared by the process of claim 5 of B.P. 265,641 (B., 1927, 325); they are suitable for machine- and piece-dyeing. 5-Chloro-2:3-tolylene-thiazthionium chloride is condensed with 4-amino-diphenyl-4'-sulphonanilide, hydrolysed to the *o*-amino-thiophenol Zn salt, and condensed with chloranil. In other examples NRR' is *p*-NH·C<sub>6</sub>H<sub>4</sub>·SO<sub>2</sub>·NHPh, *m*-NH·C<sub>6</sub>H<sub>4</sub>·SO<sub>2</sub>·NH·C<sub>10</sub>H<sub>7</sub> ( $\beta$ ), *p*-NH·C<sub>6</sub>H<sub>4</sub>·NH-SO<sub>2</sub>Ph, *pp'*-NH·C<sub>6</sub>H<sub>4</sub>·C<sub>6</sub>H<sub>4</sub>·SO<sub>2</sub>·NH·C<sub>6</sub>H<sub>4</sub>Cl (*m*), *pp'*-NH·C<sub>6</sub>H<sub>4</sub>·C<sub>6</sub>H<sub>4</sub>·SO<sub>2</sub>·NH·C<sub>6</sub>H<sub>4</sub>·OMe (*o*), and 2:7-NH·C<sub>10</sub>H<sub>6</sub>·SO<sub>2</sub>·NHPh. The shades are green-blue or blue. C. H.

**Manufacture of acylamino-compounds [of the anthraquinone series].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 399,790, 9.3.32).—An amino-anthraquinone is condensed with a quinoline or naphthaquinoline carrying CO<sub>2</sub>H in the homocyclic ring; or a halogenoanthraquinone with an amide of such an acid.



The products are vat dyes. Examples include: quinoline-6-carboxylic derivatives of 1:4- or 1:5-aminobenzamido- (scarlet-red or red-yellow), 1- or 2-amino- (green-yellow), 1:5-diamino- (red-yellow), 2-amino-3-cyano- (green-yellow), or 1:5-diamino-4:8-dihydroxy- (violet) anthraquinone;  $\alpha$ -naphthaquinoline-5-carboxylic derivatives of 1:5-aminobenzamido- (yellow) or 1:5-diamino- (green-yellow) -anthraquinone. C. H.

**Manufacture of vat dyes of the anthraquinone series.** I. G. FARBENIND. A.-G. (B.P. 399,724, 27.3.33. Ger., 26.3.32).—A 4- $\alpha$ -anthraquinonylamino-1:8-naphthalic arylimide or the corresponding naphthoyle-aryliminazole is fused with alkali to give vat dyes



The product, m.p. 400°, from 4-bromonaphthalic acid and 1-aminoanthraquinone is condensed with  $o$ - $C_6H_4(NH_2)_2$  (I) and the resulting iminazole, m.p. 373°, gives with KOH at 240° a green vat dye (II); the anil yields a blue-green. The iminazole, m.p. 241–243°, from 4-bromonaphthalic anhydride and (I) forms with  $\alpha$ -aminoanthraquinone a product, m.p. 367–368°, which gives (II) on alkaline fusion; the iminazole may be separated into isomerides, m.p. 223° and 283°, respectively, which are converted into  $\alpha$ -anthraquinonylamino-derivatives, m.p. 380° and 400°, and vat dyes, m.p. 342–343° (green) and 363° (yellow-green), respectively. The corresponding iminazole, m.p. 230°, from 4-chloro- $o$ -phenylenediamine is condensed with  $\alpha$ -aminoanthraquinone and fused with KOH to give a dull green vat dye. A bisiminazole, m.p. > 360°, is obtained from 3:3'-diaminobenzidine, and leads to a yellow-green vat dye. C. H.

**Manufacture of vat dyes [of the anthraquinone-acridone series].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 399,491, 4.4.32).—A 3-halogenoanthraquinone-2-carboxylic acid or ester (e.g.,  $CH_2Ph$  ester) carrying halogen or  $NO_2$  in position 1 is condensed with an aniline carrying < 2 halogen atoms, < 1 being in position 3 or 5, and the acridone ring is closed. The products are improved by bromination. Examples of suitable anilines are the 2:3:4:5- $Cl_4$ , 3:5- $Cl_2$ , 2:4:5- and 3:4:5- $Cl_3$  compounds. C. H.

**Manufacture of azo dyes [for wool].** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 399,916, 17.5.32).— $o$ -Aminodiphenylsulphone or a halogen, alkyl, or alkoxy derivative thereof is diazotised and coupled (acid) with  $\gamma$ -acid. The 2- $NH_2$ , 2- $NH_2$ -4'-Me-, and 5- or 4'-Cl-2- $NH_2$ -derivatives give level-dyeing, blue-red acid dyes. C. H.

**Dye composition [for home-dyeing of silk].** L. PHILIPPE (U.S.P. 1,889,491, 29.11.32. Appl., 11.7.29).—A mixture of  $H_2O$ -sol. acid or azo dye,  $Na_2SO_4$ , starch,  $NaHSO_4$ , and perfume is sealed in a gelatin capsule. H. A. P.

**Manufacture of [direct azo] dyes and intermediate products therefor.** SOC. CHEM. IND. IN BASLE (B.P. 400,059, 17.2.33. Switz., 18.2.32).—Diaminostilbenedicarboxylic acids in which the  $CO_2H$  are *ortho* to the  $NH_2$ -groups, synthesised from suitably substituted stilbenes, are tetrazotised and coupled with coupling

components. The dyes may be converted into metal complex derivatives in substance or on the fibre. 4:4'-Diaminostilbene-3:3'-dicarboxylic acid, m.p. > 280°, prepared from the  $(NO_2)_2$ -compound, is tetrazotised and coupled with: S-acid (grey-blue; +Cu, green-blue); phenyl-J-acid (red-violet; +Cu, pure violet); S-acid and acetoacetanilide (brown-grey; +Cu, olive); 1-(2'-chloro-5'-sulphophenyl)- and 1-phenyl-3-methyl-5-pyrazolones (red; +Cu, red-orange); etc. C. H.

**Manufacture of disazo dyes insoluble in water [for colouring rubber etc.].** I. G. FARBENIND. A.-G. (B.P. 400,024, 24.12.32. Ger., 24.12.31).—A dichloro- or dibromo-dianisidine or -diphenetidine is tetrazotised and coupled with an acylacetylated aromatic monoamine. Examples are: 2:2'- or 6:6'-dichlorodianisidine  $\rightarrow$  acetoacetic anilide, *o*-anisidine, *m*-4-xylylide, or benzoylacetic anilide. The yellow pigments obtained are suitable for colouring rubber or cellulose nitrate lacquers, or in Sn printing. C. H.

**Azo ethers.**—See III. **Body colours.**—See XIII.

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

**Swelling of protein fibres. II. Silk gut.** (MISS D. J. LLOYD and R. H. MARRIOTT (Trans. Faraday Soc., 1933, 29, 1228–1240).—Silk fibres immersed in  $H_2O$  or many aq. solutions absorb about 30% of their dry wt. of  $H_2O$ . In NaOH solutions > 0.01N the fibres swell continuously at a rate which is proportional to the concn. of NaOH up to 6N; meanwhile the fibres disintegrate into fibrils and the protein softens. Pretreatment with NaOH causes greater absorption of  $H_2O$  under all conditions. Alkaline swelling is probably due to an irreversible opening of a co-ordinate linking between the CO group of one peptide chain and the NH group of a parallel chain with a binding of  $H_2O$  mols. at the two groups released. This process may be followed by slow hydrolysis and disintegration of the structure. The fibres remain rigid in aq. HCl < 2N, but are readily broken into fibrils of about 0.3  $\mu$  diam. At higher concns. of HCl the fibres swell; in 10N-HCl they dissolve within 24 hr. In aq. HCl > 0.5N silk fibroin develops a purple colour. Swelling is not increased by pretreatment with acid. Acid probably destroys lateral cross-linkings, but does not cause binding of  $H_2O$ , although hydrolysis and disintegration of the structure may follow. Salts, urea, and  $CS(NH_2)_2$  weaken the structure and cause slight swelling. Silk fibres are strongly birefringent. Treatment with acid or alkali does not affect the X-ray diagram. E. S. H.

**Yolk in New Zealand wools. II. Yolk in the fleece during one year.** W. G. SUTTON (J. Text. Inst., 1933, 24, T 341–350; cf. B., 1931, 875).—The  $Et_2O$ - and  $H_2O$ -sol. fractions of the yolk show seasonal changes with a marked increase of yolk at the beginning of winter. The finer are the wools the greater is the proportion of yolk. A. G.

**Determination of the solvent power of organic liquids for cellulose esters.** I. SAKURADA and I. KIDO (J. Cellulose Inst., Tokyo, 1933, 9, 238–246).—The liquid to be tested is mixed in various proportions with  $C_6H_6$ , the solubility of the cellulose ester in each



mixture is determined, and the S-shaped concn.-solubility curve plotted. The solvent power of the liquid is measured by the concn. required to dissolve 50% of the ester, or by the concn. at which the curve is steepest. Data are given for two cellulose nitrates and for several solvents. A. G.

**Incompletely acetylated fibre cellulose.** A. RHEINER (Angew. Chem., 1933, 46, 675—681).—A lecture. R. S. C.

**Omission of stretch-drying [of viscose threads].** Z. KAWATA (J. Cellulose Inst., Tokyo, 1933, 9, 246—249).—If stretch-drying before desulphurisation is omitted the filament is slightly improved in handle, strength, extensibility, and affinity for dyes, the lustre slightly diminished, and the length unaffected. A. G.

**New autographic load-extension recorder [for textile yarns and fibres].** H. S. CLIFF (J. Text. Inst., 1933, 24, T 351—360).—A load-extension diagram with rectangular co-ordinates is obtained by applying the load by the rotation of one end of a long spiral spring; the diagram is obtained on a plate the movement of which is proportional to the angle of rotation of the spring. The record is made by a pen in an instrument for threads of breaking loads of 50—500 g., and by a photographic plate and reflected beam of light in a smaller instrument. A. G.

**Digestion of [wood] pulp.** E. HÄGGLUND (Zellstoff u. Papier, 1933, 13, 473—476).—Recent methods of external circulation and indirect heating of liquor in sulphite- and sulphate-digestion are described. D. A. C.

**Characteristic of [wood] pulps.** V. HOTTENROTH (Papier-Fabr., 1933, 31, 557—578).—Different methods of determining the various chemical consts. of pulps (bleach consumption, lignin content, Cu no., etc.) as well as results from different laboratories are compared and discussed. Determination of the OMe content as a basis of finding the lignin content gives fairly good agreement with the direct methods.  $\text{CH}_2\text{Cl}_2$  extracts of rosin give quantities comparable with those of  $\text{Et}_2\text{O}$  extracts. Unbleached sulphite pulps can be classified into groups of varying "hardness" by their lignin content, which grouping, however, is not applicable to bleached pulps, and is applicable to soda and sulphate pulps only with the use of correction factors. D. A. C.

**Hardness of wood pulps.** A. NOLL (Papier-Fabr., 1933, 31, 581—586).—Sulphite pulps (I) are classified into 24 grades of varying hardness obtained by determination of their degree of cooking (by the Johnsen, Sieber, etc. methods), each grade corresponding to a definite lignin content ( $L$ ). Soda pulps (II) having the same  $L$  will have a higher apparent hardness, as determined by these methods; their true hardness may be calc. from their  $L$  vals., and these fall within the same range as those of (I). Unlike (I), the bleach consumption of (II) depends on their apparent hardness. The rosin content has no influence on hardness determinations. The relation between the hardness, cooking degree,  $L$  val., and bleach consumption of (I) and (II) is given together with a modification of the Johnsen method applicable to all unbleached wood pulps. D. A. C.

**Strength evaluation of pulp for papermaking.** E. RICHTER (Zellstoff u. Papier, 1933, 13, 431—434, 476—480).—A method is sought for evaluating pulp characteristics, which will be independent of the treatment, age, and moisture content ( $M$ ) of the pulp, and const. at all basis wts. of the paper. The % ratio of burst factor and freeness (Canadian) to Cu no. ( $C$ ) (relative burst val.) is const. for all basis wts. above 170 g. per sq. m., and approx. const. with variations in conditions of beating, age, and  $M$  of pulp. Alkaline and acid pulps are not comparable since the  $C$  of the former is increased by the residual alkali, giving much higher vals. The relative breaking length val. is similarly const. for a range of freeness of 300—500. The relative fold val., which is independent of  $C$ , is const. between freeness 280 and 90, and is sensitive to variations in cooking and bleaching. The effect of stretch of fibre or sheet on bursting strength may be shown by calculating the burst per g. of substance per sq. in. D. A. C.

**Adsorption of alcohol by fibrous materials.** R. T. MEASE (Ind. Eng. Chem. [Anal.], 1933, 5, 317).—Paper extraction thimbles; cotton, wool, silk, and rayon adsorb EtOH and retain a considerable amount, even when dried to const. wt. at temp. considerably  $>$  the b.p. of EtOH. The phenomenon is not observed with  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{CCl}_4$ , or  $\text{C}_2\text{HCl}_3$ . E. S. H.

**Erasures and ultra-violet light.** C. A. MITCHELL (Analyst, 1933, 58, 532—533).—The erasure of pencil marks on paper by india-rubber etc. can be detected by ultra-violet light (I) only if the surface of the paper is abraded to such an extent as to become more transparent to transmitted light, in which case the rubbed place becomes dark in (I). Chemical removal of ink is plainly visible in (I), and different types of eradicators can sometimes be distinguished. E. C. S.

**Detection of a banknote forgery by means of ultra-violet light.** J. GRANT (Analyst, 1933, 58, 603—604).—The only conclusive test was examination in filtered ultra-violet light, when blue lettering appeared black in the genuine note and pale green in the forgery, indicating that different dyes had been used. The water-mark was removable by  $\text{Et}_2\text{O}$  from the forgery only, but the design was then still visible by reflected ultra-violet light, although neither the genuine nor the forged watermark was so visible before treatment. Differences in the expansion of paper on wetting may give rise to dimensional variations in the designs on genuine and forged notes. J. G.

**Gloss measurement.**—See I. Treatment of  $\text{H}_2\text{O}$ .—See XXIII.

See also A., Nov., 1108, X-Ray study of silks. 1113, Exchange of cations by sulphite-cellulose. 1114, Viscosity of cellulose nitrate solutions. 1134, Crystallite orientation in cellulose fibres. 1178, Action of KBr and of HCl on silk.

#### PATENTS.

**Washing of wool in the yolk.** L. DELFOSSE (B.P. 399,634, 2.8.32).—The yolk solution from the washing of raw wool is purified in a continuous manner by decantation and centrifugal action in two portions, which contain different proportions of impurities: (A)



that from the washing tank (*W*) and drying press and (*B*) that from the flushing of *W* and the decantation tank. *A* is maintained at sufficient concn. for re-use by recuperation of *B* after purification. F. R. E.

**Cleansing of raw or manufactured wool.** J. F. PATERSON (B.P. 399,599, 23.5.32).—Wool is dusted with finely-divided dry raw gypsum and mixed in a revolving drum. It is then discharged on to a continuously moving, vibratory, perforated belt or conveyor to separate part of the powder and dirt, and thence to a conduit where it is subjected to suctional pressure to remove the residual impurities. F. R. E.

**Cleaning of wool and other fibres.** J. T. SHEVLIN. From FITGER CALIFORNIA Co. (B.P. 398,683, 26.11.32).—Unscoured and/or unwashed fleece and pulled wools etc. are cooled to congeal the grease therein and then mechanically cleaned while at the reduced temp. F. R. E.

**Reeling-off silk cocoons.** B. LOEWE (U.S.P. 1,899,513, 28.2.33. Appl., 11.12.30. Ger., 10.6.29).—The cocoons are pretreated with an aq. solution (<0.5%) of  $\text{NH}_3$  or an  $\text{NH}_2$ -compound ( $\text{NH}_2\text{Me}$ ) and a salt having a catalytic action ( $\text{NaOAc}$ ,  $\text{HCO}_2\text{NH}_4$ ), until all the silk layers are penetrated, washed with dil. acid (0.5%  $\text{HCO}_2\text{H}$ , 0.25%  $\text{HCl}$ ), and treated at room temp. in one or more baths of dil.  $\text{NaOH}$  ( $d < 1.014$ ) for a total period of  $\leq 1$  min. in vac. They are then freed from absorbed solutions in vac. and reeled from  $\text{H}_2\text{O}$  at room temp. To increase the coherence and elasticity of the filaments, emulsions of sulphonated castor oil or fish glue with  $\text{NaHCO}_3$ , sugar, or  $\text{NH}_3$  may be added to the treating liquids or to the  $\text{H}_2\text{O}$ . F. R. E.

**Threads and like filaments for manufacture of textile-like products, particularly fabrics.** D. M. LIVSEY. From SIDAC SOC. INDUSTRI. DE LA CELLULOSE, SOC. ANON. (B.P. 398,427, 13.3.33).—Bands of cellulose film are converted into threads suitable for the manufacture of strong and lustrous dirtproof fabrics by compressing them in the direction of their width and loosely twisting them about each other or about added threads of other material (*e.g.*, metal). A. J. H.

**Production of wood cellulose comparable to cotton, with high content of alkali-resisting cellulose.** F. C. and F. PALAZZO (B.P. 398,730, 16.2.33. Italy, 24.12.32).—Sulphite-cellulose is heated at 1–2 atm. for 1–2 hr. with a liquor containing 1–1.5% of  $\text{NaOH}$  and 0.2–0.3% of an alkali-resisting emulsoid (soap) and, after washing, is brought to a concn. of 5–6% and bleached first with a feebly alkaline solution of  $\text{NaOCl}$ ,  $\text{Ca}(\text{OCl})_2$ , or  $\text{Na}_2\text{O}_2$  and then with feebly acid  $\text{HOCl}$  or with  $\text{KMnO}_4$  solution followed by dil.  $\text{H}_2\text{SO}_3$ . The material is further treated with a hot solution containing 1% of  $\text{Na}_2\text{SO}_3$  and 0.25% of soap, then with cold dil.  $\text{H}_2\text{SO}_3$ , and is finally washed. F. R. E.

[Treatment of] **cellulosic materials [for production of organic cellulose derivatives]**. BRIT. CELANESE, LTD., J. E. JONES, and S. C. BATE (B.P. 399,508, 5.4.32).—Cotton, purified wood pulp, etc. is pretreated at  $\leq$  room temp. and atm. pressure with  $\leq 40\%$  of  $\text{SO}_2$  in the gaseous or liquid form, in presence of a lower fatty acid ( $\text{AcOH}$ ) if desired. Esterification

or etherification is carried out in the ordinary way, with or without separation of the pretreatment liquid.

F. R. E.

**Manufacture of cellulose esters.** KODAK, LTD., Assees. of H. T. CLARKE and C. J. MALM (B.P. 397,906, 2.3.32. U.S., 4.3.31).—The methoxy-, ethoxy-, and propoxy-fatty anhydrides are used as impelling agents (*cf.* B.P. 287,880; B., 1930, 279) for introducing acyl radicals from unsubstituted aliphatic (including cycloparaffinic) or aromatic (alkylated, if desired) monocarboxylic acids into the cellulose mol. These anhydrides and their corresponding acids are good solvents for cellulose esters and also for the higher fatty acids, thus facilitating dope esterification. Examples are: the prep. of the higher fatty acid esters of cellulose (stearate, laurate, palmitate, etc.) containing varying quantities of  $\text{OAc}$ , by using an esterifying bath containing ethoxyacetic anhydride, a higher fatty acid (stearic), and a mild catalyst [ $\text{Mg}(\text{ClO}_4)_2$ ]. D. J. N.

**Manufacture of cellulose esters.** H. DREYFUS (B.P. 399,509, 5.4.32).—After pretreatment with a lower fatty acid ( $\text{AcOH}$ ), cellulosic material is esterified at  $< 50^\circ$  with the usual esterifying agents, in presence of 5–15 wt.-% of a hydrohalide acid ( $\text{HCl}$ ) and a hydrohalide of a N-containing base ( $\text{NH}_4\text{Cl}$ ,  $\text{NH}_2\text{Et}_2\text{Cl}$ ) as catalyst, and the product is ripened in the normal manner. F. R. E.

**Treatment of mixed organic esters of cellulose.** KODAK, LTD., Assees. of C. J. MALM and C. L. FLETCHER (B.P. 398,626, 18.7.32. U.S., 17.7.31).— $\text{COMe}_2$ -sol. mixed esters of cellulose containing  $\leq 15\%$  of acyl radical other than  $\text{Ac}$  are hydrolysed with  $\text{H}_2\text{O}$ , an org. acid ( $\text{AcOH}$ ), and a catalyst ( $\text{H}_2\text{SO}_4$ ,  $\text{H}_2\text{SO}_4 + \text{H}_3\text{PO}_4$ ,  $\text{NaHSO}_3$ ,  $\text{PhSO}_3\text{H}$ , etc.) at  $38^\circ$  for 1–360 hr. Hydrolysis is continued for a predetermined period until the colloided product attains the desired pptn. val., flexibility, and solubility characteristics. F. R. E.

**Cellulosic composition containing paraldehyde.** S. J. CARROLL, Assr. to EASTMAN KODAK Co. (U.S.P. 1,899,078, 28.2.33. Appl., 23.9.29).—10–50 pts. of paraldehyde are added as plasticiser to 100 pts. of cellulose ester (acetate) or ether in a suitable solvent, *e.g.*,  $\text{COMe}_2$ ; films, filaments, etc. produced therefrom possess high elasticity and low inflammability. F. R. E.

**Transfer and transfer composition.** W. S. LAWRENCE, Assr. to KAUMAGRAPH Co. (U.S.P. 1,899,420, 28.2.33. Appl., 26.6.28).—A mixture of ethyl- or benzyl-cellulose with smaller amounts of resinous material, a plasticiser [ $(\text{C}_6\text{H}_4\text{Me})_3\text{PO}_4$ ,  $\text{Et}$  abietate], blown castor oil, and a pigment in a suitable solvent is applied to a paper base. The marking is fusible without decomp., and is transferable to fabrics etc. by dry heat. F. R. E.

**Manufacture of artificial filaments and similar materials.** BRIT. CELANESE, LTD., and (A) E. KINSELLA, (B) W. I. TAYLOR (B.P. 398,478 and 399,822, [A, B] 12.3.32, [B] 4.4.32).—(A) During the dry-spinning of org. derivatives of cellulose, the temp. of the dope is controlled immediately prior to extrusion by surrounding the filter candles or other supply means adjacent to the spinning orifices with attemperating members, *e.g.*,



jackets or coils, each of which is removable without interrupting the flow of liquid ( $H_2O$ ) which is circulated through them in a uniform direction from common supply and discharge conduits at the desired temp. (B) The supply pipe ( $P$ ) or other means of supply ( $S$ ) for the spinning solution is passed through a zone of evaporative atm. which is kept at a temp. above that into which the filaments are extruded. For this purpose, the two zones are separated by insulating shields, whilst the air in the neighbourhood of  $P$  may be heated, cool air from outside may be introduced into the spinning zone, or convection currents of hot air produced in the evaporative medium may be directed away from the jet towards  $S$ .

F. R. E.

**Production of artificial straws or like materials.** BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 399,811, 7.4.32).—A no. of continuous artificial filaments of org. derivatives of cellulose are brought together continuously with their production in the form of a flattened thread of individual filaments, which are then united, while under tension, by treatment with a solvent, restricted solvent, or softening agent.

F. R. E.

**Production of artificial silk without employment of individual spinning pumps.** F. NINNELT (B.P. 399,920, 23.5.32. Ger., 29.5.31).—Spinning is carried out by maintaining the pressure in the main conduit higher than the spinning pressure; means for maintaining a const. supply feed, even during pressure fluctuations, are described.

F. R. E.

**Production of artificial silk and like products.** BRIT. CELANESE, LTD., R. P. ROBERTS, and E. B. JOHNSON (B.P. 399,795, 4.4.32).—In the dry-spinning of org. derivatives of cellulose by means of a countercurrent of evaporative medium ( $A$ ), an additional current ( $B$ ), induced by heating means within the spinning cell and composed of external air with a large regulated proportion of  $A$ , is directed in the vicinity of the jet on to the filaments at different points along their path.  $B$ , which is cooler than  $A$ , is prevented by a suitable baffle from entering the zone surrounding the supply pipe and/or filter where the temp. is  $>$  that around the jet.

F. R. E.

**Production of [viscose] artificial silk of reduced lustre.** F. D. LEWIS (B.P. 399,512, 6.4., 20.7., and 5.12.32).—Halogenated, e.g., penta- and/or hexa-chloro-, naphthalenes are dispersed in the viscose spinning solution. Moderately volatile solvents, e.g.,  $1-C_{10}H_7Cl$ ,  $(C_2H_4Cl)_2O$ , glycerol ditolyl ether, may be used, with or without deodorisers.

C. H.

**Treatment [desulphurisation] of artificial materials obtained from viscose.** SOIE ARTIF. DE GAND SOC. ANON., "SARGA," and M. G. LUFT (B.P. 400,035, 18.1.33. Belg., 18.1.32).—To the treatment bath containing alkali sulphite or  $SO_2$  is added a small quantity ( $< 1\%$ ) of an  $NH_4$  base or of an alkali or alkaline-earth carbonate or bicarbonate, the alkaline earths being kept in solution by excess  $CO_2$ , in order to neutralise traces of acids resulting from the coagulating process without causing deterioration of the cellulose.

F. R. E.

**Manufacture [after-treatment] of artificial silk.** I. G. FARBENIND. A.-G. (B.P. 398,738, 2.3.33. Ger.

2.3. and 30.7.32).—Washed and dried filaments are run from the spinning bobbins on to twisting bobbins ( $B$ ) the diam. of which may be varied;  $B$  are covered with a permeable, partly permeable, or impermeable cover. After diminution of the bobbin diam., the loosened lap of threads is removed with the cover, wrapped therein, and subjected to an irrigation treatment with suitable liquids.

F. R. E.

**Production of crêpe yarns and fabrics.** BRIT. CELANESE, LTD., W. A. DICKIE, and W. I. TAYLOR (B.P. 399,534, 30.3.32).—A yarn composed of unhydrolysed cellulose ester filaments (25–75%) doubled with others of the partly hydrolysed esters (75–25%), the degree of hydrolysis being equiv. to a loss in wt. of 10–30% on cellulose acetate, is highly twisted in stages with intermediate setting by wetting, steaming, etc. The twisted yarn is woven into fabric which is afterwards scoured.

F. R. E.

**Production of paper-like sheets of fibrous material for writing or printing upon.** WEBERER EBNAT A.-G., and TEXTILWERK HORN A.-G. (B.P. 399,677, 12.12.32. Switz., 31.12.31).—A cellulose woven or knitted fabric, preferably of cotton, is immersed in aq.  $NaOH$  of mercerising concn. until max. shrinkage occurs; it is then calendered, treated with a swelling agent ( $H_2SO_4$ ,  $d$  1.63), washed, impregnated with size (resin soap), and finally hot-calendered. The product, which is durable and waterproof, can be ironed to remove creases and is suitable for permanent written or printed records.

F. R. E.

**Waterproofing of paper. Fibrous product. Waterproofing of fibrous products.** G. J. MANSON, ASSR. to MANSON CHEM. CO. (U.S.P. [A, B] 1,894,566—7 and [c] 1,894,959, [A, B] 17.1.33, [c] 24.1.33. Appl., [A—c] 25.4.27).—Wax emulsions of the type described in U.S.P. 1,800,551 (B., 1932, 18), in which the wax particles are coated with (A) a finely-divided ppt., e.g., from interaction of  $MgCl_2$  and  $Na_2CO_3$ , or (c) a gel, e.g., from interaction of Na silicate and  $MgSO_4$ , are used for preparing waterproof products, e.g., paper. (B) claims the products made by the process of (c). (Cf. U.S.P. 1,882,212; B., 1933, 700.)

D. J. N.

**Production of yarns.** BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 399,838, 13.4.32).

**Production of moulded laminated [resin-impregnated] paper products.** BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. of R. E. COLEMAN (B.P. 398,618, 21.6.32. U.S., 23.6.31).

**Recovering fibres from waste rubber.**—See XIV. Depilatory.—See XXIII.

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

**[Dyeing of] vat dyes on regenerated cellulose artificial silks.** W. M. TODD (J. Soc. Dyers and Col., 1933, 49, 312—316).—The satisfactory dyeing of cellulose rayon is favoured by using a dyebath at 35–45° containing a retarding agent (e.g., 1 g. of glue or Protectol per litre) and those vat dyes which exhaust slowly and at the same rate. The relative rates of exhaustion of vat dyes with cotton and cellulose silks are about equal; exhaustion data are given for 26 Caledon dyes.



Certain combinations of vat dyes, *e.g.*, Caledon Yellow 5G and Jade Green XS, and Caledon Brilliant Violet RS and Blue RC, do not yield satisfactory dyeings. In dyeing cotton and cellulose silk mixtures, the distribution of the vat dye between the two fibres is mainly determined by the temp. of the dyebath and but little by its alkali content; a low dyeing temp. assists absorption of dye by the cotton, whereas a high temp. assists the cellulose silk. Even distribution is definitely favoured by the presence in the dyebath of a retarding agent, *e.g.*, Permalin W (I.C.I.). The fastness properties, except as regards light, of vat dyes are similar on both cotton and cellulose silks; light-fastness is generally higher on a cellulose silk. In dyeing with combinations of vat dyes the use of hard H<sub>2</sub>O may cause uneven results unless Lissapol A (I.C.I.) is present. A. J. H.

**Measurement of the fastness [of dyed materials] to light.** G. MARTIN, J. NIEDERHAUSER, J. PINTE, and R. TOUSSAINT (Rev. Gén. Mat. Col., 1933, 37, 281—283).—Examination, by the method described previously (B., 1933, 745), of the fastness of the standard dyes (recommended by the German Fastness Commission) Brilliant Wool Blue FFR extra, Wool Blue N extra, Brilliant Indocyanine 6B, Wool Fast Blue GL, Cyananthrol RX, Alizarin Direct Blue A2G, Indigosol AZG, and Indigosol Blue AGG, shows that they are not reliable unless the period of exposure is  $\geq$  4000 "lux-heures" (l.-h.) (= 100 hr. of bright sunshine at Paris). Curves are given showing the progressive fading of these dyes during 8000 l.-h.; the degrees of fading (after 1000 l.-h.) were 675, 425, 310, 150, 50, 40, 25, and 12, respectively. It is suggested that the German standards really relate to particular dyeings rather than to colours, and that in determining the fastness of a coloured material it is necessary to expose beside it patterns dyed with the above standard dyes. The difficulty of comparing faded patterns not having the same tone or depth of shade is emphasised. A. J. H.

**Action of electrolytes on wool fibre.** D. A. WILSON and N. E. GORDON (J. Amer. Chem. Soc., 1933, 55, 3896—3897).—Whereas pure wool gives a very indistinct X-ray diagram, fibres treated with electrolytes, especially of an acidic nature, or dyed industrially, afford rings corresponding with the spacings 4.08 and 3.72 Å. The phenomenon may be due to a partial crystallisation of the wool protein, the crystals being unoriented along the fibre axis. J. G. A. G.

**Propylene dichloride mixture controls clothes moths, and carpet beetles in rugs and over-stuffed furniture.** E. I. McDANIEL (Mich. Agric. Exp. Sta. Quart. Bull., 1933, 16, 13—15).—Successful results were obtained by fumigation with a 9:1 mixture of C<sub>3</sub>H<sub>6</sub>Cl<sub>2</sub> and CCl<sub>4</sub> for 48 hr. A. G. P.

**Estimation of tautness of doped fabrics.** G. M. KLINE (Amer. Paint & Varnish Manufs.' Assoc., Aug., 1933, Circ. No. 443, 266—273).—The tautness of fabric doped with cellulose nitrate and acetate, vinyl chloride and acetate resins, PhOH-CH<sub>2</sub>O and chlorodiphenyl resins was determined by measuring (a) the linear deflection under a definite load with a dial micrometer (McGowan), (b) the rebound of a dropped steel ball, (c) from

the pitch of the note emitted on tapping the fabric, (d) with the "hardness rocker" (B., 1932, 867). The results (tabulated) are not concordant and method (a) is recommended and described. S. M.

**Treatment of H<sub>2</sub>O.**—See XXIII.

#### PATENTS.

**Dyeing and other treatment baths for textile materials made of or containing animal fibres.** SOC. CHEM. IND. IN BASLE (B.P. 400,031, 12.1.33. Switz., 16.1.32).—Sulphonated condensation products from benzoic and phenols are added to dye-baths etc. to diminish the affinity of dyes for wool, *e.g.*, in tone-dyeing, reserving, and levelling, and to prevent bleeding during acid fulling. Suitable phenolic compounds for condensation are Schäffer acid, R-acid, phenol-*m*-sulphonic acid, etc. C. H.

**Dyeing [of fabrics].** G. E. BURGESS, BURGESS, LEDWARD & Co., LTD., and BROWN & ADAM, LTD. (B.P. 398,887, 24.3.32. Ger., 25.3.31).—Fabric in rope form is dyed in an Obermaier type of dyeing machine; excellent penetration and levelling are thereby obtained. In dyeing with vat dyes it is desirable to circulate the liquor outwardly through the fabric. A. J. H.

**Coloration of cellulose ester textiles.** C. S. BEDFORD (B.P. 399,533, 29.3.33).—Sulphonated CH<sub>2</sub>O-cresol condensation products are used to disperse insol. dyes for acetate silk etc. C. H.

**Coloration of materials made of or containing cellulose esters or ethers [to give reserve effects].** BRIT. CELANESE, LTD., G. H. ELLIS, and H. C. OLPIN (B.P. 399,559, 9.4.32).—The material is printed with an inorg. mild reducing agent (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> or SnCl<sub>2</sub>) and a suitable dye (2:4-dinitro-4'-aminodiphenylamine, 1:4-diaminoanthraquinone, *p*-aminophenol → 1-phenyl-3-methyl-5-pyrazolone, Duranol-blue G), padded with aniline-black liquor (or other oxidation base), and oxidised. C. H.

**[Sizing and tinting] treatment of [artificial silk] textile threads.** W. H. CHARCH, ASSR. to DU PONT RAYON Co. (U.S.P. 1,902,833, 28.3.33. Appl., 23.4.29).—The processes are effected simultaneously by impregnation with a composition containing a suspended or dissolved dye (Me-violet base); oil-sol. dyes are used when the composition contains lubricating oils. A. J. H.

**Treating [restoring the colour to] artificial grass.** E. H. HAABESTAD, ASSR. to HYDROL CHEM. Co., INC. (U.S.P. 1,897,900, 14.2.33. Appl., 12.11.30).—Shredded palm leaves (artificial grass) are sprayed with a MeOH or EtOH solution of malachite-green (shaded with auramine) containing a hygroscopic salt (CaCl<sub>2</sub>, MgCl<sub>2</sub>) and a lustre-restoring agent (Turkey-red oil). A. J. H.

**Treatment of cellulosic fibres [for modifying their lustre and dyeing properties].** E. STOCKER, ASSR. to MUNITEX CORP. (U.S.P. 1,895,298, 24.1.33. Appl., 29.1.30. Ger., 20.2.29).—Uniform esterification without excessive shrinkage is obtained by treating the fibres with a caustic alkali at a high temp. (< 100°)



before application of the usual esterifying agents, *e.g.*, BzCl.  
A. J. H.

**Discharging preparation.** G. REDDELIEN, H. LANGE, A. FISCHER, and H. FREUND, Asss. to GEN. ANILINE WORKS, INC. (U.S.P. 1,889,154, 29.11.32. Appl., 18.5.31. Ger., 16.4.30).—The use of phenyl- $\alpha$ -naphthylmethyl-dialkyl (dimethyl) ammonium salts and their sulphonated derivatives in discharging pastes (containing a reducing agent), particularly for resistant vat dyes (*e.g.*, Indanthrene, Helindone, Algol, and Hydron types) is claimed.  
H. A. P.

**Colour-discharge printing.** F. R. BYROD, Assr. to SUSQUEHANNA SILK MILLS (U.S.P. 1,864,582, 28.6.32. Appl., 10.12.31).—Finely-divided  $\text{TiO}_2$ , with or without ZnO, is added to the discharge colour paste to protect the unreduced colour.  
C. H.

**Wetting agents [for mercerising liquors].** RÜTGERSWERKE A.-G., and L. KAHL (B.P. 400,135, 9.6.33. Ger., 28.7.32).—*iso*Amyl alcohol, preferably  $\geq 2\%$ , is mixed with PhOH, cresols, and/or xylenols poor in or free from *o*-cresol.  
C. H.

**[Manufacture of] wetting agents and treatment of textile fibres therewith.** IMPERIAL CHEM. INDUSTRIES, LTD., F. SCHOLEFIELD, W. F. A. ERMEN, and W. TODD (B.P. 399,537, 1.4.32).—A product (A) of B.P. 274,611 or 311,885 (B., 1927, 841; 1929, 639) is mixed with pine oil or terpineol or similar terpene alcohol (B) and  $\text{H}_2\text{O}$  (C), the % proportions of A : B : C being 40 : 55 : 40—55 : 9—18 (41 : 2 : 45 : 6 : 13 : 2).  
C. H.

**Purification of liquids which have been used for washing textile materials.** E. C. DUHAMEL, and COMP. GÉN. DES IND. TEXTILES (B.P. 399,826, 7.4.32. Fr., 7.4.31).—Spent liquors from textile washing processes (suint liquors) are regenerated by exposure to a vac. which de-aerates them and facilitates settling of suspended particles. The process is aided by keeping for a time, by centrifuging, or by addition of mineral acid (insufficient to set free all org. acids present) prior to vac. treatment; in the last case the liquors are subsequently neutralised.  
H. A. P.

**Treatment of yarns.** O. TUDOR-HART (B.P. 399,800, 11.1.32).—Coir yarn is boiled with dil. alkali (1.25%, followed by a  $\text{Na}_2\text{CO}_3$ -sulphonated soap bath), and then subjected to rubbing processes to confer lustre. Even penetration of dyes is claimed.  
H. A. P.

**[Delustring of cellulose ester or ether] textile materials.** BRIT. CELANESE, LTD. (B.P. 398,371, 1.12.32. U.S., 1.12.31).—The tendency of oil-stained cellulose acetate (etc.) silk materials to delustre unevenly in boiling soap baths is removed by pre-soaking them in an aq. or org. liquor containing an emulsified free higher fatty acid above  $\text{C}_7$  (*e.g.*, fatty acids derived from coconut oil, and stearic, oleic, and palmitic acids).  
A. J. H.

(A) [Swelling] treatment of silk. (B) Production of effects on silk. HEBERLEIN & Co. A.-G. (B.P. 397,878 and 397,838, [A] 23.11.31, [B] 25.11.31. Ger., [A] 24.11.30, [B] 26.10.30).—(A) An increase in transparency accompanied by a stiffening, an effect on lustre, and a modification of the dyeing properties of silk are obtained by application (with subsequent removal) of a swelling agent (*e.g.*, conc. HCl,  $\text{H}_2\text{SO}_4$ , NaOH, aq.  $\text{ZnCl}_2$ , and

cuprammonium solution) such that a permanent swollen condition is produced without deleterious effect on the silk. [Stat. ref.] (B) Hydrolysis of the silk is prevented in the treatments described in (A) by adding to the acid swelling agents a protective substance such as amyl alcohol, glycerol,  $\text{C}_5\text{H}_5\text{N}$ , and  $\text{CH}_2\text{O}$ .  
A. J. H.

**Sizing of textile fibres.** I. G. FARBENIND. A.-G. (B.P. 399,905, 6.5.32. Ger., 16.5.31. Cf. B.P. 345,207; B., 1931, 535).—A mixture of  $\leq 2$  polyvinyl alcohols of widely differing  $\eta$  is used in aq. solution. Any or all of these may be chemically modified providing that  $\text{H}_2\text{O}$ -solubility is not destroyed and a difference in  $\eta$  is maintained, *e.g.*, by esterification, etherification, or interpolymerisation. Examples of modified  $(\text{CH}_2\text{:CH}\cdot\text{OH})_n$  include the laurate, mixed acetate and chloroacetate,  $(\text{CH}_2)_2\text{O}$  derivatives, and a product obtained by alkaline hydrolysis of the interpolymerisation product of  $\text{CH}_2\text{:CHCl}$  and maleic anhydride.  
H. A. P.

**Scrubbing, impregnating, sizing, starching, or other like machines for treating fabrics with liquids.** J. H., D. G., and K. BAYLISS (B.P. 399,006, 25.8.32).—A wet-brushing machine is described.  
A. J. H.

**Making of flexible treated [electrical insulating] material.** B. H. F. RICHARDS and A. H. HAROLDSON, Asss. to CONTINENTAL DIAMOND FIBRE CO. (U.S.P. 1,897,651, 14.2.33. Appl., 17.3.30).—Crêped paper or similar material is impregnated with a PhOH- $\text{CH}_2\text{O}$  resin and the whole subjected to heat and pressure.  
A. J. H.

**Treatment of textile materials [to render them flexible and fireproof].** BRIT. THOMSON-HOUSTON CO., LTD., Asses. of H. W. TURNER (B.P. 398,909, 25.3.33. U.S., 25.3.32).—Electrical insulation fabric is singed, kneaded with a powdered filler (*F*) (*e.g.*, talc or soapstone) to render it airtight and substantially non-inflammable, impregnated with a de-gelled oil (*e.g.*, linseed oil rendered temporarily fluid by heating), dried, and again kneaded with *F*.  
A. J. H.

**Treatment of textile materials [with synthetic resins].** BRIT. CELANESE, LTD. (B.P. 397,711, 7.11.32. U.S., 7.11.31).—Cellulose acetate (etc.) fabric is made more durable and less subject to laddering and unravelling at its cut edges by impregnation with 0.25—5% of a polymerised vinyl compound.  
A. J. H.

**Treatment of cellulose fibres and fabrics.** A. C. THAYSEN (B.P. 399,952, 23.7. and 19.10.32).—Cotton, linen, hemp, etc. in the raw or manufactured state, after pretreatment with glacial AcOH,  $\text{CO}_2$ , boiling  $\text{H}_2\text{O}$ , or aq. NaOH, is partly acetylated with 15 times its wt. of a mixture of  $\text{Ac}_2\text{O}$  ( $\geq 10\%$ ) and AcOH with  $\text{HClO}_4$  ( $\geq 0.25\%$ ) as catalyst, the reaction being arrested before the increase in wt. is  $> 18\%$ . In this way the material is rendered immune from deterioration by destructive organisms without materially changing the normal textile properties, *e.g.*, tensile strength.  
F. R. E.

**Cryst. Al salts. Preservative against moths.**—See III. Transfer. Paper-like sheets.—See V. Finishings.—See XIII. Rubber-impregnated products.—See XIV. Enzyme preps.—See XVIII.



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

**Hydrofluoric acid and the toxic fog which occurred in the Meuse valley in 1930.** J. MAGE and G. BATA (*Chim. et Ind.*, 1933, 30, 787—788).—The official committee of enquiry into this matter considered that the fatalities were not due to F compounds arising from the one superphosphate works in the district for the following reasons: (1) the works was operating at < 25% capacity at the time, (2) only 10—20% of the total F present passed into the atm.; (3) meteorological considerations, and the absence of any centering of fatalities around the works in question. The total emission of F compounds was calc. to be 200 kg. per day, against 70 tons of S compounds in the area. Calculations suggest that the SO<sub>3</sub> content of the fog might well have been much > the lower toxic limit. C. I.

**Solubility of phosphates.** J. D'ANS, F. POHLE, and W. SCHUPPE (*Z. Pflanz. Düng.*, 1933, 32, A, 70—84).—Data for a no. of phosphatic materials are given. Variations in solubility resulting from differences in the ratio wt. of sample: vol. of citric acid used for extraction are not of the same kind for all substances examined. The total P dissolved by repeated extraction is also influenced by the ratio adopted. The citric acid solubility and, to a similar though smaller degree, the NH<sub>4</sub> citrate solubility of phosphates is reduced by additions of CaO, CaCO<sub>3</sub>, and K<sub>2</sub>CO<sub>3</sub> to extents which decrease in the order named. The above variations in PO<sub>4</sub> solubility are accompanied by similar changes in the amount of Ca<sup>++</sup> extracted. A. G. P.

**Iodine content of iodised common salt.** G. PRANGE (*Z. Unters. Lebensm.*, 1933, 66, 369—374; cf. B., 1931, 1048).—12 samples of the commercial salt (I) contained 0.4—20.3 mg. KI per kg.; 4—22% of KI was lost during 10—12 months' storage. It is suggested that official supervision of (I) is required, such salt to contain 5 mg. ± 10% of KI per kg. and to be marked with the date of prep. E. C. S.

**Intensive drying of cylinder oxygen with silica gel.** F. B. KRULL (*Z. kompr. fluss. Gase*, 1933, 30, 1—3; *Chem. Zentr.*, 1933, i, 3906).—H<sub>2</sub>O is removed in high-pressure absorbers with SiO<sub>2</sub> gel which is regenerated with warm N<sub>2</sub>. 2—3 mg. H<sub>2</sub>O per cu. m. remain. A. A. E.

**Reactions of sulphur in air at low temperatures.** R. DUBRISAY (*Chim. et Ind.*, 1933, 30, 767—769).—The action of S on Ag, Cu, and Hg takes place at room temp. if the metals are merely placed near it without being in actual contact. This arises from the low but appreciable v.p. of S, and it is possible that a similar cause can be assigned to the fungicidal action of S. Elementary S in the soil is readily oxidised by bacteria. Flowers of S suspended in H<sub>2</sub>O are also oxidised by the action of ultra-violet light, the products being H<sub>2</sub>S<sub>3</sub>O<sub>6</sub> and H<sub>2</sub>SO<sub>4</sub>. The reaction with ozonised O<sub>2</sub> is much slower and no H<sub>2</sub>S<sub>3</sub>O<sub>6</sub> is produced. Ultra-violet light produces a similar effect in alkaline solution. C. I.

**Determination of small amounts of selenium in pyrites.** K. BRÜCKNER (*Z. anal. Chem.*, 1933, 94, 305—322).—Sources of error and variability in the

usual methods are examined, and a modified procedure is advanced. The rock is digested with HNO<sub>3</sub>, and the solution evaporated down with H<sub>2</sub>SO<sub>4</sub>. The solution is diluted, HCl added, and Se pptd. by SO<sub>2</sub> + N<sub>2</sub>H<sub>4</sub>. J. S. A.

**Slag glass.**—See VIII. **Checking caustic corrosion.** **Analysis of Ni-plating salts.**—See X.

See also A., Nov., 1115, **Prep. of Ag and S sols.** 1129, **"Glacial metaphosphoric acid."** 1130, **Prep. of SO.** 1132, **Iodimetry.** 1142, **Prep. of Ti K oxalate.**

### PATENTS.

**Manufacture of sulphuric acid by the contact process.** METALLGES. A.-G. (B.P. 400,143, 26.6.33. Ger., 4.8.32).—Circulation of conc. H<sub>2</sub>SO<sub>4</sub> is dispensed with in apparatus in which contact masses of low sensitivity, e.g., V, are used; e.g., a shaft packed with quartz is used for filtering the burner gases and a bell absorber for absorbing the SO<sub>3</sub>. L. A. C.

**Manufacture of alkali cyanates.** N. V. STIKSTOF-BINDINGSIND. "NEDERLAND," Assecs. of N. RUSTING (B.P. 399,820, 8.2.32. Holl., 9.2.31).—Alkali salts, e.g., Na<sub>2</sub>CO<sub>3</sub>, Na<sub>2</sub>S, Na<sub>2</sub>SO<sub>3</sub>, Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, with the addition of CaO if desired, are treated with excess CO<sub>2</sub> and/or COS and NH<sub>3</sub> in presence of a gas (other than a hydrocarbon) capable of decomposing H<sub>2</sub>O (preferably CO) at a temp. < 800° but high enough for the decomp. of H<sub>2</sub>O by the CO. The reaction may be effected under pressure or vac. and in presence of non-reacting solids and gases. L. A. C.

**Sintered magnesia and refractory materials containing it.** ALTERRA A.-G. (B.P. 399,126, 23.4.32. Austr., 25.4.31. Cf. B.P. 394,115; B., 1933, 703).—Ferromagnetic MgO, Fe<sub>2</sub>O<sub>3</sub> is added to the MgCO<sub>3</sub> charge as a flux before dead-burning. A. R. P.

**Cryst. Al salts. Detecting gases containing Cl<sub>2</sub>.**—See III. **Purifying roaster gases.**—See X.

## VIII.—GLASS; CERAMICS.

**Constitution of glass. II. Theoretical basis of glass coloration.** W. WEYL and E. THÜMEN (*Glastech. Ber.*, 1933, 11, 113—120; *Chem. Zentr.*, 1933, i, 3613—3614).—Colour changes in Co and Ni glasses are attributed to dissociation-association equilibria; red Co and yellow Ni colours are due to co-ordinatively saturated mols., whilst dark blue Co glasses are due to co-ordinatively unsaturated mols. Experimental evidence for these conclusions is adduced. A. A. E.

**Slag nephelite glass.** A. N. DAUVALTER (*Keram. i Steklo*, 1933, 9, No. 3, 26—27).—The slag (SiO<sub>2</sub> 53, CaO 36%) obtained in the production of P afforded a glass which melted easily, was easily worked and purified, and had no tendency to devitrification. CH. ABS.

**Test for chemical resistance of glass containers.** F. C. FLINT and A. K. LYLE (*Bull. Amer. Ceram. Soc.*, 1933, 12, 296—299).—The bottle, filled with distilled H<sub>2</sub>O, is autoclaved at 138° (35 lb./sq. in.) for 6 hr. and the alkali liberated is titrated (Me-red). The total alkali is a measure of the durability of the glass itself; the alkali, expressed as g. per litre (influenced by the



“shape factor”), indicates the efficiency of the particular bottle as a container. J. A. S.

**Effects of particle size of a potter's “flint” and a felspar in whiteware.** R. F. GELLER, D. N. EVANS, and A. S. CREAMER (Bur. Stand. J. Res., 1933, 11, 327—340).—A no. of bodies containing combinations of flint and felspar fractions ranging from 10 to 75  $\mu$  were fired at 1150°, 1200°, 1250°, 1300°, and 1350°. Tests of shrinkage, porosity, bending strength, and thermal expansion were made. The degree of vitrification and the relative fusion and dissolution in the body were estimated microscopically and compared with the sp. surfaces of the flint and felspar. Whiteware bodies containing more finely-ground materials may be vitrified without auxiliary fluxes at temp. significantly below those at present used in the industry. J. A. S.

**Relation between particle size and duration of melting or sintering of a ceramic mass.** E. HERLINGER and P. BECK (Sprechsaal Keram., 1933, 66, 145—147; Chem. Zentr., 1933, i, 3483).—The diminution in vol. of a melting cube varies as the third power of the time. The time of melting is proportional to the size of the original grains. If the time of complete fusion for a certain particle size is known, under certain conditions the time for partial melting and for other particle sizes ( $10^{-6}$  to  $10^{-1}$  cm.) can be calc. A. A. E.

**Reducing firing of earthenware.** A. DAHL (Tonind.-Ztg., 1933, 57, 323—324; Chem. Zentr., 1933, i, 3483).—After sintering has begun, C cannot be burned out of the pores. It is doubtful whether bodies containing C have increased strength. A sintered product of good quality can be produced in a neutral or slightly reducing or oxidising atm. A. A. E.

**Discoloration of refractories by iron.** J. W. MELLOR (Trans. Ceram. Soc., 1933, 32, 403—414).—The thermal decomp. of clays is described, and explanations are given of the red and buff coring of bricks. With clays  $\text{Fe}_2\text{O}_3$  (I), produced under oxidising conditions, gives a brownish-red, and, formed under reducing conditions, the lower oxides  $\text{FeO}$  and  $\text{Fe}_3\text{O}_4$  (II), a greenish-blue colour. (I), but not (II), forms with  $\text{Al}_2\text{O}_3$  (III) [but less readily with (III) preheated at  $> 700^\circ$  (polymerised)] a solid solution (IV), of a buff colour in fireclays, and paler than a mixture of the same proportions of (I) and (III); in (IV), (I) has greater thermal stability. Buff bricks with red cores originate when, during burning, oxidation of (II) in the core is delayed until after (III) has polymerised; (I) is finally produced, not in solution with (III), and exerts its normal red colour. Where the proportion of (III) is relatively low, (IV) may break down under oxidising conditions at high temp.; this action, starting at the surface and extending inwards, gives a red-crust, buff-cored brick. Bricks in which both these processes occur simultaneously have red crusts and cores, and intermediate buff zones. White bands encircling the black cores of under-burnt bricks result from the mutual bleaching of the almost complementary colours of (I) and (II).  $\text{CaO}$  bleaches  $\text{Fe}$  oxide, but if, during the burning of  $\text{CaO}$ -bonded  $\text{SiO}_2$  bricks and calcareous building bricks,  $\text{Ca}$  and  $\text{Fe}$  sulphates, decomposing only at relatively high temp., are formed on the surface and

prevent the formation of pale-coloured  $\text{Ca}$  ferrite, surface discolorations of (I) may be produced by the subsequent decomp. of  $\text{FeSO}_4$ . A. L. R.

**Refractory materials for melting pure metals.** H. B. WAHLIN, O. D. FRITSCHKE, and J. F. OESTERLE (Physical Rev., 1932, [ii], 42, 911).—When heated in a vac. porcelain and  $\text{MgO}$  crucibles volatilise and are reduced sufficiently to contaminate pure metals contained in them. Crucibles of  $\text{ThO}_2$ , fired at  $1800^\circ$ , are the most satisfactory and withstand prolonged heating in a vac. The formation of  $\text{ThC}_2$  which, owing to the action of atm.  $\text{H}_2\text{O}$  disintegrates the crucibles, is deleterious. L. S. T.

**Determining moisture.**—See I. Cr deposits on glass.—See X.

See also A., Nov., 1135, Cu-glass seals.

#### PATENTS.

**Working of glass.** P. H. D. DE FONBRUNE (B.P. 400,109, 28.4.33. Belg., 28.4. and 20.6.32).—The micro-apparatus, to be attached to the stage of a microscope, consists of an electric resistance and suitable screw-operated mechanism which enable the drawing of fine needles etc. (e.g., for biological use) to be carried out with greater precision. J. A. S.

**Manufacture of articles [e.g., astronomical mirrors] of refractory material such as silica.** BRIT. THOMSON-HOUSTON CO., LTD. (B.P. 399,749—50, [A] 8.5.33, [B] 9.5.33. U.S., [A, B] 10.5.32).—A layer of transparent fused  $\text{SiO}_2$  ( $d$  2.2) is deposited upon a support of sintered  $\text{SiO}_2$  (heated to approx.  $1000^\circ$ ) by feeding the powdered material through the hot zone of a suitably impinging flame. After a relatively thin transparent layer has been slowly deposited (to avoid occlusion of bubbles) a thicker backing layer ( $d$  2.0) is more rapidly deposited without attempting to obtain it free from bubbles. The compound article having a facing of transparent  $\text{SiO}_2$  is then broken away from the support and given its final treatment. The surface layer of the article may be roughly defined by shaping the support. J. A. S.

**MgO refractories.**—See VII. Enamelled Cu wire.—See X.

#### IX.—BUILDING MATERIALS.

**Use of Rankin's diagram in cement burning.** S. SOLACOLU (Mitt. Zementtech. Inst. Tech. Hochsch. Berlin, 1932, No. 41, 110 pp.; Chem. Zentr., 1933, i, 3765).—Actual and theoretical equilibrium relationships in the system  $\text{CaO-SiO}_2\text{-Al}_2\text{O}_3$  are discussed. The properties and constitution of a series of cements of const.  $\text{Fe}_2\text{O}_3$  content have been studied. A. A. E.

**Grinding of cement clinker and lime-clay to produce new cements.** ANON. (Concrete, 1933, 41, 27—30).—The heat evolution ( $H$ ) in the hydration of rapid-hardening Portland cements (I) is due chiefly to the  $3\text{CaO.SiO}_2$  content. A new (I) with a low  $H$  is described. Quicklime and moist lump clay are ground together and hydrated. The product is finely ground with Portland cement clinker under controlled conditions. Higher mortar compression strengths than those of a standard (I) are claimed. T. W. P.



**Anhydrite cement, its preparation and properties.** P. P. BUDNIKOV (Chem.-Ztg., 1933, 57, 801—803, 822—823).—Gypsum burned at 600—700° for 30 min. no longer rehydrates in contact with H<sub>2</sub>O unless a catalyst is added; the best catalysts for this purpose are (a) 1% NaHSO<sub>4</sub>, (b) CaO 1 and FeSO<sub>4</sub>·7H<sub>2</sub>O 0.5%, (c) NaHSO<sub>4</sub> 0.6 and CuSO<sub>4</sub>·5H<sub>2</sub>O 0.8%. Anhydrite cement (I) prepared in these ways has a tensile strength of 45—50 kg. per sq. cm. after setting for 28 days. Concrete for interior floors, stairs, etc. of good durability may be prepared by mixing 1 pt. of (I) with 3 pts. of sand; it is resistant to frost and to short-time contact with H<sub>2</sub>O. A. R. P.

**Comparison of the rotary [cement] kiln and the high-capacity shaft kiln employing the new burning process.** O. FREY (Cement, 1933, 6, 321—326).—The advantages which the rotary kiln possesses in the finely-dispersed condition of the raw material and the large area of contact with the heating gases are claimed for the new shaft kiln. In this plant the material is passed through a multi-cord press which produces granules of < 0.28 in. diam. After passing the drying and preheating shaft (independent), dust is screened out so that the charge in the calcining shaft does not impede the flow of hot gases. C. A. K.

**Hydration [of cement].** R. GRÜN (Zement, 1933, 22, 143—147, 155—158; Chem. Zentr., 1933, i, 3765—3766).—The effect of conditions of hardening on strength and on dissolution by H<sub>2</sub>O has been studied. In Portland cement, but not in mixed (trass, blast-furnace slag) cements, hardening is attended by greater solubility of CaO. CaS in cryst. slags is readily, and in vitreous slags sparingly, sol. A. A. E.

**Setting of aluminous cement.** R. SALMONI and H. E. SCHWITTE (Zement, 1933, 22, 523—526).—The heat evolution and the swelling of the cement grains on hydration occurs after the final set (Vicat needle). The *p*<sub>H</sub> of the mixing H<sub>2</sub>O also increases suddenly at this point. The phenomena are delayed or accelerated when the cement is gauged with solutions of alkali salts and hydroxides, alkaline-earth hydroxides, dil. salt solutions, or salts of the weaker bases. Admixtures producing an increase of *p*<sub>H</sub> are accelerators; those giving a decrease, retarders. Conc. solutions of salts of Li and of bi- and ter-valent metals alter the phenomena completely. A colloid theory of setting is developed to account for the influence of *p*<sub>H</sub> on the setting phenomena, particularly that of the swelling of cement grains. T. W. P.

**Significance in cement and fine structure of tricalcium silicate.** A. GUTTMANN and F. GILLE (Zement, 1933, 22, 204—207; Chem. Zentr., 1933, i, 3614).—A discussion. A. A. E.

**Jointings, putties, and cements for chemical plant.** ANON. (Ind. Chem., 1933, 9, 381—388).—Tables showing the composition and uses of proprietary and other materials are given.

**Effect of tar acids on the wetting of wood by coal-tar oils.** F. H. RHODES and I. ERICKSON (Ind. Eng. Chem., 1933, 25, 1132—1133).—The wetting power (I) of PhMe or limpid oil prepared from coal-tar creosote dead oil, as indicated by the heat of wetting, is

increased 70% by the addition of 5% of PhOH. *o*-Cresol also increases (I). Although they do not increase the fungicidal power, phenols, by increasing (I), serve a useful purpose in creosote oil. E. C. S.

**Rotary kilns. Cyclonic dust collectors.**—See I. **Tar emulsions. Road-tar consistency.**—See II.

See also A., Nov., 1119, Ca aluminates. 1120, System CaO-FeO-SiO<sub>2</sub>. 1128, Zn phosphate cement.

#### PATENT.

**Emulsion for surfacing roads.** R. J. LACAU (B.P. 400,045, 31.1.33. Fr., 7.3.32).—A mixture of bitumen (about 50), tars from a continuous process of coal distillation (about 50), and either Swedish residuary oil (3%) or middle phenolic oil (2—5%) is emulsified with H<sub>2</sub>O 12—15 wt.-%. C. A. K.

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

**Influence of the method of blast introduction on the blast-furnace operation.** A. MICHEL (Stahl u. Eisen, 1933, 53, 1073—1078).—The movement of the charge in the blast furnace (*F*) and hence the operation of *F* is determined to a large extent by the shape of the combustion zone (*C*) in front of the tuyères (*T*). Increase in the vol. of blast (*B*) broadens *C*, whilst rise in temp. of *B* reduces the breadth and depth of *C*; *C* becomes broader with increase in the internal diam. of *T*, but its depth is independent of the shape of *T*. The length of *T* determines the distance between the end of *C* and the centre of *F*. A new type of *T* is described by the use of which the shape and size of *C* can be altered as desired; it consists of the usual conical mouthpiece in which a movable, H<sub>2</sub>O-cooled regulator is centrally disposed. Considerable coke economy is claimed to be obtained by its use. A. R. P.

**Reduction of iron ores by mineral gas.** C. CÂNDEA and J. KÜHN (Rocz. Chem., 1933, 13, 482—494).—Reduction of Fe<sub>2</sub>O<sub>3</sub> to Fe<sub>3</sub>O<sub>4</sub> by mineral gas (94% CH<sub>4</sub>) commences at 450°, of Fe<sub>3</sub>O<sub>4</sub> to FeO at 700—850°, and of FeO to Fe at > 850°. Fe catalyses the reaction CH<sub>4</sub> → C + H<sub>2</sub>; C formation is at a min. at certain temp. and velocities of flow of CH<sub>4</sub>, depending on the nature and degree of comminution of the ore used (magnetite, limonite, siderite, oligist). Somewhat better results are obtained by increasing the H<sub>2</sub> content of the gas by previously passing it over Fe at 900°. R. T.

**Dephosphorising and desulphurising [steel] in the high-frequency induction furnace.** P. BARDENHEUER and W. BOTTENBERG (Arch. Eisenhüttenw., 1933—4, 7, 229—232).—Laboratory and small-scale experiments are described which show that S and P are quickly removed from steel under a slag containing 15% of CaF<sub>2</sub> and 85% of CaO in a high-frequency induction furnace at 1550°. A. R. P.

**Manganese and phosphorus equilibria [in steel manufacture] in the light of new temperature measurements. II.** C. SCHWARZ (Arch. Eisenhüttenw., 1933—4, 7, 223—226; cf. B., 1933, 967).—The formulæ previously put forward for the equilibrium of Mn and P between metal and slag in the basic open-hearth process



are shown to be applicable to high-P slags. Nomograms for calculating the composition of the metal from that of the slag, or vice versa, are given. A. R. P.

**Nitrogen in the Thomas process [of making steel].** O. QUADRAT and M. PILZ (Chim. et Ind., 1933, 29, Spec. No., 694—697).—Pig Fe from the blast furnace, containing 0.001% N, contained 0.0017% N after treatment in the mixer, 0.0171% N after blowing in the Thomas converter, and 0.165% N after deoxidation. A sample ingot of the finished steel contained only 0.016% N, showing that the large proportion of the N is evolved during solidification of the ingot. Analyses of various portions of the ingot showed a small tendency for the N to segregate towards the centre. A. R. P.

**Temperatures of the steel and slag in the basic open-hearth process.** E. SCHRÖDER (Stahl u. Eisen, 1933, 53, 873—881).—Comparison of the readings given by the Optix radiation pyrometer (I) and a Mo-W thermocouple on steel (A) and slag (B) in the basic open-hearth furnace shows that (I) gives accurate readings with an emission factor of 0.5 for A and 0.6 for B. With the aid of (I) the variations in the temp. of A and B during all stages of the melting, refining, and tapping processes have been determined for many different steels, and the effects of additions of ore, CaO, and Fe-Mn alloy on the temp. differences between slag and metal ascertained. It is concluded that the casting temp. should be about 100—150° > the m.p. of the metal, and a table of suitable casting temp. is given for steels containing < 0.04—0.85% C. A. R. P.

**Slow graphitisation [in iron] at low temperatures.** A. LE THOMAS and E. MORLET (Bull. Assoc. tech. Fonderie, 1932, 6, Suppl. 9, 19—23; Chem. Zentr., 1933, i, 2303).—Graphitisation is shown dilatometrically to be appreciable after 15 hr. at 450°; at 350° the first indication of decomp. of cementite occurs after 200 hr. A. A. E.

**Effect of hydrogen on the A3 and A1 transformations of iron.** H. ESSER and H. CORNELIUS (Stahl u. Eisen, 1933, 53, 885—886).—The appearance of two peaks in the heating and cooling curves of Fe at the A1 and A3 points found by Harrington and Wood (Trans. Amer. Soc. Steel Treat., 1930, 18, 632; 1932, 20, 528) occurs only when the experiment is made in an atm. of H<sub>2</sub>; only one peak occurs in each case when the experiment is made in vac., hence the double effect is due to H<sub>2</sub>. A. R. P.

**Action of hydrogen on steel at high temperatures and pressures.** I. D. V. ALEXEEV and V. V. OSTROUMOV (J. Appl. Chem. Russ., 1933, 6, 621—629).—The mechanical properties of steel containing > 0.35% C are unaffected by heating at 350—400° during 90 hr. in an atm. of H<sub>2</sub> at pressures > 450 atm. At 450—470° decarbonisation takes place when the pressure is > 50 atm., with consequent deterioration of the steel. R. T.

**Ageing brittleness of open-hearth steel prepared from scrap.** H. WILHELM and L. JAHN (Arch. Eisenhüttenw., 1933—4, 7, 201—203).—Curves are given showing the notched-bar impact strength at —100° to 300° of steels prepared in the open-hearth furnace from 100% scrap and subsequently normalised and

aged; they show that a high % of scrap in the open-hearth charge does not increase the tendency to embrittlement on ageing, but has rather the reverse effect. A. R. P.

**Process of fusion of grey cast iron.** F. VANÍŠ (Chim. et Ind., 1933, 29, Spec. No., 663—672).—From micrographic examination of samples of cast Fe which have been heated for a short time just below the m.p. and then quenched, conclusions have been drawn as to the mechanism of fusion of the metal. The results show that just below the m.p. there is a rapid dissolution of the C, which produces a lowering of the m.p. and consequent fusion of the metal between the graphite lamellæ. A high P content accelerates dissolution of C, since the liquid phase commences to form from the areas rich in the P eutectic. Si has the opposite effect since it reduces the solubility of C in Fe. A. R. P.

**Heating of sand [moulds] during casting [of cast iron].** A. MITINSKY (Chim. et Ind., 1933, 29, Spec. No., 612—615).—Heating curves for sand moulds after tapping cast Fe into them are given; these indicate that the composition of the clay binder should be such that it is decomposed only when the gases evolved from the metal and from the mould have escaped, and that recovered sand from old moulds is a poorer heat conductor than new sand. To get the best results from using a proportion of old sand, efficient dust removal is essential. The actual rate of heat dissipation through the walls of a sand mould depends not only on their thickness, but also on the thickness and configuration of the casting. A. R. P.

**Oxygen in cast iron.** W. F. CHUBB (Metallurgia, 1933, 8, 125—126, 147—148).—Recent work on the effects of O in Fe is reviewed, with special reference to the effect of O on the mechanical properties of cast Fe. A. R. P.

**Oxygen in cast iron.** J. COURNOT, A. LE THOMAS, and L. HALM (Bull. Assoc. tech. Fonderie, 1932, 6, Suppl. 9, 24—35; Chem. Zentr., 1933, i, 2304).—The effect of O in Fe on the production of grey or white cast Fe has been studied. A. A. E.

**Phosphide eutectic in cast iron.** R. MITSCHÉ (Giesserei, 1932, 19, 497—499; Chem. Zentr., 1933, i, 2304).—Daeves' etching reagent is used for the recognition of the stable and metastable phosphide eutectics. A. A. E.

**[Heat]-treated black-heart malleable cast iron.** LEROYER (Rev. Mét., 1933, 30, 366—369).—The effect of various heat-treatments (I) and of cementation on the structure of this type of cast Fe is illustrated by photomicrographs, with brief notes on the change in mechanical properties produced by (I). A. R. P.

**Pearlite in black-heart malleable cast iron.** O. QUADRAT, A. BENDA, and J. KORITTA (Chim. et Ind., 1933, 29, Spec. No., 660—662).—Black-heart malleable cast Fe (A) should contain practically all the C as graphite; the combined C can be present (a) throughout the casting either as undecomposed cementite or as pearlite (I), or (b) as a thin layer of (I) just below the outer decarbonised layer of ferrite. The presence of C as (a) indicates incorrect heat-treatment in the malleablising process. Theoretically the presence of (b)



should also indicate incorrect heat-treatment, but it is almost impossible in practice to ensure absence of (b) even under the best conditions; it is shown by mechanical tests that a thin layer of (I) has very little effect on the characteristic properties of *A*.  
A. R. P.

**Martensitic cast iron.** M. WAHLERT and R. HANEL (Giesserei, 1932, 19, 458—464; Chem. Zentr., 1933, i, 2304).—The effect of Si, Mn, Ni, and Cr on the formation of graphite and cementite and on the transformation temp. and physical properties is discussed.  
A. A. E.

**Permanent set in certain cast non-ferrous alloys and austenitic cast irons.** J. E. HURST (Engineering, 1933, 136, 429—431).—The load required to produce a definite amount of closure or deformation of the gap in a slotted-ring specimen can be taken as a measure of elasticity, and the after-difference in the gap represents the permanent set (*A*). No clear relationship existed between the magnitude of (*A*) after several loadings and other properties. Al-Si and -Cu alloys possessed substantial *A* at low stress vals.; Al-Cu-Zn alloys have a less val. Austenitic cast Fe of the Ni-Resist type possessed high vals. which were of the same order as those of non-ferrous alloys even when heat-treated. During the prep. of the rings many cases of internal stress were observed by the opening or closing of the gap due to the release of the stress.  
C. A. K.

**Effect of nickel and silicon on growth of cast iron.** O. BORNHOFEN and E. PIVOVARSKY (Arch. Eisenhüttenw., 1933—4, 7, 269—274).—In cast Fe with C 3, Mn 0.03, P 0.02, S 0.015, Si 0.25—5.8, and Ni 0—5.7%, Si promotes growth when the structure is pearlitic or ferritic and retards it when the structure resembles that of temper cast Fe. When the Si is substituted by sufficient Ni to give the same graphite content the growth is considerably reduced. In the absence of another graphitising element Ni tends to promote growth on annealing, but its effect in the presence of a const. Si content is not additive. Alloys with the graphite in a form resembling temper-C show the least, and those with coarse graphite lamellæ in a pearlitic ground-mass the most, growth, whilst alloys with a ferritic ground-mass behave in an intermediate manner. Electrical conductivity measurements indicate that the chief cause of growth is oxidation of the Si.  
A. R. P.

**Volume changes of iron alloys in the vicinity of the m.p.** F. SAUERWALD and F. FLEISCHER (Arch. Eisenhüttenw., 1933—4, 7, 219—221).—During the solidification of steel and white cast Fe the increase in the C content of the  $\gamma$ -phase is accompanied by expansion, and conversely the reduction of the C content on melting is accompanied by a contraction. In graphitic cast Fe the dissolution of the graphite in the solid solution and in the liquid metal produces a contraction and the pptn. on cooling an expansion. In ordinary cast Fe these effects mask one another to a large extent and the effect of dissolved gases or gases produced by reactions becomes of the greatest importance when considering vol. changes.  
A. R. P.

**Influence of the diameter of the specimen on the endurance limit of steel in the rotating bending**

**test.** R. FAULHABER, H. BUCHHOLTZ, and E. H. SCHULZ (Stahl u. Eisen, 1933, 53, 1106—1108).—The val. of the endurance limit (*L*) of steel in this test is the smaller the larger the diam. (*D*) and is therefore not an abs. characteristic of the metal. With notched bars having a standard notch, *L* tends to a min. with increasing *D*, this min. being apparently a characteristic of the steel.  
A. R. P.

**Mechanism of break-down of steel. III.** M. KURODA (Bull. Inst. Phys. Chem. Res., Tokyo, 1933, 12, 814—817; cf. B., 1932, 309).—Microscopical study of C steels in successive stages of deformation leads to the view that plastic yield occurs as well as slip along cleavage planes.  
D. R. D.

**X-Ray investigation of the ageing effect in quenched carbon steels.** Z. NISHIYAMA (Sci. Rep. Tôhoku, 1933, 22, 565—569).—At room temp. a part of tetragonal martensite is slowly transformed into harder cubic martensite, resulting in age-hardening. The transformation is more conspicuous in low-C steels.  
E. S. H.

**Lattice distortion in nitrided steels and theory of hardness.** W. A. WOOD (Phil. Mag., 1933, [vii], 16, 719—727).—Extreme hardness, due to nitridding at 500°, is associated with an abnormally diffused and weakened X-ray spectrum. This is due to lattice distortion and a disturbance of the electron distribution, and not to small grain size. Electronic derangement interferes with slipping within the grain, and so increases the hardness.  
H. J. E.

**Nitrided steel.** F. GIOLITTI (Metallurg. ital., 1933, 25, 157—166; Chem. Zentr., 1933, i, 3351).—A discussion.  
A. A. E.

**High-chromium iron alloys for castings.** W. F. FURMAN (Met. & Alloys, 1933, 4, 147—150).—Castings containing 20—30% Cr, usually 27—30%, have been used extensively for resistance both to corrosion (*C*) and to oxidation (*O*) at high temp. Whilst, generally, *C* decreases as the C content increases, with the high-Cr alloys it is considered that satisfactory resistance is obtained with 0.25% C, which ensures a sounder casting than with less C. Increase in C, up to 3%, is almost without effect on *O*. Castings of this composition have been used on account of their resistance to S, e.g., for rabble arms in ore-roasting furnaces, but do not compare favourably with Ni-Cr alloys in their strength at high temp.  
C. A. K.

**Influence of silicon on the critical points and constitution of chromium[iron] alloys.** E. VALENTA and F. POBOŘIL (Chim. et Ind., 1933, 29, Spec. No., 633—648).—The effect of 0.5—2.6% Si in cast Fe containing 0.6—2.7% C and 14—37% Cr has been studied by thermal and dilatometric analysis and by micrographic examination of the alloys after various heat-treatments. Si enlarges the zone within which primary  $\alpha$  separates, reduces the extent of the homogeneous  $\gamma$ -field, raises considerably the transformation temp., and reduces the amount of C in the eutectic. These effects are illustrated by equilibrium diagrams of the Fe-Cr-C system based on the results of earlier workers and on deductions from the authors' work.  
A. R. P.



**Manufacture of nickel-chromium steels.** L. LE COZ (Chim. et Ind., 1933, 29, Spec. No., 655—656).—To prevent loss of Cr in the slag in preparing Ni-Cr steels by adding an excess of ferrochromium (I) just prior to casting, it is suggested that the slag be first saturated with Cr by addition of a cheap salt, e.g.,  $K_2Cr_2O_7$ ; addition of the theoretical amount of (I) should then give a steel with the desired Cr content.

A. R. P.

**Prevention of grain-boundary corrosion of steels with 18% Cr and 8% Ni.** E. HOUDREMENT and P. SCHAFFMEISTER (Arch. Eisenhüttenw., 1933—4, 7, 187—191).—Corrosion at the grain boundaries of austenitic non-rusting steel occurs usually after annealing at 500—800°; the cause is the pptn. of Cr-rich carbides in a finely-divided state at the grain boundaries and the consequent impoverishment of the steel in Cr in their vicinity. This trouble can be avoided by keeping the C content at <0.07% or adding elements, e.g., Ti, which have a high affinity for C and, by reducing the solubility of the carbides in austenite, thus prevent intercryst. corrosion (I). If the steel is cold-rolled to induce the formation of numerous slip planes (II) in the austenite crystals and then annealed in the crit. range (600—800°) the carbides are pptd. in a coagulated state in the interior of the crystals along the (II). (I) can also be prevented by heat-treating the steel to obtain a ferrite-austenite structure. The addition of Ti is probably the better method, since it prevents the development of brittleness on annealing at 700—900°.

A. R. P.

**Heat-resisting chromium-nickel-iron alloys for furnace construction. I. Load-carrying ability. II. Selection of alloys for specific conditions of use.** L. J. STANBERY (Met. & Alloys, 1933, 4, 127—135, 159—164).—I. A no. of load tests and creep vals. at high temp. have been collected from published sources and curves are given as an aid to designing furnace parts. The curves are admittedly extrapolations from scanty known vals., but represent a middle course between too optimistic and too pessimistic designing.

II. Highly reducing atm. may reduce the carbides in the grain boundaries. S gases are harmful, but in combination with sufficient Cr considerable amounts of Ni may be present even with a fairly high S content, particularly if present as  $SO_2$  or  $SO_3$ . Attack by S is, however, serious at >1100°. The choice of an alloy also rests on the absence of metastability of the alloy and on its suitability for mechanical working. When considering castings, fluidity, shrinkage, and general soundness influence the composition.

C. A. K.

**Composition of the carbides in chromium steels.** E. MAURER, T. DÖRING, and H. BUTTIG (Arch. Eisenhüttenw., 1933—4, 7, 247—255).—Analyses have been made of the carbides isolated by chemical or electrolytic treatment of steels with C 0.4—1.2, Cr 0.5—30, and Ni 0—5%. In steels with >7% Cr only  $Cr_3C_2$  was found and in alloys with >7% Cr only  $Cr_4C_3$ . No evidence of the existence of  $Cr_7C_3$ ,  $Cr_5C_2$ , or  $Cr_4C$  could be obtained.

A. R. P.

**Effect of heat-treatment on corrosion-resistance of stainless iron.** C. G. MERRITT (Trans. Amer. Inst.

Min. Met. Eng., 1932, 100, 272—292).—Tempering at about 536° produces marked susceptibility to corrosion, impact weakness, and general breakdown. Tensile properties and corrosion-resistance of heat-treated stainless Fe are excellent when tempering is above or below the susceptibility range. The brittle point and range of lessened corrosion are coincident with the conversion of Cr-martensite into Cr-troostite. The phenomenon is attributed to the formation of a very fine-grained ppt. of carbides at the grain boundaries and within the grains.

CH. ABS.

**Checking caustic corrosion with Ni and its alloys.** H. E. SEARLE and R. WORTHINGTON (Chem. Met. Eng., 1933, 40, 528—530).—The results of corrosion tests on various metals with aq. and fused NaOH and fused NaOH +  $Na_2O_2$  (5%) are given. For conc. and fused NaOH the use of cast Fe containing C 2.6—3.0, Si 0.8—1.4, Ni 18.0—22.0, and Cr 2.0—4.0% is indicated, but the use of S in the final processing of fused NaOH tends to offset the advantage of high Ni content of caustic-pot Fe.

D. K. M.

**Influence of solution concentration on severity of corrosion fatigue.** A. J. GOULD (Engineering, 1933, 136, 453—454).—Conc. saline solutions were much more corrosive than was distilled  $H_2O$ , though 2M and 0.025M solutions of KCl were practically identical in effect. Below the latter concn. the severity of attack decreased. Inhibitors retarded corrosion fatigue in accordance with concn., and the stress-no. of reversals curves plotted semi-logarithmically were straight lines in the  $10^5$ — $10^7$  reversals region.

C. A. K.

**Origin of non-rusting steels.** E. MAURER (Z. Elektrochem., 1933, 39, 820—822).—A criticism of Strauss' claim (B., 1927, 781) to have initiated the use of such steels.

E. S. H.

[Origin of non-rusting steels.] B. STRAUSS (Z. Elektrochem., 1933, 39, 822).—A reply to Maurer (cf. preceding abstract).

E. S. H.

[Origin of non-rusting steels.] E. MAURER (Z. Elektrochem., 1933, 39, 822—824).—Polemical (cf. preceding abstracts).

E. S. H.

**Determination of zirconium in plain-carbon and alloy steels.** T. R. CUNNINGHAM and R. J. PRICE (Ind. Eng. Chem. [Anal.], 1933, 5, 334—335).—Further details of procedure are given (cf. B., 1931, 298).

E. S. H.

**Determination of titanium in plain-carbon, high-chromium, and 18:8-chromium-nickel steels.** T. R. CUNNINGHAM (Ind. Eng. Chem. [Anal.], 1933, 5, 305—306).—The sample is dissolved in dil.  $H_2SO_4$ , Ti is pptd. by cupferron in presence of  $Fe^{II}$ , the ppt. is collected, ignited, and fused with  $K_2S_2O_7$ , and the melt dissolved in 10%  $H_2SO_4$ . Ti is then determined colorimetrically by the  $H_2O_2$  method.

E. S. H.

**Modulus of elasticity of metals at various temperatures.** A. MITINSKY (Chim. et Ind., 1933, 29, Spec. No., 616—619).—The modulus of elasticity ( $M$ ) of hard-drawn steel falls with rise in temp. to a min. at 150—200°, then rises at about 250° to a max. which is greater the harder is the steel, and falls abruptly at 300° to the same val. as at 150°; above 400° there is first a



rapid and then a slower fall in  $M$ , followed by a slight rise at the transformation point ( $T$ ). For annealed steel  $M$  rises slightly up to  $100^\circ$  and then falls slowly to  $500^\circ$  and rapidly to  $700^\circ$ ; again there is a slight rise in  $M$  at  $T$ . For various brasses and for Cu  $M$  falls slowly to  $600^\circ$  and then more rapidly; in the hard-worked state there is a slight upward tendency of the curve at  $300^\circ$ .

A. R. P.

**Impact torsion test.** I. M. ITHARA (Tech. Rep. Tôhoku, 1933, 11, 16—50).—An illustrated description is given of an improved form of impact torsion-testing machine which automatically records on a sensitised paper the change in the twisting moment ( $M$ ) and angle of twist ( $\theta$ ) with time. With the aid of this machine  $M$ - $\theta$  diagrams for annealed mild steel and annealed Cu have been obtained which show that the yield point is raised by increasing the twisting speed ( $S$ ), and that the  $M$ - $\theta$  curve approaches a horizontal line which is higher the higher is  $S$ . Comparison has also been made of the sliding speeds of impact tension, compression and torsion tests, and of the  $M$ - $\theta$  diagrams of notched-bar impact and static torsion tests.

A. R. P.

**Distribution of porosity in aluminium and copper ingots with some notes on inverse segregation.** N. P. ALLEN (Inst. Metals, Sept., 1933. Advance copy, 16 pp.).—The order and manner in which solid constituents separate out from the molten ingot are of greater influence than is the movement of gas bubbles. Progressive increase in porosity as solidification proceeds may be due to the greater rate of cooling on the outside, the segregation of dissolved gas to the centre, or the fall in hydrostatic pressure during cooling. Ingots which have a long freezing range often produce a sound cone at the centre of the bottom of the ingot, and the use of such a range is a probable cure for castings which are liable to suffer from large cavities or localised spongy patches.

C. A. K.

**Experiments in wire drawing. III. Annealing of H.-C. copper wires drawn to varying degrees of hardness.** W. E. ALKINS and W. CARTWRIGHT (Inst. Metals, Sept., 1933. Advance copy, 19 pp.; cf. B., 1931, 1055).—Increase in the duration or rise in temp. of annealing caused a progressive loss of strength, and the more lightly drawn wires annealed to considerably lower strengths than the harder specimens. It was evident that the tensile strength of a "completely" annealed high-conductivity (H.-C.) Cu wire was not a characteristic property of the material, but that it was related to the prior working treatment undergone. The greater the amount of cold-work performed the lower is the temp. at which the wire begins to lose its hardness and the greater the rate of loss at any temp.

C. A. K.

**Development of internal stresses and season-cracking in cold-drawn brass tubes.** J. FOX (Engineering, 1933, 136, 375—376).—Aq.  $\text{HgNO}_3$  is the best medium for detecting a tendency to season-crack in cold-drawn brass tubes; for those drawn over a mandrel aq.  $\text{NH}_3$  should be used. The presence of incipient cracks after the test can be ascertained by applying a bulging test between the compression plates of a Buckton testing machine. Rules for the manufacture of brass tube free from any tendency to season-crack are given.

A. R. P.

**Surface tension of type-metal alloys.** H. V. WHITE (Bull. Virginia Polytech. Inst. Eng. Exp. Sta., Series Bull., 1933, No. 13, 35 pp.).—The surface tension ( $I$ ) of various alloys does not vary greatly; impurities and slight oxidation have little effect. Zn greatly increases ( $I$ ).

CH. ABS.

**Determination of tin in babbitt metal by reduction with zinc.** A. A. BOCHVAR and A. M. ПОПОВА (Тзвет. Мет., 1932, 319—321).—The sample (0.2—0.5 g.) is dissolved in HCl (30 c.c.) with a little (0.5—1 g.)  $\text{KClO}_3$ . After boiling for 10 min. 3 g. of Zn are added; the mixture is boiled for 15—20 min. until all the Zn is dissolved, and then 1—2 min. longer to ensure complete dissolution of Sn.  $\text{CaCO}_3$  is then added and the mixture cooled and titrated with I.

CH. ABS.

**Herculoy—a copper-silicon-tin-zinc alloy.** R. A. WILKINS (Met. & Alloys, 1933, 4, 123—126).—The alloy contains Cu 94.75, Si 3.25, Zn 1.5, and Sn 0.5% and has the structure of an  $\alpha$ -bronze. Annealing of the forged and drawn rod at  $290^\circ$  for 30 min. increases the yield point from 39.5 to 47 tons per sq. in., whereas the tensile strength remains const. at about 60 tons per sq. in. and the elongation at 11%. The  $d$  is 8.54, coeff. of thermal expansion  $1.7 \times 10^{-5}$ , thermal conductivity 0.05 g.-cal. per sec. per c.c. per  $^\circ\text{C}$ ., m.p.  $1023.5^\circ$ , resistivity (annealed wire) 21.75 microhms per c.c., temp. coeff. of resistance  $4.6 \times 10^{-4}$ , modulus of resiliency 17.5 (soft), 375 (hard) in.-lb. per cu. in., and fatigue strength 13 tons per sq. in. ( $6 \times 10^7$  reversals). It has a high resistance to corrosion in acid, alkaline, and saline solutions.

A. R. P.

**Application of the Waelz process to Karabash (Russia) copper-zinc ores and mixed concentrates.** A. N. VOLSKI, R. A. AGRACHEVA, and N. G. SEREBRENNIKOVA (Тзвет. Мет., 1932, 322—346).—The treatment of the ores, containing sphalerite, chalcopyrite, tennantite, pyrite, etc. (Cu 3—4, Zn 3—7, Pb 0.2—0.5, As 0.2—0.9, Fe 33—40%, Au 13—66 g., Ag 1.2—1.9 g. per ton, S 43—46%), is described.

CH. ABS.

**Extraction of metal from slags containing zinc.** V. A. VANYUKOV and G. V. RUIKOV (Тзвет. Мет., 1932, 467—492).—The reduction of metals from suspended powdered slags with gas ( $\text{CO} + \text{H}_2$ ) can be economically applied to the extraction of Zn, Pb, and part of the Sn in the form of a mixture of their oxides. Cu, Sn, and Ni are obtained in the form of a melt easily separable from the slag. Average recovery is: Zn 96—98, Pb 94—96, Cu 85—90, and Sn 80—85%.

CH. ABS.

**Treatment of residues and rationalisation of zinc-distillation processes.** G. V. RUIKOV (Тзвет. Мет., 1932, 456—467).—Application of the gas process to residues, slags, old retorts, etc. is economical. The treatment raises the recovery of Zn in the distillation process to 95—97%.

CH. ABS.

**Testing thickness of zinc on galvanised wire.** Z. DYAKONOVA (Тзвет. Мет., 1932, 550—552).—Zn and ZnO are removed from the cleaned ( $\text{C}_6\text{H}_6$  or  $\text{Et}_2\text{O}$ ) wire by a solution of  $\text{As}_2\text{O}_3$  in  $\text{H}_2\text{SO}_4$ ; in 15 sec. from the first appearance of bubbles a layer 0.0001 cm. thick is removed. The wire is then washed in  $\text{H}_2\text{O}$  and dipped in conc. aq.  $(\text{NH}_4)_2\text{S}$ ; black spots indicate bare Fe.



FeS is detected by brief treatment with conc. AcOH and treatment of the acid with  $K_3Fe(CN)_6$ . CH. ABS.

**Determination of calcium in lead-calcium alloys of low calcium content.** B. L. CLARKE and L. A. WOOTEN (Ind. Eng. Chem. [Anal.], 1933, 5, 313—315).—The sample is dissolved in  $HNO_3$ ,  $H_2SO_4$  is added,  $PbSO_4$  filtered off, the solution made alkaline with  $NH_3$ , and  $Ca^{++}$  pptd. by  $(NH_4)_2C_2O_4$  in presence of EtOH. A slight modification of the usual volumetric procedure for determining Ca is recommended. The error is  $< \pm 0.002\%$  Ca. E. S. H.

**Applications of silver solders in chemical industry.** R. J. SNELLING and E. R. THEWS (Chem. Fabr., 1933, 6, 443—445).—A review. E. S. H.

**Purification of mercury containing metallic impurities.** W. F. ALEWIJN (Chem. Weekblad, 1933, 30, 687).—Air is passed through the Hg at  $90^\circ$ , in the light, when Zn, Pb, etc. oxidise readily and may be removed by filtration. Final treatment with  $HNO_3$  is recommended. The air which has passed through the Hg should be bubbled through  $H_2O$  to condense Hg vapour. H. F. G.

**Concentration of Akdjal arsenic-bearing sands.** V. I. TRUSLEVICH (Tzvet. Met., 1932, 492—505).—Extraction of As concentrates from various residual products of treatment of Au ores has been studied. Flotation using turpentine + a solution (I) of thio-carbanilide in *o*-toluidine, (I) + xanthate, or (I) + xanthate + pine oil permitted 90% recovery. CH. ABS.

**Quantitative rapid micro-analysis of pure aluminium. I. Determination of iron, copper, and manganese.** F. PAVELKA and H. MORTH (Mikrochem., 1933, 13, 305—312).—Fe is determined colorimetrically as Prussian blue in a  $H_2SO_4$  solution of the metal by addition of  $K_3Fe(CN)_6$ . Cu is determined by means of its accelerating effect on the reaction between  $Na_2S_2O_3$  and  $Fe(CNS)_3$ . The time for decolorisation under standard conditions is observed, and the amount of Cu present interpolated from a standardisation curve (cf. B., 1922, 962). Mn is first pptd. with Fe by addition of  $FeSO_4 + Br-H_2O + excess NaOH$ , the ppt. dissolved in  $H_2SO_4 + H_2O_2$ , and Mn oxidised by means of  $(NH_4)_2S_2O_8$  to  $MnO_4^-$ , which is determined colorimetrically. J. S. A.

**Dilatometry of an aluminium alloy.** O. QUADRÁT and R. POSPÍŠIL (Chim. et Ind., 1933, 29, Spec. No., 620—622).—Dilatometric tests on "Y" alloy (Al 92, Cu 3.87, Ni 1.95, Mg 1.61, Si 0.27, Zn 0.26, and Fe 0.20%) castings showed that, in spite of a certain amount of segregation of the Cu and Ni, there is no appreciable change in the coeff. of thermal expansion of various parts of the casting. Samples taken from a piston casting showed max. deviations of 0.35% Cu and 0.12% Ni from the mean. A. R. P.

**Aluminium-chromium alloys.** G. GALLO and G. FRAGAPANE (Aerotech., 1931, 11, 1539—1554).— $< 2\%$  Cr noticeably altered hardness and resistance to corrosion. Cr does not improve the corrosion-resistance of duralumin. CH. ABS.

**Protal process for protection of light alloys.** J. COURNOT (Chem. & Ind., 1933, 891—892).—Al or an Al alloy is immersed for 40 min. in a boiling solution ( $pH$  6) of certain heavy-metals salts, including halides, to produce an adherent film containing  $Al_2O_3$  and a complex halogen compound of the metal. Complete protection is afforded by impregnating this film with a  $H_2O$ -repellent finish. A. R. P.

**Hardness of low-melting alloys at different temperatures. II. Binary alloys the components of which combine chemically.** V. P. SHISHOKIN and V. A. AGEVA (Tzvet. Met., 1932, 119—136).—Hardness, m.p., and temp. coeff. of hardness (I) were determined for Pb-Bi, Tl-Bi, Tl-Pb, Te-Bi, Al, Mg, and Te. The formation of intermetallic compounds is indicated. For Mg, Al, Zn, Cd, Sn, Te, Tl, Pb, and Bi the at. vol. varies inversely (except for Te and Bi) with hardness. Except for Sn (I) is parallel with the change in coeff. of expansion (II). (II) is inversely proportional to the m.p. In the binary eutectic systems (I) is  $>$  in pure metals, max. vals. being observed at or near the eutectic composition. CH. ABS.

**New differential dilatometer.** O. QUADRÁT and R. POSPÍŠIL (Chim. et Ind., 1933, 29, Spec. No., 623—630).—The apparatus makes use of two round rods, one of the metal under test and the other (standard) of "Baros" alloy, the thermocouple being inserted in a hole in the latter. The movement of the rods on heating is transferred by means of levers to an optical registering device employing AgCl paper. Some results obtained on steels are recorded. A. R. P.

**Cleaning of metals. III. Theory of the alkaline cleaner. IV. Some practical aspects of alkaline cleaning.** S. WERNICK (Ind. Chem., 1933, 9, 275—277, 308—311).—III. For the removal of fat, this must be emulsified for some time. The addition of soap (2%) to  $H_2O$  reduces the surface tension (I), alkali (2%) does not. The addition of alkali to soap solution reduces the interfacial tension (II) against oil, but (I) is increased. Low (II) promotes the formation of an emulsion, but there is an optimum  $pH$  for its permanence. The relative detergent vals. of soaps differ for different types of (solid) "dirt." For each soap solution there is an optimum detergent concn. within the range 0.2—0.4%. Rise in temp. of the detergent results in a reduction of the detergent val. The relative detergent vals. of NaOH,  $Na_2CO_3$ ,  $Na_3PO_4$ , and  $Na_2O.4SiO_2$  are given.

IV.  $C_2HCl_3$  and  $CCl_4$  are used for the removal of grease. Aq. NaOH,  $Na_2CO_3$ , Na silicate (A),  $Na_3PO_4$ , KOH,  $K_2CO_3$ , with or without the addition of an insol. substance, e.g., china clay,  $Al(OH)_3$ , are also used with or without previous degreasing treatment. Zn may be cleaned by aq. Na zincate and A, and Al by aq. Na aluminate and A; Cu, brass, and Ni-silver are cleaned by aq. KCN (1—2% solution). D. K. M.

**Measurement of throwing power of galvanoplastic baths.** A. ONITCHENKO (Z. Elektrochem., 1933, 22, 815—818).—A rectangular cathode bent at an angle of  $90^\circ$  is used. The throwing power (T) is determined by the ratio of the wt. of metal on the two end quarters of the cathode to that deposited on the two middle quarters.



Experiments with Cr baths containing 300 g.  $\text{CrO}_3$  per litre show that  $T$  is impaired when the  $\text{H}_2\text{SO}_4$  content is  $> 3$  g. per litre. The optimum condition is reached when the ratio  $\text{CrO}_3:\text{H}_2\text{SO}_4 = 150:1$ .  $T$  is also impaired by increasing c.d., but is unaffected by temp.

E. S. H.

**Rôle of the "armoured chromium bath" in chromium plating.** R. BILFINGER and G. ELSSNER (Metallwar.-Ind. Galv.-Tech., 1933, 31, 147—149; Chem. Zentr., 1933, i, 3624).—Claims are rejected. A. A. E.

**Significance of the bath temperature in chromium electrolytes.** E. WERNER (Metallbörse, 1933, 23, 241—242, 350—351; Chem. Zentr., 1933, i, 3355).—Experiments with baths containing various addenda [ $\text{PhOH}$ ,  $\text{Na}_2\text{SiF}_6$ ,  $\text{Na}_2\text{SO}_3$ ,  $\text{NaF}$ ,  $\text{HgCl}_2$ , and  $(\text{NH}_4)_2\text{SO}_4$ ] at 30—70° are described. A. A. E.

**Deposition of chromium on glass.** R. C. WILLIAMS (Physical Rev., 1932, [ii], 41, 255).—Cr is electroplated on a W filament and then quickly evaporated in a vac. on to the glass surface. A firm, highly-reflective surface for 4200—2900 Å., and one preferable to Ag, is obtained. L. S. T.

**Rational analysis of nickel-plating salts.** J. MALY (Chem.-Ztg., 1933, 57, 823—824).— $\text{H}_3\text{BO}_3$  and free  $\text{H}_2\text{SO}_4$  are determined by extraction with  $\text{Et}_2\text{O}$  in a Soxhlet thimble, and titration of the  $\text{H}_2\text{SO}_4$  (Meorange) and of the  $\text{H}_3\text{BO}_3$  after addition of glycerol (phenolphthalein) with  $\text{NaOH}$ . Ni is determined by electrolysis in ammoniacal solution and Mg in the filtrate with Schaffgot's reagent; the filtrate from the Mg is used for the determination of the alkalis. Citric acid is determined by oxidation of the solution with  $\text{KMnO}_4$  in presence of  $\text{HgSO}_4$  and weighing the pptd. Hg salt.  $\text{Cl}^-$ ,  $\text{NH}_3$ , total  $\text{SO}_4^{2-}$ , and  $\text{H}_2\text{O}$  are determined in the usual way. A. R. P.

**Refractories for metal-melting.**—See VIII. Coreless vac. induction furnaces. Electric heating wires.—See XI. Coatings for metal signs.—See XIII. Effect of metals on raspberries etc.—See XIX.

See also A., Nov., 1111, Al-Cu. Solid solutions of Pb and Sn in Bi. Systems Fe-Se, Cu-Ni-Sn, Fe-C-Si. Pb-Sn bronzes. Mol. compounds in liquid steel. 1122, Electrochemistry of corrosion. 1125, Salt effect in corrosion of metals. 1135, Cu-glass seals. 1200, Metal containers and food-stuffs.

## PATENTS.

**Nitrogenisation of ferrous austenitic [iron] alloys.** SOC. ANON. COMMENTRY, FOURCHAMBAULT ET DECAZEVILLE (B.P. 398,834, 14.3.32. Fr., 14.3.31).—Stable  $\gamma$ -Fe alloys containing  $< 1$  of the following: C  $\nabla 1$ , Al  $\nabla 7$ , Cu  $\nabla 20$ , Si  $\nabla 5$ , W  $\nabla 10$ , Mo  $\nabla 10$ , V  $\nabla 2$ , Ti  $\nabla 2$ , Zr  $\nabla 2$ , B  $\nabla 4\%$ , are nitrated in  $\text{NH}_3$  at 600—950° and then annealed in vac. The process is especially applicable to the surface-hardening of Ni-Cr non-rusting steels. A. R. P.

**Annealing of steels and alloys.** MOND NICKEL CO., LTD. (B.P. 399,049, 9.12.32. U.S., 9.12.31).—Ni-Cr steels and alloys of the stellite type are bright-annealed after rolling or drawing by heating them in 5—30%

$\text{HNO}_3$  or by treating them electrolytically to produce a passive surface and then heating them in a reducing atm. at 920—1000° for 2 hr. A. R. P.

**Manufacture of steels and alloy steels.** VEREIN. STAHLWERKE A.-G., Assees. of P. KÜHN (B.P. 399,401, 6.3.32. Ger., 10.3.32. Addn. to B.P. 369,258; B., 1932, 470).—The alloying elements (Cr, Mo, V, Ti, W, Cu, or Ni) specified in the prior patent are added in the form of ore to the slag in the basic open-hearth furnace and therefrom reduced into the metal bath. A. R. P.

**Heat-resisting steel alloy.** H. J. SCHIFFLER (B.P. 398,680, 25.11.32).—The alloy contains C  $< 1$ , Cr 2—24, Al 0.2—4, P 0.06—1, S  $< 0.05$ , and Ni 0.2—10%. Up to 2% of Mn, Mo, V, Co, Ti, Cu, Be, or Zr may be added also or up to 3% (in all) of  $< 2$  of these. A. R. P.

**Purification of gases derived from roasting processes.** I. G. FARBENIND. A.-G. (B.P. 397,851, 29.2.32. Ger., 1.5.31).—Gases containing  $\text{SO}_3$  or  $\text{H}_2\text{SO}_4$  vapour are freed from dust in a dry electrostatic plant at  $> 300^\circ$ , cooled to  $< 100^\circ$ , moistened with a  $\text{H}_2\text{O}$  spray, and treated in a wet electrostatic plant to condense a relatively conc.  $\text{H}_2\text{SO}_4$ . A. R. P.

**Sintering fine [lead, copper, or iron] ores or the like.** F. KRUPP GRUSONWERK A.-G. (B.P. 399,382, 10.2.33. Ger., 2.3.32).—Fe sponge is mixed with the charge for sintering in a blast apparatus to act partly or wholly as fuel and to assist in slagging the gangue in the subsequent smelting process. A. R. P.

**Production of enamelled copper wire for electrical purposes.** P. FUSTIER, and SOC. ANON. J. BOCUZE & Co. (B.P. 399,685, 10.1.33. Fr., 3.2.32).—Irregularities or projections in the wire are removed by anodic dissolution in aq.  $\text{CuSO}_4$ , using a Pt cathode, and the wire is then enamelled as usual. A. R. P.

**Copper alloys [substitutes for bronze].** N. D. CHOPRA (B.P. 399,219, 1.4.32).—Claim is made for a hard bronze-like alloy containing Cu 80, Fe 1, Al 2, Zn 13, and Sn 4%. The Fe and Al are added as a hardener alloy made by heating Al and Fe with part of the Cu until a vigorous exothermic reaction occurs. A. R. P.

**Manufacture of alloys containing copper and zinc.** H. W. BROWNSDON, M. COOK, H. J. MILLER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 399,177, 24.3.32).—Alloys of Cu and Zn consisting of pure  $\alpha$  may be hardened by addition of 1—3% of Al and  $< 3$  times as much Ni, quenching from 900°, and reheating at 300—500° preferably after cold-work (cf. B., 1933, 921). A. R. P.

**Hard alloys.** TOOL METAL MANUFG. CO., LTD. (B.P. 398,661, 22.10.32. Ger., 23.10.31).—Claim is made for a sintered hard alloy containing 10—50% of a difficultly fusible nitride (I) and  $< 10\%$  of a difficultly fusible boride and/or silicide (II), the sum of (I) and (II) being  $< 55\%$ . Up to 20% of a hard carbide (III) may also be present, and the mixture is bonded with 6—10% of Co and/or Ni. The metal constituents of (I), (II), and (III) are preferably Ti, V, and Ta. A. R. P.

**Hard alloys [containing tungsten-chromium carbide].** TOOL METAL MANUFG. CO., LTD. From



F. KRUPP A.-G. (B.P. 398,932, 8.4.32).—The alloy is made by sintering a mixture of 30–97% of  $(3Cr_3C_2, W_2C)$  with  $\leq 1$  metal of the Fe or Cr group and with (or without)  $\leq 1$  of the carbides or carbonitrides of Fe, Cr, V, and Ti. A. R. P.

**Casting of magnesium or its alloys.** E. H. MOORE (B.P. 399,124, 15.5.33).—The sand for the mould is mixed, or the finished mould sprayed, with a solution of  $ZnCl_2$  in  $COMe_2$  diluted (1:1 by vol.) with  $CCl_4$ . A. R. P.

**Alloys containing beryllium and silver.** H. A. SLOMAN (B.P. 399,261, 25.5.32).—Ag alloys having a high resistance to tarnishing by S compounds consist of Ag  $> 90$  (92.5)%, Be  $\geq 1.5$  (0.5)%, and Cu the remainder (7%). A. R. P.

**[Grain-refining of] aluminium and aluminium-base alloys.** A. H. STEVENS. From ALUMINIUM, LTD. (B.P. 400,026, 30.12.32).—Addition of 0.01–1 (0.03–0.5)% Ti is claimed. A. R. P.

**Aluminium-magnesium alloys.** A. H. STEVENS. From ALUMINIUM, LTD. (B.P. 398,080, 9.1.33).—Claim is made for Al alloys containing 5–16 (10)% Mg and  $\geq 0.1$ % Fe with Si  $>$  the Fe content so as to increase the resistance to corrosion. A. R. P.

**Manufacture of ["modified" silicon]-aluminium alloys.** E. H. MOORE (B.P. 400,121, 6.5.33).—Modification of Al alloys with 5–15% Si is effected with 0.5–10% of  $KNO_3$  and/or  $NaNO_3$  at 850–900°. A. R. P.

**Disazo dyes [for Sn printing].**—See IV.

## XI.—ELECTROTECHNICS.

**Construction and operation of a coreless vacuum induction furnace.** W. BOTTENBERG (Arch. Eisenhüttenw., 1933–4, 7, 233–236).—The coil and crucible are totally enclosed in a steel cylinder (I) provided with external channels and a Zn casing so arranged that cooling- $H_2O$  can be passed along the channels to prevent undue heating of (I) by induced currents. Provision is made for introducing the leads ( $H_2O$ -cooled flat Cu tubes) and for evacuating (I). Diagrams and examples of the use of the furnace are included. A. R. P.

**Economical [electric] muffle furnace operation.** C. G. HARREL and S. DUOOS (Cereal Chem., 1933, 10, 452–456).—A considerable saving in rheostat power consumption may be effected by substituting the rheostat of furnaces operating on a.c. by a transformer and those operating on d.c. either by an instrument which may be termed a high-temp. thermostat, or by a potentiometer ( $P$ ) actuated by a thermocouple in the furnace;  $P$  may be of the type which automatically either (a) records the temp. on a chart and controls several furnaces, or (b) controls one furnace and indicates the temp. with a pyrometer. E. A. F.

**Electric heating elements.** I. ASAKI (Japan Nickel Rev., 1933, 1, 311–321).—The life of Ni-Cr resistance wires depends largely on the method of drawing. Longer life is obtained with a fine-grained structure. Oxidation is not very serious in wire of good quality, as the greater part of the oxide film consists of  $Cr_2O_3$ , which scales off

with difficulty. The variation of life ( $L$ ) with temp. ( $T^\circ C.$ ) is given by  $L = (A/T)^\gamma$ , where  $A$  and  $\gamma$  are consts. E. S. H.

**Life of electric heating wires.** M. HORIOKA (Japan Nickel Rev., 1933, 1, 292–310).—The relation between the life ( $L$ ) of resistance wire and the abs. temp. ( $K$ ) of service is given by  $L = Ae^{-\theta/K}$ , where  $A = \alpha r_0(1-\gamma)$ ,  $r_0$  is the radius of the wire, and  $\theta$ ,  $\alpha$ , and  $\gamma$  are consts. Tests have been carried out in NaCl solution and in air and  $O_2$ . At temp. above 1100° the wt. of oxide film produced on Ni-Cr wires has a direct relation to the life of the wire. E. S. H.

**Forced life test of [electric] heating wires.** S. TOGO (Japan Nickel Rev., 1933, 1, 322–341).—Testing equipment is described. The life of Ni-Cr resistance wires depends on the physical structure and uniformity of diam., and on the adhesion of the oxide film. E. S. H.

**Seven years' technical  $p_H$  measurement with the indicator leaf.** P. WULFF (Chem. Fabr., 1933, 6, 441–443).—Applications of the method (A., 1927, 221) are discussed. E. S. H.

**New rectifier.** M. ANASTASSIADES (Compt. rend., 1933, 197, 677–678).—The rectifier consists of Cu,  $CuS$ ,  $Cu_2S$ , Cu. C. A. S.

**Boiler feed- $H_2O$ .**—See I. Active carbons.—See II. Dephosphorising (etc.) steel. Cr-plate. Cr deposits on glass. Ni-plating salts. Throwing power of [plating] baths.—See X. Test for  $H_2O$  in paint.—See XIII. Soil sterilisation. Determining  $p_H$  of soils.—See XVI. Sugar-boiling control.—See XVII. Determining chlorogenic acid etc.—See XIX.

See also A., Nov., 1103, Dielectric losses of insulating oils. 1121, Effect of glass containers on Weston normal cells. 1122, Electrochemistry of corrosion. 1127, Reduction of aromatic ketones. Oxidation of  $NH_2$ -acids. 1132, Potentiometric titration. Electrometric iodimetry. Determining As. 1134, Thermo-regulators. Na lamp. Ag filter for ultra-violet light. 1135, Measurements of crit. potentials. Sb electrode for soil measurements. Standard-cell comparator. Rectifier photo-cells for daylight measurement. Photoelectric cells and their uses. 1139, Analysis of liquid org. substances.

## PATENTS.

**Electric furnaces.** SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST. GOBAIN, CHAUNY & CREY (B.P. 400,020, 19.12.32. Fr., 26.12.31.).—In a rotary furnace for fusing materials by radiation from a C resistor, the latter is immovably supported within a chamber independently of the walls thereof and is surrounded by an inert atm. J. S. G. T.

**Electric storage batteries or accumulators [for motor vehicles].** F. TEMPLE (B.P. 399,882, 16.4.32).

**Insulating material.**—See VI. Annealing steels and alloys. Purifying roaster gases. Enamelled Cu wire.—See X. Plastics for conductors. Moulded insulators.—See XIII.



## XII.—FATS; OILS; WAXES.

**Phytosteryl acetate test as a routine method in the examination of butter fats with border-line Reichert-Meißl values.** H. HAWLEY (Analyst, 1933, 58, 529—531).—5% of vegetable fat (I) in butter is detected by the raising of the m.p. of the steryl acetates by phytosteryl acetate. The sterols are isolated as the digonitides, and converted by boiling with  $\text{Ac}_2\text{O}$  into the acetates, which are recrystallised from 90% EtOH. In the absence of (I) the m.p. is 114—115°; 10% of (I) raises this to 117° and, on further recrystallisation, to 120°.

E. C. S.

**Shortening value of plastic fats.** J. D. FISHER (Ind. Eng. Chem., 1933, 25, 1171—1173).—The shortening power of the following fats, determined by measuring the breaking strength of wafers made with a standard mixture, increases in the order: refined lard, vegetable-oil compound, hydrogenated lard, animal stearin-vegetable oil compound, hydrogenated cottonseed oil.

E. C. S.

**Determination of water content of fatty acids.** L. RZĄDKOWSKI (Przemysł Chem., 1933, 17, 219—220).—The  $\text{H}_2\text{O}$  content of fatty acids, pure, or containing neutral fats and soaps, can be calc. from determinations of the acid val. before and after drying with anhyd.  $\text{Na}_2\text{SO}_4$ .

R. T.

**Determination of oxidised fatty acids.** J. GROSSER (Seifensieder-Ztg., 1933, 60, 49—50; Chem. Zentr., 1933, i, 3818).—Flocks of oxy-acids, obtained on extraction of the hydrolysate of fats containing them with light petroleum, contain fatty acids and salt solution; washing and ignition are necessary. Small amounts of the acids remain in the acid solution after extraction.

A. A. E.

**Production of "Kernseifen" [curd and settled soaps].** R. KRINGS (Allgem. Oel- u. Fett-Ztg., 1933, 30, 495—500).—The manufacture of curd soaps ("Oberseife") is briefly described and future developments of the German soap industry are discussed.

E. L.

**Soap. III. Settled soap.** A. FURIA (Chimica, 1933, 1, 230—231).—Fitting and finishing (cooling, mulling, etc.) processes are described.

E. L.

**Soap-boiling control.** M. SALIPO (Masloboino Zhir. Delo, 1932, No. 10, 19).—The use of the centrifuge in the control of soap-boiling operations is described.

CH. ABS.

**Rancidity of oils and fats.** A. TAFFEL and C. REVIS (Chem. & Ind., 1933, 880; cf. B., 1933, 1016).—The ordinary peroxide test for rancidity should be made at room temp. as there is a possibility that the labile peroxides characteristic of the early stages of oxidation may change to more stable forms at higher temp.; e.g., peroxide vals. for a blown arachis oil of 1.8 and 4.8 were recorded at room temp. and 100°, respectively. (A few c.c. of  $\text{CCl}_4$  may be added to the AcOH in order to dissolve solid fats.)

E. L.

**Rancidity of oils and fats.** C. H. LEA (Chem. & Ind., 1933, 917—918; cf. preceding abstract).—Attention is drawn to the test described in the Food Investigation Board Report, 1929, 31 (Analyst, 1930, 55, 508; cf. Lea, B., 1931, 726), whereby 1 g. of fat, dissolved

in  $\text{AcOH}-\text{CHCl}_3$ , is heated with solid KI, zero blank tests being obtained with perfectly fresh fats. E. L.

**Determination of iodine values by the pyridine sulphate [di]bromide method.** H. HAWLEY (Analyst, 1933, 58, 601).—A comparison of results obtained by the Rosenmund and Kuhnhehn (A., 1923, ii, 886) pyridine sulphate dibromide method (I), and the Wijs, Margosches, and Hübl methods for sesamé (containing mineral or arachis) oil, for arachis, olive, linseed, and safflower oils, and for the solid fatty acids from ghee, shows that (I) gives figures which can be duplicated and are independent of moderate variations in time of reaction or excess reagent, but, being lower, must be accepted with considerable caution in comparisons with the other methods.

J. G.

**New reagent for determination of iodine values.** L. HUNTER and F. F. HYDE (Analyst, 1933, 58, 523—527).—The reagent is prepared by the interaction of KI in glacial AcOH with dichloramine-T and is used in the same way as Wijs' reagent. Parallel determinations of the I vals. of 13 oils by the 2 methods are given. The new method gives, on the whole, slightly the lower results. The I val. of tung oil rises rapidly to 156 after exposure for 5 min., and reaches a steady val. of 197 in 6 days.

E. C. S.

**Polymerisation of linseed oil.** H. I. WATERMAN and D. OOSTERHOF (Rec. trav. chim., 1933, 52, 895—900).—An apparatus for the distillation of sensitive substances [e.g., linseed oil (I)] of mol. wt. 800—1000 in a high vac. is described. (I) is thus distillable practically completely without alteration in physical consts. Distillation of stand oil (II) gives a distillate of free fatty acids and unpolymerised oil; the residue consists of polymerised material which possesses better drying properties than the original (II).

H. B.

**System of control for oil-palm factories.** C. D. V. GEORGI (Malay. Agric. J., 1933, 21, 413—428; cf. B., 1933, 27, 397).—In the system fully detailed all raw, intermediate, finished, and waste products are weighed, sampled, and analysed, so that the efficiency of the factory may be regularly checked.

E. L.

**Cost reduction in oil works.** F. WEISZFEILER (Allgem. Oel- u. Fett-Ztg., 1933, 30, 485—492).—Cost analysis and process control are discussed in relation to oil production.

E. L.

**Soya-bean extraction.** M. JUNKER (Allgem. Oel- u. Fett-Ztg., 1933, 30, 492—495).—Mucilage (I) may be separated in the hydrated condition by treating the extracted oil with small amounts of  $\text{H}_2\text{O}$ ; the oil is dried and centrifuged and (I) is worked up for recovery of oil, phosphatides, etc.

E. L.

**Russian soya-bean oil.** S. JUSCHKEVITSCH (Fettchem. Umschau, 1933, 40, 197—200).—Analyses of 13 samples from various districts in Russia and a sample from Manchuria are detailed. The vals. range:  $n_D^{25}$  1.4724—1.4760, sap. val. 188.1—192.3, I val. 115.9—143.4, CNS val. 74.3—83.9, hexabromide val. 0—10.1, saturated acids (Bertram) 13.9—20.6% (of total acids?), unsaponifiable matter 0.52—0.99%. Oils from the more northern districts showed higher I, CNS, and hexabromide vals., and had a higher linolenic and lower



oleic and saturated acid content than oils from the southern districts. The linoleic acid content of all samples was about the same (50% of the acids).

E. L.

**Rubber-seed oil.** L. P. HART (Amer. Paint & Varnish Manufrs.' Assoc., July, 1933, Circ. No. 438, 232—233).—Recent samples of the hot- and cold-pressed oil proved more suitable for use in paints etc. than samples examined earlier (*ibid.*, Circ. Nos. 118 and 154). Much less tendency to develop free fatty acids was observed, but the possibility of reaction with basic pigments still exists. Chemical and physical consts. of the oils as received and after extraction from 6-months-old paints made therefrom are tabulated. S. S. W.

**Composition of the oil from the physic nut, *Jatropha curcas*, L.** (MLLE.) M. T. FRANÇOIS and (MLLE.) S. DROIT (Bull. Soc. chim., 1933, [iv], 53, 728—741).—The oil, extracted from the ground kernels by various solvents, has  $d_{4}^{20}$  0.9168—0.9198,  $n_D^{20}$  1.4720—1.4730, f.p. —15° to —13°, sap. val. 176—180, I val. (Hanus) 97—98, Ac val. (André) 4—8, free acid (as % oleic) 0.4—0.6. A small amount of the oil is sol. in EtOH; this contains the major part of the unsaponifiable matter (from which a phytosterol, m.p. 135—136°, is isolated) and a little resin. The fatty acids obtained by hydrolysis of the part insol. in EtOH are palmitic, stearic, myristic, oleic, and linoleic; OH-acids could not be detected. H. B.

**Tar from olive husks. Petroleum hydrocarbons from fats etc.**—See II. "Lorol."—See III. Vitamins in cottonseed products.—See XIX.

See also A., Nov., 1141, Prep. of sulphonic acids of high mol. wt. 1142, Capillary fractionation of fatty acids. 1144, Fats and SH·CH<sub>2</sub>·CO<sub>2</sub>H. 1183, Larva-fat of the beetle. Rat-body fats. 1211—3, Vitamins (various). 1216, Volatile oil of *Sarothra gentianoides*. 1217, Kernel-fats of the *Palmae*. Fat and oil from seeds of *Actinodaphne Hookeri*. Oil from seeds of *Tectona grandis*. Wax from alkanet root.

## PATENTS.

**Production of stearine.** W. SCHRAUTH (U.S.P. 1,904,546, 18.4.33. Appl., 26.2.31. Ger., 25.2.30).—The soaps produced by the process described in B.P. 370,769 (B., 1932, 612) are decomposed with mineral acid. E. L.

**Antioxidant for fats and oils.** R. C. NEWTON and D. P. GRETTIE, Assrs. to SWIFT & Co. (U.S.P. 1,903,126, 28.3.33. Appl., 11.4.32. Cf. B., 1933, 878).—Gum guaiac (< 0.1%), being insol. in H<sub>2</sub>O and sol. in oils, is an excellent antioxidant for fats in bakery products etc. containing H<sub>2</sub>O. E. L.

(A) Treatment of China-wood [tung] oil to produce a liquid ungelled oil. [B] Non-drying oil and product thereof. F. M. REECE, Assr. to O'BRIEN VARNISH Co. (U.S.P. [A] 1,903,686 and [B] 1,903,656, 11.4.33. Appl., [A] 6.10.30, [B] 26.11.30).—(A) Gelation is prevented if the oil is very rapidly heated (*e.g.*, during its passage through a narrow heated coil) through the temp. range (up to 357°) in which gelling normally occurs, and then rapidly chilled, whereby a superior

drying oil ( $n < 1.499$ ) is obtained. (B) If the oil is similarly heated to 330—370° a non-drying oil ( $d < 0.920$ , I val. < 100,  $n < 1.499$ ) is obtained which is useful as plasticising and softening agent for cellulose ester lacquers. E. L.

**Manufacture of sulphonated products [oleines].** H. KANTOROWICZ and A. KÖHLER, Assrs. to FETTSÄURE-U. GLYCERIN-FABR. G.M.B.H. (U.S.P. 1,904,791, 18.4.33. Appl., 9.4.32. Ger., 14.4.31).—Commercial oleine which has been destearinised by chilling to —5° to 10° is sulphonated at low temp. with H<sub>2</sub>SO<sub>4</sub> or ClSO<sub>3</sub>H. E. L.

**Extraction of oil and moisture from "fish press-cake."** W. T. CONN, Assr. to U.S. GOVT. (U.S.P. 1,903,503, 11.4.33. Appl., 8.8.29).—The press-cake (from rendered oily fish) is chopped or masticated and extracted with a volatile solvent. E. L.

**Saponification of waxes and separation of the alcohols therefrom.** IMPERIAL CHEM. INDUSTRIES, LTD., W. A. SEXTON, and D. WARD (B.P. 398,807, 17.3.32).—Sperm oil or spermaceti is saponified by heating with dry NaOH and/or KOH, and the alcohols are removed from the soap by distillation with superheated steam. E. L.

**Refining [of fats, oils, waxes, resins].** L. ROSENSTEIN (U.S.P. 1,900,132, 7.3.33. Appl., 20.4.31).—Impurities and free fatty acids are extracted from the raw materials by treatment with liquid NH<sub>3</sub>. E. L.

**Alcohols from fats etc. Preps. from castor oil.**—See III. Germicides.—See XXIII.

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

**Construction defects and other factors which cause paint failures or which influence the durability of exterior paints on wood surfaces.** H. A. GARDNER (Amer. Paint & Varnish Manufrs.' Assoc., April, 1933, Circ. No. 428, 107—157).—An illustrated survey is given of the nature and causes of various types of failure of exterior paints on wood surfaces, *e.g.*, chalking, checking, gas discoloration, Cu and other stains, mildew, spotting, dirt collection, flaking, and blistering, and suggestions are made for their prevention. Proper construction and surface-conditioning, and correct application of the paint are important factors in determining durability of house paint. S. S. W.

**Casein paints and casein emulsion media.** G. TRÖGER (Farbe u. Lack, 1933, 472, 487—488, 499—500, 516).—A description is given of the properties of casein (I), the prep. of (I)-H<sub>2</sub>O and (I)-oil emulsion paints, and of the properties of the dried films. Recent patents are reviewed with particular reference to paints which undergo phase change on drying so that the H<sub>2</sub>O becomes dispersed in the emulsoid. Such films approach oil films in their behaviour. S. M.

**Ageing of coal-tar coatings.** A. LÉAUTÉ (Compt. rend., 1933, 197, 751—752).—Plates coated as described previously (B., 1933, 755) and then heated in sand to 250° when slowly bent to a radius of 1 cm. showed complete plasticity of the coating if the filler consisted



of powdered coal containing 33% of volatile matter of which half passed 300-mesh/in., but cracked badly if the filler was powdered chalk or was absent.

C. A. S.

**Qualitative test for water in paint.** H. A. GARDNER (Amer. Paint & Varnish Manufrs.' Assoc., May, 1933, Circ. No. 431, 211—213).—The intensity of the blue colour developed when white paints are rubbed up or otherwise mixed with methylene-blue is used as a qual. and roughly quant. indication of  $H_2O$  content. The possibility of a quant. measurement of the passage of current through a paint containing a high amount of  $H_2O$ , by adding NaCl, thus converting the paint into an electrolyte, is indicated.

S. S. W.

**Standardisation of kauri-butanol test for paint and lacquer thinners.** L. C. BEARD, V. L. SHIPP, and W. E. SPELSHOUSE (Ind. Eng. Chem. [Anal.], 1933, 5, 307—309).—Variation in properties of different specimens of kauri gum render the above test inaccurate. It is proposed to standardise gums by their behaviour towards PhMe and  $C_6H_{14}$  (kauri-BuOH vals. 102 approx. and 26), which cover the range of ordinary lacquer thinners. The amount of gum used in making the test, temp., and  $H_2O$  content must also be standardised.

E. C. S.

**Physical tests and their bearing on manufacture and use of protective materials.** G. F. NEW (J. Oil Col. Chem. Assoc., 1933, 16, 292—305).—The colour changes during fading and the rate of fading ( $R$ ) are plotted for a lake pigment (I) in admixture with varying proportions of blanc fixe (II) and dispersed in a Si ester. The initial  $R$  increases rapidly with the quantity of (II) present because the faded dye is transparent; hence to obtain weak tints in white pigments large concns. of dyes having weak tinting power should be used. Since various lakes do not fade in a parallel manner, testing should be done with 10% mixtures of (I). A triangular chart illustrates the immediate effect on the red, green, and blue components of 8 pigments and dyes when diluted with increasing quantities of (II). In some cases the colour does not move directly to the white centre, probably because intense surface reflexion for parts of the spectrum is first obliterated; this behaviour is one factor in the bronzing of pigments. Simple apparatus is outlined for measuring hiding power and gloss; the former may be increased as much as 100% in the case of a white pigment by adding 1% of a black pigment. A pressure adjuster for use with the Ostwald viscosimeter is also described.

S. M.

**Instrument for hiding-power determinations [of paint].** G. G. SWARD (Amer. Paint & Varnish Manufrs.' Assoc., July, 1933, Circ. No. 433, 217—218).—A rotary "cryptometer" is described in which the wedge of the Pfund instrument is replaced by a circular glass plate mounted in a metal frame which is threaded so that the plate is raised or lowered by rotating it. A circular scale indicates the crit. paint-film thickness. The difficulty of obtaining smooth movements of a wedge is overcome, but the method has the objection that bubbles of air are sometimes introduced in operation. An alternative is suggested whereby a central black glass plug is the only movable part.

S. S. W.

**Apparatus for testing texture and dispersion [of ground paints].** H. A. GARDNER (Amer. Paint & Varnish Manufrs.' Assoc., July, 1933, Circ. No. 432, 214—216).—The ground paste is thinned with an equal vol. of white spirit or turpentine and flowed on an inclined sheet of glass. The appearance of the film, viewed by transmitted light from a frosted electric light bulb behind the glass, is compared with standards representing different grades of grinding.

S. S. W.

**Fluorescence testing.** E. STOCK (Farben-Ztg., 1933, 38, 1557—1558).—A simple apparatus is described whereby paint and varnish raw materials etc. are observed for ultra-violet fluorescence effects in a portable, enclosed box, natural or artificial light (filtered through a dark glass filter) being used as light source in place of Hg-vapour lamps etc.

S. S. W.

**Impact adhesion and brittleness tester [for paint etc. films].** L. P. HART (Amer. Paint and Varnish Manufrs.' Assoc., July, 1933, Circ. No. 435, 223—225).—A hammer attached to a pivoted arm is allowed to drop on the coated specimen which is placed on a cushioning rubber sheet with a hole in it under the area of impact. For testing adhesion the specimen may be struck on the coated or uncoated side, but for brittleness best results are obtained if the coated side is face down. Tests should preferably be made under controlled temp. and humidity conditions. Different types and degrees of cracking under this test are illustrated.

S. S. W.

**Solid colour alkyd (glycerin phthalate) coatings.** L. P. HART and H. A. GARDNER (Amer. Paint & Varnish Manufrs.' Assoc., July, 1933, Circ. No. 436, 226—229).—A summarised report is given on the condition after 12 months' exposure of the panels described earlier (cf. B., 1933, 975). Bodied tung oil finishes, though chalking more than alkyd finishes in general, showed less cracking and better adherence than some of the alkyds. Adhesion and brittleness tests by the "impact tester" (cf. preceding abstract) are also reported and illustrated.

S. S. W.

**Mildew prevention on painted surfaces.** H. A. GARDNER, L. P. HART, and G. G. SWARD (Amer. Paint & Varnish Manufrs.' Assoc., Aug., 1933, Circ. No. 442, 242—265).—Linseed-oil paints containing white-lead (I), ZnO-titanox-(I), chrome-green, and a mineral brown oxide were treated with 56 toxic substances and spread on panels which were sprayed, when almost dry, with a dispersion of moulds in agar and then exposed under mildewing conditions in Florida for 60 days. The discoloration was greatest with (I), but none took place in presence of  $HgCl_2$  (II), mercuraphen (III), Hg Ph acetate (IV),  $As_2O_3$  (V),  $BaCl_2$  (1 : 300 of paint), or thymol (1 : 150). The fungicides were also tested by adding a ring or drop of the treated paints to an inoculated culture medium in Petri dishes which were incubated at 85% R.H. and 30°. (II)—(V), mustard oil, *p*-chloro-*m*-cresol, BzOH, phthalic anhydride, Paris-green, and  $ZnCrO_4$  were the most effective (final conditions are photographed). Mildew formation in raw linseed oil paints is reduced by using high pigment concn. and by partial substitution of lithopone, titanox, and, to a small extent, (I) by ZnO. Badly mildewed



surfaces can be prepared for ordinary, non-fungicide paints by applying a 25% solution of thymol in aq. EtOH and allowing to dry. S. M.

[German] technical terms [in the paint industry]. W. VAN WÜLLEN-SCHOLTEN (*Farben-Ztg.*, 1933, 38, 1581—1583).—The use of German words, e.g., particularly "Farbe," in many different senses renders standardisation desirable, and suggested definitions for various paint terms in use are tabulated. S. S. W.

Painting technique of J. van Eyck. (A) J. MAROGER (*Compt. rend.*, 1931, 193, 740—741). (B) J. MAROGER and G. MOURIER-MALOUF (*Ibid.*, 1933, 197, 766—767).—(A) The medium appears to have been an emulsion prepared probably by rendering purest linseed oil, drying by gentle heating with 3—6% of a 1:1:1 mixture of calcined white lead, litharge, and amber until the froth turns brown, adding resin (0.5—1 pt. to 1 pt. of oil), and emulsifying the resultant varnish by mixing with gum arabic and sufficient H<sub>2</sub>O.

(B) The medium is transparent when dry, and consists of minute oleoresinous droplets embedded in the stabilising material; the particles of pigment are thus kept dispersed. C. A. S.

German ochres. E. MAYER (*Farben-Ztg.*, 1933, 38, 1529).—The possibility of removing admixed earthy impurities from German ochres by elutriation processes, thus improving colour tone and tendency to settle out in paints, is discussed. S. S. W.

Effect of acidity in the oil-absorption capacity of a pigment. S. VAKUBOVICH and M. GOLDBERG (*Lakokras. Ind.*, 1933, No. 1, 15—21).—Increase in acidity generally reduces the oil-absorption capacity (I) of a pigment if no side reactions are involved. Basic pigments increase (I). CH. ABS.

Plasticiser absorption by pigments. A. W. VAN HEUCKEROTH (*Amer. Paint & Varnish Manufrs.' Assoc.*, July, 1933, Circ. No. 441, 239—241; cf. B., 1933, 399).—The absorptions of 21 lacquer pigments for (C<sub>6</sub>H<sub>4</sub>Me)<sub>3</sub>PO<sub>4</sub> (I), Bu<sub>2</sub> phthalate (II), and linseed oil (III) are tabulated. Vals. for (I) are usually slightly > those for (II); both these are usually much > vals. for (III). Addition of 10% of boiled (III) considerably reduces the quantity of plasticiser absorbed. S. M.

Vehicles for some luminous pigments. A. W. VAN HEUCKEROTH (*Amer. Paint & Varnish Manufrs.' Assoc.*, July, 1933, Circ. No. 440, 237—238).—Satisfactory vehicles for CaS (2 pts.) were: (a) Resoglaz resin (2 pts.), Ph<sub>2</sub> phthalate (1 pt.), and PhMe (10 pts.), (b) 30% dammar solution in turpentine (8 pts.), (c) 20% chlorinated rubber solution in PhMe (7 pts.), and 50% chlorodiphenyl resin solution in turpentine (3 pts.). Si and cellulose ester media gel and evolve H<sub>2</sub>S; linseed oil and oleo-resins reduce the luminosity. Grinding the pigment into the vehicle has the same effect. S. M.

Lacquer liquids. A. W. VAN HEUCKEROTH (*Amer. Paint & Varnish Manufrs.' Assoc.*, April, 1933, Circ. No. 429, 158—180).—A list is given of the liquid solvents, diluents, and plasticisers now available, their principal physical and chemical properties and their trade names

being tabulated, together with an indication of their uses. A list of trade names of cellulose and synthetic plastics and related products is also included. S. S. W.

Clear coatings for metal signs. H. A. GARDNER (*Amer. Paint & Varnish Manufrs.' Assoc.*, July, 1933, Circ. No. 437, 230—231).—The use of clear top varnishes on lithographed metal advertising signs, to obviate the chalking and fading of certain colours, is recommended. Tests on a range of varnishes are briefly outlined; promising results were shown by nitrocellulose and Reziglas plasticised with 25 and 50%, respectively, of Ph<sub>2</sub> phthalate, nitrocellulose lacquers with high resin content of Alkyd No. 19 and Alkyd balsam No. 33, and a "44-gal." modified PhOH-CH<sub>2</sub>O-tung oil varnish. The need for a baking oil primer is indicated. S. S. W.

Resistance of lacquers towards [liquid] fuel mixtures. J. KORECKÝ (*Chem. Obzor*, 1933, 8, 47—48; *Chem. Zentr.*, 1933, i, 3801).—Addition of 20% of EtOH to benzine, and particularly of greater quantities of MeOH, increases corrosion. A. A. E.

Adhesion studies on exposed plasticised lacquers. A. W. VAN HEUCKEROTH (*Amer. Paint & Varnish Manufrs.' Assoc.*, July, 1933, Circ. No. 439, 234—236).—Nitrocellulose lacquers which contain substantial amounts of chrome-yellow and one of 9 common plasticisers were found to possess great durability. The adhesion and brittleness were determined after exposure. S. M.

Lithographic bubble viscosimeter. J. R. STEWART (*Amer. Paint & Varnish Manufrs.' Assoc.*, July, 1933, Circ. No. 434, 219—222).—A series of viscosity standards for lithographic varnishes (cf. Gardner-Holdt standards for varnishes in general) is put forward. These adjusted mixtures of non-changing mineral oils have viscosities such that the time of ascent of the air bubble over a measured distance gives abs. viscosity in poises. S. S. W.

Resistant coatings based on pure phenolic resins. J. HARLASS (*Farben-Ztg.*, 1933, 38, 1466—1467).—The development of oil-sol. types of PhOH-CH<sub>2</sub>O resins is traced and the manufacture, by hot and cold processes, of oil varnishes containing Super Beck-acite 1001 is described. 13 stoving enamels based on such varnishes and varying in stoving time from 1½ to 4 hr. at 190—200° were made; oil length (1:1—1:3), nature of oil component (linseed-tung stand oil, tung stand oil, raw tung oil), and amount of pigment and thinner were also varied. Details of bending and impact tests, and resistance to alkali at 20° of these enamels are tabulated, and the general properties of such products are outlined. S. S. W.

Gloss measurement.—See I. Oil-refinery by-products and the pigment industry.—See II. Tautness of doped fabrics.—See VI. Rubber-seed oil.—See XII. Artificial horn.—See XV.

See also A., Nov., 1108, Liquocryst. resins and lacquers. 1165, Action of heat on resin acids. 1166, Prep. of pimaric acids. Furfuraldehyde-PhOH condensation. 1216, Oleoresin of *Pinus monticola*.



## PATENTS.

**Luminous paints, moulded articles, coatings, or lacquers.** I. G. FARBENIND. A.-G. (B.P. 400,068, 1.3.33. Ger., 1.3.32).—To obtain durable films ZnS or other luminous pigment is incorporated with polymerised styrene, vinylnaphthalene, or vinyltetrahydro-naphthalene together with a plasticiser. To obtain foil, sheets, etc. the product is polymerised by heating with  $H_2SO_4$  and then poured into suitable moulds.

S. M.

**Manufacture of pigments.** IMPERIAL CHEM. INDUSTRIES, LTD., J. E. CLAPHAM, and A. J. HAILWOOD (B.P. 399,497, 31.12.31).—The tinctorial power and dispersibility of a pigment are increased by grinding it with a small proportion of the  $H_2SO_4$  ester of a  $C_{10-20}$  alcohol, e.g., dodecyl, cetyl, or stearyl alcohol.

S. M.

**Manufacture of aqueous finishings and body colours.** I. G. FARBENIND. A.-G. (B.P. 399,816, 11.4.32. Ger., 10.4.31).—The  $H_2O$ -sol. org. colours used in aq. finishings are applied as  $NH_4$  (or mixed  $NH_4$  and alkali-metal) salts instead of as alkali-metal salts; e.g., the dye, diaminostilbenedisulphonic acid  $\rightarrow$  2 mols. of  $PhOH$ , alkylated, is converted into the  $NH_4$  salt, treated with  $CO(NH_2)_2$ , and added in aq. solution to an aq. casein syrup.

C. H.

**Manufacture of [cellulose ester] plastic materials.** KODAK, LTD., Assees. of H. T. CLARKE and C. J. MALM (B.P. 399,814, 11.4.32. U.S., 9.4.31).— $Ph_3$ , tritoyl, tolyl phenyl, and trinaphthyl phosphates can be used to plasticise and reduce the inflammability of films made from an ester of cellulose and a soap-forming acid, e.g., the stearate, laurate, oleate, and from cellulose phenylacetate or crotonate.

S. M.

**Manufacture of softening agents [for plastic materials].** I. G. FARBENIND. A.-G. (B.P. 399,817, 11.4.32. Ger., 10.4.31).—Softeners for resins prepared from casein, or other albuminous material, and  $CH_2O$  are made by partly sulphonating an animal or vegetable oil with 10–40% of  $H_2SO_4$ ; the product is washed with  $H_2O$ , and  $(NH_4)_2SO_4$  and  $NH_3$  are added until it is weakly alkaline.

S. M.

**Manufacture of moulded articles, particularly for electrical insulating purposes.** INTERNAT. GEN. ELECTRIC Co., INC., Assees. of ALLGEM. ELEKTRICITÄTS GES. (B.P. 399,738, 18.4.33. Ger., 18.4.32).—The formation of surface-leakage paths in phenol-aldehyde and other resins is reduced by a covering layer of an  $NH_2Ph$  or other amine resin which is scattered on the article during the pressing operation.

S. M.

**Manufacture of plastic materials for production of insulated conductors.** D. TRAILL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 399,868, 14.4.32).—Dibenzyl or diphenyl methylene ether, or one of their lower alkyl or alkoxy-derivatives, is incorporated with a  $H_2O$ -resistant cellulose ether, e.g., benzyl- or ethyl-cellulose, by heating between hot rolls at 130–140° or by preparing a dope. Fillers, colouring matter, etc. may be added.

S. M.

**Plastic masses for dental impressions.** I. G. FARBENIND. A.-G. (B.P. 399,842, 13.4.32. Ger., 13.4.31).—Copal resin (15–40), shellac (1–20), stearic acid

(10–35), and talc or other filler (30–70 pts.) are heated with a trace of colouring matter, e.g.,  $Fe_2O_3$ , at 60–350° until a homogeneous mass results.

S. M.

**Manufacture of artificial resins.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 399,232, 5.4.32).—Compounds of the  $C_6H_6$  or  $C_{10}H_8$  series carrying  $\leq 1$  olefine side-chain are polymerised in admixture with  $\alpha$ -unsaturated ketones, preferably in emulsion. Examples are  $CH_2=CH\cdot COMe$  or  $CH_2=CMe\cdot COMe$  with styrene emulsified in presence of oleic  $\beta$ -diethylaminoethoxy-anilide hydrochloride.

C. H.

**Castor oil derivatives.**—See III. Disazo dyes [for varnishes].—See IV. Transfer composition.—See V. Flexible treated material.—See VI. Products from tung oil. Refining resins.—See XII. Anti-halation layers for plates etc.—See XXI.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Ageing processes of rubber. II. Gold reaction on products of incipient oxidation of rubber.** B. DOGADKIN and V. BALANDINA (Kautschuk, 1933, 9, 146–148).—Pale crêpe rubber which has been irradiated by a quartz lamp or milled for a short time on cold rolls gives an extract to cold  $H_2O$  which reduces a faintly alkaline solution of  $AuCl_3$  to colloidal  $Au$ ; hot mastication does not have this effect. Smoked sheet rubber is similarly affected and exhibits a max. result after approx. the same period of irradiation as crêpe, but it yields an extract with some reducing power before exposure. The aq. extracts of the rubber show a steady increase in electrical conductivity on irradiation for  $> 30$  min. The phenomena are doubtless associated with the chemical changes accompanying ageing.

D. F. T.

**Antioxygens [for rubber].** F. JACOBS (Caoutchouc et Gutta-Percha, 1933, 30, 16,294–16,296, 16,328–16,329, 16,357–16,358, 16,578–16,580).—A description of various commercial antioxygens, giving their chemical nature, properties, and uses.

**Machine for laboratory evaluation of fatigue of rubber compounds flexed under compression.** L. V. COOPER (Ind. Eng. Chem. [Anal.], 1933, 5, 350–351).

E. S. H.

**Balance-plastometer.**—See I. Rubber-seed oil.—See XII.

See also A., Nov., 1115, Solutions of rubber in PhMe.

## PATENTS.

**Treatment of artificial caoutchouc [factice] preparatory to mixing and vulcanising with natural caoutchouc.** V. KAUFMANN (B.P. 399,009, 1.9.32).—In order to prevent retardation of vulcanisation by  $HCl$  liberated by factice (I) prepared from saponifiable oils and  $S_2Cl_2$ , the (I) is treated with  $H_2O$  or steam in presence of neutralising agents such as carbonates or hydroxides, for the removal of loosely-bound  $Cl$ . Vulcanisation accelerators may be added to the treated (I) before its incorporation in rubber.

D. F. T.

**Manufacture of rubber compositions.** DUNLOP RUBBER Co., LTD., F. J. PAYNE, E. W. MADGE, and W. G. GORHAM (B.P. 399,940, 22.6.32).—Sponge rubber or



cellular rubber material, reduced to a uniform particle size  $< 0.5$  in. by subjecting it to a rapid succession of shearing impacts, *e.g.*, in a disintegrator of the revolving-arm type, is used for the production of articles by bonding with suitable agents such as aq. dispersions of rubber (which may previously be frothed) or rubber solutions. D. F. T.

#### Manufacture of rubber-impregnated products.

G. A. RICHTER, W. B. VAN ARSDEL, and R. B. HILL, Assrs. to BROWN Co. (U.S.P. 1,900,951, 14.3.33. Appl., 22.4.29).—Fibrous sheet material, *e.g.*, cellulose, impregnated with rubber latex, after being dried and, if desired, vulcanised with  $S_2Cl_2$ , is mechanically worked with an aq. solution, *e.g.*, of NaOH. This treatment, which is followed by washing and drying, removes non-caoutchouc solids and enhances the pliability of the product. D. F. T.

#### Manufacture of (A) rubber-bonded asbestos products, (B) products containing rubber-bonded fibres which normally coagulate rubber latex.

DEWEY & ALMY, LTD. (B.P. 399,870—1, 15.4.32. U.S., 17.4.31).—(A) An aq. suspension of asbestos is contacted with an aq. dispersion of electropositive, colloidal rubber particles which may be obtained by the addition of aq.  $AlCl_3$  to diluted  $NH_3$ -preserved latex. The rubber becomes associated with the asbestos but retains its colloidal dimensions. (B) Fibres which normally coagulate aq. dispersions of rubber are incorporated with a rubber dispersion after this has been aggregated, *e.g.*, to an average particle size between  $50 \mu$  and  $200 \mu$ . Suitable aggregating agents are  $Al_2(SO_4)_3$ ,  $Na_2SiF_6$ ,  $Th(NO_3)_4$ , the effect being controllable by the proportion of the aggregant, dilution of the latex, and the pre-addition of suitable protective agents. D. F. T.

**Recovery of fibrous material and rubber from waste rubber stock.** J. K. MITCHELL, Assr. to DISPERSIONS PROCESS, INC. (U.S.P. 1,900,944, 14.3.33. Appl., 1.8.28).—Waste rubber (I) stock associated with fibrous material (II) is manipulated as a plastic coherent mass in the presence of a hydrophilic colloid which is also desirably a plasticising agent, *e.g.*, a sulphonated oil with or without glycerol, so that (I) is separated from (II), with as little damage as possible to the fibres themselves. (II) works its way to the surface of the mass and may be removed and washed. Alternatively, if  $H_2O$  be present during the manipulation, a suspension of (II) in an aq. dispersion of (I) may be obtained which can be beaten and, after coagulation of the rubber on the fibre, then fed to a paper machine. D. F. T.

**Manufacture of decorated rubber articles.** B. F. GOODRICH Co., Asses. of C. W. LEGUILLON (B.P. 399,051, 17.12.32. U.S., 21.12.31).—Articles with an embedded pattern are produced by using a patterning stock of lower plasticity than the main stock and applying the former in liquid form, *e.g.*, as compounded latex. On drying and vulcanisation under pressure, the locally applied deposits become embedded in the surface of the main stock without substantial distortion of outline. D. F. T.

**Vulcanisation of rubber.** C. COLEMAN, Assr. to NAUGATUCK CHEM. Co. (U.S.P. 1,901,582, 14.3.33. Appl., 5.9.31).—A compound of the probable structure

$R'S \cdot CH_2 \cdot NHR''$ , where R' is benzthiazyl and R'' an aryl group, obtained, *e.g.*, by the reaction of equimol. proportions of a mercaptobenzthiazole (I),  $CH_2O$ , and a primary arylamine, or of (I) and a methylenearylimide  $CH_2 \cdot NR$ , is used to accelerate vulcanisation. D. F. T.

**Manufacture of age-resisting vulcanised rubber products.** J. R. INGRAM, Assr. to RUBBER SERVICE LABS. Co. (U.S.P. 1,902,405, 21.3.33. Appl., 14.6.30).—A compound of the structure  $R'(NHR'')_3$ , where R' is glyceryl and R'' is an aryl radical, *e.g.*, the condensation product of glycerol and an aromatic amine such as  $\beta\text{-}C_{10}H_7 \cdot NH_2$ , is incorporated in rubber before vulcanisation, for antioxidant purposes. D. F. T.

**Rubber composition and method of preserving rubber.** A. W. CAMPBELL, Assr. to B. F. GOODRICH Co. (U.S.P. 1,902,115, 21.3.33. Appl., 14.3.32).—The deterioration of rubber is retarded by a secondary aromatic amine obtained by the reaction of a triaryl-methyl chloride and a diarylamine in presence of  $AlCl_3$ , *e.g.*,  $p\text{-}CHPh_2 \cdot C_6H_4 \cdot C_6H_4 \cdot NPh$ . D. F. T.

**Preservation of rubber.** I. WILLIAMS and A. M. NEAL, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,902,005, 21.3.33. Appl., 12.3.32).—The reaction product of  $H_3BO_3$  and an  $o\text{-}(OH)_2$ -aromatic compound such as pyrocatechol, used in the form of a salt, *e.g.*, of K,  $NH_4$ , or an org. base, retards the deterioration of rubber in which it is incorporated. D. F. T.

**Producing a hardened surface on rubber goods.** E. FRÖLICH (B.P. 400,001, 25.11.32. Ger., 27.11.31).—A layer of rubber containing an oxidisable oil (linseed oil) is applied to the surface of unvulcanised rubber sheet and the whole is vulcanised. The surface layer hardens on exposure to air. D. F. T.

**Automatic mill-mixing, warming, or the like working of rubber or the like, and apparatus therefor.** DUNLOP RUBBER Co., LTD., and E. E. QUINTON (B.P. 399,850, 15.3. and 19.8.32).

Disazo dyes for rubber.—See IV.

## XV.—LEATHER; GLUE.

**Relation of the fat content of hides to conditions of curing.** P. WHITE and F. G. CAUGHLEY (New Zealand J. Sci. Tech., 1933, 15, 163—168).—The fat of "greasy" hides differs from natural hide fat and resembles tallow oil. Application of pressure to fatty tissue adhering to hides squeezes out an oil. In the salt-curing process adhering fat restricts the penetration of salt and some bacterial putrefaction occurs, causing a loosening of the fibre structure. Slow curing is associated with slow reduction of the  $H_2O$  content of the hide. Under such conditions oil expressed from fatty tissue penetrates the hide, causing greasiness. A. G. P.

**Disposal of [hide and skin] fleshings.** F. O'FLAHERTY and E. K. MOORE (J. Amer. Leather Chem. Assoc., 1933, 28, 476—490).—Limed hide fleshings were ground, dried on wire-screen trays in an oven to reduce the  $H_2O$  to 8%, and the product was subjected to hydraulic pressure or solvent extraction (to remove a portion of the grease) and used as a poultry or pig food. It had the food val. of a 60% protein tankage. D. W.



**"Kidney grease" in heavy hides and leather.** R. W. FREY, I. D. CLARKE, and L. S. STUART (J. Amer. Leather Chem. Assoc., 1933, 28, 490—511).—Fat-staining (I) was increased by the heavy rolling of the leather. No connexion was traceable between the feeding of the animals and (I). Evidence was obtained of the presence of large quantities of fat in the fresh hide as distinct from the cured hide, and of the accumulation of kidney fat consequent on heavy feeding of the animals. The % fat in the hides was not much reduced by various soaking experiments. 35—56% of fat was removed by different experimental CaO liquors. The hides should be green-fleshed, limed with new liquors at a max. temp., and thoroughly scudded to reduce (I). (Cf. B., 1933, 980.) D. W.

**Removal of kidney grease stains from tanned sole leather.** J. S. ROGERS (J. Amer. Leather Chem. Assoc., 1933, 28, 511—525).—Grease-stained dry leather is immersed for 24—48 hr. in a fermented solution ("sour dip") of glucose and MgSO<sub>4</sub> at a temp. > 50° but sufficient to liquefy the grease, occasionally handled during the process, and the grease is run off from the liquor before the leather is removed. D. W.

**Examination of leather for the presence of extractable chromium compounds.** F. E. HUMPHREYS and H. PHILLIPS (Analyst, 1933, 58, 509—517).—The H<sub>2</sub>O extract of 13 samples of leather (I) after oxidation with HNO<sub>3</sub> and H<sub>2</sub>O<sub>2</sub> contained 0.0—0.120 mg. CrO<sub>3</sub> per g. (I) suspected of causing dermatitis could not be differentiated from normal (I). CrO<sub>3</sub> added to vegetable-tanned and semi-chrome (I) was almost completely, but only gradually, fixed, whereas full-chrome (I) fixed only 30% but the whole of the fixation occurred in 1 day. Solutions resembling sweat in composition did not extract much more Cr than did H<sub>2</sub>O, but acid solutions extracted somewhat > neutral ones. E. C. S.

**Determination of strength of animal glues.** M. PIEKARSKI (Przemysł Chem., 1933, 17, 220—223).—The quality of glues is a function of the greatest dilution at which gelation takes place; using this test, results are obtained for a no. of hide and bone glues analogous to those with the more complicated methods commonly employed. R. T.

**Working of artificial horn.** C. STARK (Gummi-Ztg., 1933, 47, 1327—1328, 1375—1376).—A general account is given of its manipulation, and the process of colouring it by dyes in hot or cold aq. acid media is described; a list of appropriate dyes is given. S. S. W.

**Treatment of H<sub>2</sub>O.**—See XXIII.

See also A., Nov., 1184, **Determination of gelatin.**

#### PATENT.

**Preparation of hides or skins for tanning.** J. R. GEIGY A.-G., Assees. of R. BIEDERMANN (B.P. 398,524, 15.2.32. Ger., 21.2.31).—Raw hides or skins are treated with an aq. extract containing a catheptic ferment at  $p_H$  5—7 and 37° with the addition of NaSH and/or H<sub>3</sub>BO<sub>3</sub>. D. W.

#### XVI.—AGRICULTURE.

**Sterilisation of soil by means of an electric current.** W. DIX and E. RAUTERBERG (Arch. Pflanzen-

bau, 1933, 10, 172—190).—Temp. records of soils heated by an electrical device are given. A. G. P.

**Use of electrometric  $p_H$  measurements for determining bacterial numbers in soils.** D. FEHÉR (Arch. Mikrobiol., 1933, 4, 257—270).—Under laboratory conditions, differences in the  $p_H$  of sterilised and natural soil samples permit the indirect determination of bacterial nos. A. G. P.

**Are variations in soil  $p_H$  due to microbiological or physico-chemical effects?** G. DEINES and R. KLEINSCHMIDT (Arch. Mikrobiol., 1933, 4, 271—279).—Soils dried in a vac. (30°/25 mm.) suffer no  $p_H$  change due to biological activity. A. G. P.

**Action of lime on soils and the correction of acidity.** R. CHAMINADE (Ann. Agron., 1933, 3, 453—477).—The rate of decrease in  $p_H$  of aq. Ca(OH)<sub>2</sub> following admixture with an acid soil is rapid during the first hr., declining subsequently and becoming very slow after 48 hr. Reacidification of soil so treated liberates much less Ca, up to a given  $p_H$ , than was absorbed by the original soil in the attainment of the same  $p_H$ . The bearing of this on determinations of the CaO requirement of soils is discussed. A. G. P.

**Influence of the lime condition of a soil on the assimilating power of plants for potassium and phosphoric acid.** W. LESCH (Z. Pflanz. Düng., 1933, 32, A, 20—32).—The availability of P and K as determined by the Neubauer and citric acid solubility methods is compared in soils in which reaction changes had been induced by fertiliser treatment. The physiological reaction of the fertiliser in itself had no influence on P solubility, but applications of CaO lowered the root-assimilable P content (Neubauer) without affecting the citric solubility. In manurial trials liming did not reduce the efficiency of superphosphate. Assimilation of K by plants was greater from physiologically alkaline fertilisers, and liming did not alter this relationship. The ratios Ca:P and Ca:K in crops were greatest under conditions producing smallest yields and were unaffected by the CaO status of the soil. A. G. P.

**Nitrogen reserves of soil and their mobilisation in the calcareous soils of Champagne.** LEBRUN and RADET (Ann. Agron., 1933, 3, 478—492).—Addition to soils of salts of Na, K, Mg, and NH<sub>4</sub> did not affect the rate of nitrification of org. N, but large dressings of S retarded the process. Treatment with CO<sub>2</sub>-saturated H<sub>2</sub>O facilitated nitrification by dissolution of Ca and liberation of org. N reserves. A. G. P.

**The phosphate question. VI. Determination of the phosphorus requirement [of soils] by chemical methods.** O. ARRHENIUS (Z. Pflanz. Düng., 1933, 32, A, 1—20; cf. B., 1931, 774).—Comparison is made in a large no. of soils of the citric acid-(I), lactate-(II), and H<sub>2</sub>O-sol. PO<sub>4</sub><sup>'''</sup> (III) contents, and results of field manurial trials. The (III) content bears no relationship to the P-nutrient status of soil. The (I) and (II) vals. are closely related to the cropping trials. Results are examined statistically and limiting vals. indicative of P deficiency are discussed. A. G. P.

**Determination of the phosphoric acid requirement of soils by means of *Aspergillus niger*.**



H. NIKLAS, G. VILSMEIER, and F. KOHL (Z. Pflanz. Düng., 1933, 32, A, 50—70).—No general relationship exists between the solubility of soil P and the  $p_H$  of the solvent solution, but for any individual acid (e.g., citric) the  $PO_4'''$  solubility increases with the  $p_H$ . The *Aspergillus* method gives results which in this respect resemble those of the purely chemical extraction methods. Comparison of the *Aspergillus*, Neubauer, and chemical methods is made in a no. of soils, and limiting vals. in the first-named method indicative of the P requirement of soils are assigned. A. G. P.

**Determination of the phosphoric acid requirement of soils.** Y. K. KUDZIN and E. N. SHUSTOVA (Nauk. Zapiski Tzuk. Prom., 1933, 10, No. 28, 63—68).—Truog's method was the most accurate of the six compared. During the test the room temp. should not vary by  $> 2-3^\circ$ . CH. ABS.

**Determination of manurial requirement [of soils] by means of the root-, citric acid-, and water-soluble phosphoric acid.** E. KNICKMANN (Z. Pflanz. Düng., 1933, 32, A, 84—95).—Comparison is made of vals. obtained for a no. of soils by the three methods. Correlation data are considered. A. G. P.

**Determination of readily soluble phosphate in cultivated soil.** H. EGNÉR (Kung. Landtbruks-Akad. Handl. Tidskr., 1933, 72, 30—63; Chem. Zentr., 1933, i, 3771—3772).—Two buffer solutions, (a) 0.005N- $H_2SO_4$  and 0.015N- $(NH_4)_2SO_4$  ( $p_H$  2.4), and (b) 0.02N-Ca lactate and 0.01N-HCl ( $p_H$  3.5), are employed for the colorimetric determination, the sample being shaken for 2 hr. with 250 c.c. 200 kg. of  $P_2O_5$  per hectare are provisionally regarded as the limit for soils poor in  $P_2O_5$ . A. A. E.

**Are surface applications of superphosphate equal to those incorporated with the soil?** C. KRÜGEL and C. DREYSPRING (Z. Pflanz. Düng., 1933, 12, B, 449—460).—Differences in crop yields resulting from the two methods of application were small. A. G. P.

**Movement of added phosphate in soil.** I. B. W. DOAK (New Zealand J. Sci. Tech., 1933, 15, 155—162).—The depth distribution of  $PO_4'''$  in fertilised soils indicates that in light sandy loams excessive leaching losses may occur and that these are greater when the fertiliser is applied in one heavy dressing than when divided into successive smaller applications. A. G. P.

**Determination of assimilable potash in soils of Indo-China.** T. T. KHOI (Ann. Agron., 1933, 3, 522—528).—In the cobaltinitrite method for determining K satisfactory conditions for the pptn. and filtration of the K salt necessitate the complete removal of Fe, Al, and org. matter. The acid soil extract is partly evaporated with a little  $H_2O_2$ , neutralised with NaOH, and filtered. The filtrate is acidified with AcOH and evaporated to dryness on a sand-bath. The aq. extract of the residue is filtered, acidified with AcOH, treated with the customary cobaltinitrite reagent, and the whole evaporated to dryness. The residue is again extracted with  $H_2O$  and the ppt. collected, washed with AcOH and finally with  $(NH_4)_2SO_4$  solution, and titrated with  $KMnO_4$ . In the preliminary removal of Fe and

Al, NaOH may be replaced by aq.  $NH_3$ , in which case the residue is calcined to remove  $NH_3$  and org. matter prior to pptn. of the cobaltinitrite. A. G. P.

**Absorption of potassium by plants as affected by decreased exchangeable potassium in the soil.** E. L. PRÖBSTING (J. Pomology, 1933, 11, 199—204).—The K-supplying power of soils cannot be measured by the amount of  $H_2O$ -sol. or exchangeable K contents. Non-exchangeable K may be an important source for plants. Different species of plants vary considerably in their ability to obtain K from soil. A. G. P.

**Effect of potassium on the higher plants.** A. RIPPPEL, G. BEHR, and R. MEYER (Z. Pflanz. Düng., 1933, 32, A, 95—109).—The nature of the growth curve of oats with varying N supply differs from that with varying K supply. K deficiency produces similar conditions in the plants to those given by excessive N feeding: there is a high N accumulation. The  $H_2C_2O_4$  contents of certain plants is not influenced by lack of K. Relationships between K supply and carbohydrate metabolism are discussed. A. G. P.

**Comparison of laboratory methods of soil examination with field manurial trials.** W. WÖHLBIER and SCHULZE (Z. Pflanz. Düng., 1933, 12, B, 460—468).—Dirks' method for determining the available K in soil agreed with the results of field trials only in 50—60% of the cases examined. Better agreement was obtained with Neubauer tests for certain crops. A. G. P.

**Effect of nitrogenous fertilisers on growth and yield of wheat and barley in South Australia. I. Wheat grown after fallow and after stubble.** A. E. V. RICHARDSON and H. C. GURNEY (Empire J. Exp. Agric., 1933, 1, 193—205).—On well-worked bare fallow, wheat did not respond to dressings of  $(NH_4)_2SO_4$  (I) during seasons in which rainfall prior to sowing was light. Increased yields resulted when heavy rain preceded sowing. The  $NO_3'$  present in soils in the two seasons was in accord with the cropping results. The benefit of fallowing is associated more with  $NO_3'$  accumulation than with  $H_2O$  conservation. Among customary N fertilisers  $NaNO_3$  gave highest increases. Urea, "nitro-chalk," and (I) showed similar efficiencies and  $(NH_4)_2HPO_4$  (II) was definitely inferior. Superphosphate and (II) gave better results when drilled with the seed than when broadcast. The method of distribution did not affect the action of (I). The effects of (I) on the growth of wheat included slightly depressed establishment of plants, vigorous tillering with an increased no. of ear-bearing tillers, slightly increased length of ears, weaker and more sappy growth, decreased % of grain in total dry matter, and increased weed growth. The natural N accumulation in fallow soils produced sturdier tillers with a greater survival rate at harvest than did (I). A. G. P.

**Influence of microflora of the soil on growth of wheat.** G. TRUFFAUT and M. LEFOUIN (Compt. rend., 1933, 197, 787—789).—Relative to fallow soil (control) the bacterial content of soil in which wheat is growing is increased by approx. 30% ( $2150 \times 10^6$  to  $3690 \times 10^6$  per g.) up to harvesting, and then diminishes again by 49% (to  $1890 \times 10^6$  per g.; fallow soil,  $1750 \times 10^6$ ). J. W. B.



**Rice fertilisation.** L. C. KAPP (Arkansas Agric. Exp. Sta. Bull., 1933, No. 291, 37 pp.).—In soils examined, application of P fertilisers did not increase the yield of rice nor the  $[\text{PO}_4^{''}]$  of 1:5  $\text{H}_2\text{O}$  extracts of the soils. The  $\text{PO}_4^{''}$  content of the plants declined in cases where manurial treatment produced increased yields, but was unaffected by heavy liming. The % Ca in plants was not closely related to their  $\text{PO}_4^{''}$  contents. Irrigation of soils after treatment with  $(\text{NH}_4)_2\text{SO}_4$  or  $\text{NaNO}_3$  resulted in considerable losses of N. No accumulation of  $\text{NO}_2^-$  occurred in submerged soils. Addition of  $\text{Ca}(\text{OH})_2$  to soils decreased their total N content, but increased that of the plants. The effect of fertilisers on the relative production of grain and straw and on the proportion of sterile glumes is recorded. Incorporation of rice straw and roots with soil decreased ammonification.

A. G. P.

**Influence of season and of ammonium sulphate on the chemical composition of perennial rye grass and of white clover.** H. O. ASKEW (New Zealand J. Sci. Tech., 1933, 23, 143—154).—Variations in composition due to growth conditions are recorded. Drought conditions lower the % P, N, K, and sol. ash, but not that of Ca and  $\text{SiO}_2$ , in the plants. White clover is less affected by seasonal changes than is rye grass. With both plants the increased N content following applications of  $(\text{NH}_4)_2\text{SO}_4$  (I) is mainly confined to the first samples taken after treatment. Dressings of (I) on rye grass increase the K, sol. ash, total and inorg. S contents and reduce that of Ca, P, and  $\text{SiO}_2$  to a small extent. In the case of white clover Ca, P, N, and sol. ash increase after each application of (I), but subsequently fall to vals. < those of untreated samples. The K content is slightly lower in N-treated clover. The N:P<sub>2</sub>O<sub>5</sub> ratio in rye grass decreases and in clover increases during the summer season.

A. G. P.

**Correction of the unproductivity of a peat soil for lettuce.** B. D. WILSON and G. R. TOWNSEND (J. Amer. Soc. Agron., 1933, 25, 523—527).—Liming peat soils prevented chlorosis and leaf curl in lettuce seedlings. Seed treatment with CuO increased germination and reduced damping-off. Addition of  $\text{CuSO}_4$  was less effective in this respect. Treatment of field soils with CaO and  $\text{CuSO}_4$  produced a normal crop.

A. G. P.

**Inoculation of legumes as related to soil acidity.** W. A. ALBRECHT (J. Amer. Soc. Agron., 1933, 25, 512—522).—In soils having  $p_{\text{H}} < 5.0$ , nodulation failure is mainly controlled by acidity. In less acid soils failure may result from deficiency of available Ca.

A. G. P.

**After-effects of the nutrition of seedlings on the growth of rutabagas.** W. STEINMETZ (Arch. Pflanzenbau, 1933, 10, 129—171).—Relatively excessive manuring of seed beds in P-rich soils with urea primarily favoured leaf development in the seedlings. Root growth was also stimulated, N assimilation was increased, the % dry matter (A) in the plants declined, and dry-matter production per unit N intake was accelerated. K manuring improved the general development of the plants and reduced A, especially in the leaves. In some cases the % N in leaves was reduced, but the N intake of roots was increased. The effect of manuring on the morphology of plants is examined. The form of the roots

produced is correlated with the nutrient status of the seed bed.

A. G. P.

**Influence of the soil carbonic acid on growth of sugar beets.** P. J. H. VAN GINNEKEN, K. DE HAAN, and J. R. BRUINSMA (Meded. Inst. Suikerbiet., 1933, 3, 61—110).—An extensive investigation of the amount of  $\text{CO}_2$  produced by soils on which beets of varying yield were grown showed that whilst some of the fields produced more  $\text{CO}_2$  than others the variation had no apparent relation to the yield of beets. In other experiments  $\text{CO}_2$  was released under the beet leaves at the rate of 1 g. per sq. m. day and night from July 11th to the end of Sept., but no significant difference in the yields of beets or of sugar was found.

J. P. O.

**Action of different nitrate fertilisers on sugar-beet yields.** PAASCH (Zuckerrübenbau, 1933, 15, 96—98).—Experiments were made on soils having  $p_{\text{H}}$  7.0—7.7, using Leuna nitrate,  $\text{Ca}(\text{NO}_3)_2$ ,  $\text{NaNO}_3$ ,  $\text{NH}_3$ -Ca nitrate, and Chili saltpetre as side-dressings. Basic fertilisers succeeded best in wet years on alkaline soils, but those having physiologically acid reactions gave better results in dry years.

J. P. O.

**Importance of boron for sugar beet.** E. V. BOBKO and M. A. BELVOUSSOV (Ann. Agron., 1933, 3, 493—504).—Optimum growth of sugar beet occurred in nutrients containing 5 mg.  $\text{H}_3\text{BO}_3$  (I) per litre. In soils (I) counteracted the ill effect of excessive liming on the growth of beet.

A. G. P.

**Inheritance of sugar content and weight in hybrids from sugar beets and fodder beets.** V. STEHLÍK (Chim. et Ind., 1933, 29, Spec. No., 1127—1140).—In hybrids from sugar beets and bulbous fodder beets the sugar content showed a very clear correlation with morphological and physiological characters such as shape, size, and colour of roots, no. of vascular rings, shape of hypocotyl, and wt. of foliage, and like these it was transmitted according to Mendelian laws. In both first and second generations of hybrids the average vals. for the sugar content and related characters were rather closer to those for the parent sugar beets than to those for the fodder beets. (Cf. Colin, B., 1933, 839.)

J. H. L.

**Commercial ripening of bananas in New Zealand. II. Practical ripening on a small scale.** J. B. HYATT and C. W. O. TURNER (New Zealand J. Sci. Tech., 1933, 15, 117—127).—Details are given of the structural equipment, and means of controlling the temp., R.H., ventilation, and % of coal gas used in the ripening chamber.

A. G. P.

**Influence of culture plants, mineral fertilisation, and acidity on the C:N ratio in soil and on the composition of the soil humus.** T. BLOK (Roczn. Nauk. Roln. leśn., 1933, 29, 271—288; Chem. Zentr., 1933, i, 3485).—Experiments on a loess soil rich in P and poor in K at  $p_{\text{H}}$  5—7 disclosed no influence on the C:N ratio (9.6:1) or on the org. N. Beets decrease the hemicellulose (I) in soil humus (II). An acid reaction favours (I) and cellulose, and mineral fertilisation increases the (I) in (II).

A. A. E.

**Rôle of thermophilic bacteria in the preparation of artificial farmyard manure.** A. DUNÉZ (Ann.



Agron., 1933, 3, 504—521).—Organisms causing humification of cellulose at 65° are described. The process is markedly affected by N supplies, notably the degradation products of proteins. Acidity developed is greater under anaërobic than under aërobic conditions.

A. G. P.

Danish trials of autumn and spring manuring with cattle manure. K. IVERSEN (Arch. Pflanzenbau, 1933, 10, 203—236).—Losses in manurial val. following application in spring and in autumn are compared in various soil types. Winter losses affect only the following crop, the residual action on second and third crops being the same irrespective of the time of application. Effects of climatic conditions on the volatilisation of  $\text{NH}_3$  and on the leaching of  $\text{NO}_3$  from manure are recorded. Liquid manure applied in spring had a similar action, with reference to its N content, to stall manure. A definite after-effect on the second and third succeeding crops is established.

A. G. P.

Colloidal resin as a wetting agent and adhesive [for fungicides].\* A. PALLIER (Compt. rend. Acad. Agric. France, 1933, 19, 543—547).—Effective results were obtained by the addition of small amounts of a colloidal solution (50%) of colophony to Cu sprays.

A. G. P.

Increasing the activity of plant materials containing rotenone. N. SCHMITT (Anz. Schädlingsk., 1933, 9, 2 pp.; Chem. Zentr., 1933, i, 3617).—A mixture of *Derris* root extract and rotenone has a greater effect on goldfish than would be anticipated from consideration of the constituents alone.

A. A. E.

Casein-ammonia, a practical emulsifying agent for preparation of oil emulsions by orchardists. E. J. NEWCOMER and R. H. CARTER (J. Econ. Entom., 1933, 26, 880—887).—A satisfactory prep. contains oil 100 gals.,  $\text{H}_2\text{O}$  33 gals., casein 3 lb., aq.  $\text{NH}_3$  (28%) 1 quart.

A. G. P.

Biology and control of *Chrysomphalus dictyospermi* (Morg.). A. W. CRESSMAN (J. Econ. Entom., 1933, 26, 696—706).—Complete control was obtained by the use of 1.0—1.3% mineral oil emulsions prepared with K fish-oil soap.

A. G. P.

Efficiency of lubricating and tar oil emulsions against scurfy scale (*Chionaspis furfura*, Fitch). L. E. AULL and R. W. DEAN (J. Econ. Entom., 1933, 26, 912—913).—Dormant spraying with 6% lubricating oil emulsions or 6% tar oil washes killed 93% and 96%, respectively, of the egg masses.

A. G. P.

Spraying to control gladiolus thrips (*Taniothrips gladioli*, M. & S.) in Michigan, 1932. E. I. MCDANIEL (J. Econ. Entom., 1933, 26, 835—836).—A Pb arsenate-*Derris* prep. containing glue gave best results.

A. G. P.

Detection of traces of arsenic in presence of mercury in grain from pickled seed. H. KÜHL and B. CZYZEWSKY (Z. ges. Getreide-Mühlenw., 1933, 20, 68—70; Chem. Zentr., 1933, i, 3812).—The material is treated with HCl and  $\text{KClO}_3$  and  $\text{Cl}_2$  is removed from the solution by means of a current of air. Zn foil is then added; Hg alone gives a white deposit, As alone a greyish-brown to brownish-black deposit. As + Hg

gives a brown deposit, the liquid becoming yellowish-brown.

A. A. E.

Control of *Dendroctonus valens*, Lec. E. WALTHER (J. Econ. Entom., 1933, 26, 828—831).—A method of  $\text{CCl}_4$  injection is described.

A. G. P.

Insecticidal action of vegetable oils utilised in the winter treatment of scale insects on forest trees. M. A. BALACHOWSKY (Compt. rend. Acad. Agric. France, 1933, 19, 497—508).—“Quick-breaking” emulsions ( $\geq 2\%$ ) of arachis or colza oils prepared with  $\text{NH}_4$  oleate gave high % control. \* Injury to buds was much  $<$  that caused by carbolineum washes.

A. G. P.

Mechanism of the action of copper compounds on [vine] mildew. RIBEREAU-GAYON (Compt. rend. Acad. Agric. France, 1933, 19, 550—555).—Cu is absorbed by cells from very dil. solutions and displaces other bases (K, Mg, Ca) from insol. salts of org. acids within the tissues. Renewal of [Cu<sup>++</sup>] in the external solution, e.g., contact with solid particles in Cu fungicides, permits a continuance of the displacement process until the Cu content of the cell reaches injurious proportions.

A. G. P.

Control of root nematodes (*Heterodera Schachtlii*, Schm.) by means of stimulants. B. RADEMACHER and O. SCHMIDT (Arch. Pflanzenbau, 1933, 10, 237—296).—Substances stimulating the activation of cysts in soil were, in general, oxidising agents or distillation products of org. materials. Many reducing substances, nitrites, and PhOH derivatives were toxic. In many cases substances which were stimulative in certain ranges of concn. were toxic in others. Partial disinfection of soils was obtained by treatment with activating materials.

A. G. P.

Fumigation with propylene dichloride mixture against *Pyrausta nubilalis*, Hubn. C. B. DRIBBLE (J. Econ. Entom., 1933, 26, 893—895).—A 9:1 mixture of  $\text{C}_3\text{H}_6\text{Cl}_2$  and  $\text{CCl}_4$ , applied at the rate of 2 lb. per 120 cu. ft. for 24 hr., successfully controlled the borer in maize stems.

A. G. P.

Effects of various commercial calcium arsenates on bean foliage. N. F. HOWARD and F. W. FLETCHER (J. Econ. Entom., 1933, 26, 914).—Injury to bean foliage by Ca arsenate may be corrected by additions to the spray liquid (I) of CaO, CaO + S, or Bordeaux mixture. The proportion of  $\text{H}_2\text{O}$ -sol. As in (I) is not an index of foliage injury; this is influenced to a large extent by the rate of evaporation of (I) from the leaves.

A. G. P.

Naphthalene as a fumigant against the peach tree borer (*Aegeria exitiosa*, Say.) and sod insects. J. R. STEAR (J. Econ. Entom., 1933, 26, 903—906).—Current methods are discussed.

A. G. P.

Determination of nicotine and pyridine [in nicotine soap sprays] in presence of one another. L. GISIGER (Mitt. Lebensm. Hyg., 1933, 24, 42—53; Chem. Zentr., 1933, i, 3772).—Nicotine is pptd. from a dil., and both bases are pptd. from a conc., solution as silicotungstate; the ppts. are weighed and the wt. corr. for solubility.

A. A. E.

Determining moisture.—See I. [Citric acid] solubility of phosphates. Reactions of S at low temp.



—See VII. Sugar-cane  $P_2O_5$ .—See XVII. Parasitoides containing nicotine.—See XX.

See also A., Nov., 1133, Determining Mg. 1135, Soil reaction measurements with the Sb electrode. 1138, Malayan soils. 1201, Determining peroxidase in agricultural products. 1214, Recording catalase activity of plant tissues. 1216, Dihydrorotenone.

### XVII.—SUGARS; STARCHES; GUMS.

**Phosphate content of [sugar] cane juices in Mauritius.** N. CRAIG (Rev. agr. Maurice, 1933, No. 69, 99—104).—Different cane varieties may contain very different amounts of  $P_2O_5$ , the content of which may vary in a given variety from one locality to another. In the case of White Tanna, the variation was found to be 0.0044—0.08%. This variety did not react to applications of phosphate when the  $P_2O_5$  content of its juice was 0.032—0.035%, but increases were obtained when the content was only 0.0037%. J. P. O.

**Rationalising beet drying.** I. B. MINTZ, G. S. BENIN, N. P. BOLOTOV, A. A. MAKSIMOV, and I. I. SAENKO (Nauk. Zapiski Tzuk. Prom., 1932, 9, No. 16—17, 1—11).—On liming cosettes before drying to  $p_H$  6.9—9.0 the sugar loss varies within the same limits as in drying without liming. Drying is facilitated by liming, and the cosettes contain less reducing substance.

CH. ABS.

**Effects of glucose and fructose on the sucrose content in potato slices.** J. M. NELSON and R. AUCHINCLOSS (J. Amer. Chem. Soc., 1933, 55, 3769—3772).—Contrary to Schroeder *et al.* (A., 1922, i, 906; 1931, 1197), Waterman (Chem. Weekblad, 1914, 11, 332), and de Wolff (A., 1926, 1183; 1927, 80), desiccation of potato slices is not necessary in order to effect marked increases in the sucrose (I) content. When slices are dipped in aq. glucose or fructose and then kept in moist air, a larger increase in (I) occurs than by desiccation; in  $N_2$ , little or no increase occurs (desiccation in  $N_2$  gives similar results), showing that  $O_2$  is necessary for the synthesis of (I). H. B.

**Control of the final carbonatation [of beet juice].** K. SOLON (Deut. Zuckerind., 1933, 58, 441—442).—The optimum alkalinity for the second carbonatation has been found to be just half the natural alkalinity figure, so that it is convenient to determine the latter val. and divide it by 2. J. P. O.

**Standard alkalinity and calcium salts in beet-sugar production.** A. M. PSHENICHNI and B. P. SHUMKOV (Nauk. Zapiski Tzuk. Prom., 1933, 10, No. 27, 133—147).—At Tzuibulevski the natural alkalinity was 0.018—0.034% CaO; the remaining CaO was 0.002—0.005%. The second carbonatation must be periodical in order to maintain uniform alkalinity.

CH. ABS.

**Boiling of [sugar] syrups to masseccutes. Evaporation of viscous solutions.** H. CLAASSEN (Z. Ver. deut. Zucker-Ind., 1933, 83, 615—660).—From data on the boiling of masseccutes of purities 96, 91, and 82%, in 3 calandria pans having vertical steel tubes 4 in. wide and 3 ft. high and a central downtake, using steam at  $118^{\circ}/0.25$  atm. in the pans, the author computes rates of evaporation, heat-transmission coeffs.,

and rates of circulation, at various stages. During the second half of the boiling process the temp. difference of  $53^{\circ}$  between the heating steam ( $118^{\circ}$ ) and that leaving the pan ( $65^{\circ}$ ) is accounted for thus: b.-p. elevation due to solids in solution 13— $23^{\circ}$ , further elevation of b.p. at the heating surface due to the static head of masseccute ( $> 6$  ft. at the final stages) 18— $23^{\circ}$ , and a balance of 10— $22^{\circ}$  representing the temp. difference at the two sides of the heating surface. From the last figures transmission coeffs. are calc. in the ratio 4:2:1 for the masseccutes of purities 96, 91, and 82% during the main boiling period, with a rapid fall at the end. The temp. of a charge in a vac. pan during boiling is practically the same at all depths as at the free surface, differing from that of  $H_2O$  only by the elevation due to the solids in solution, yet owing to the static head the b.p. at the heating surface must be about  $20^{\circ}$   $>$  this general masseccute temp. during the later stages. The viscosity of masseccutes at the temp. of discharge is 12—23 times that of  $H_2O$ , vol. for vol., or 18—34 times, wt. for wt., and the amount of syrup present corresponds only to a uniform film about 0.17 mm. over the surface of the crystals. In such a material convection currents are practically impossible and transmission of heat is mainly mol. and too slow to effect condensation of steam passing through it. The superheated steam bubbles rising from the calandria of a vac. pan are cooled, not by communication of heat to the masseccute, but mainly by expansion against diminishing pressure as they rise. At the free surface they have about twice their original vol. and escape at about the temp. of the masseccute, *e.g.*,  $20$ — $24^{\circ}$  hotter than the saturated vapour above the charge. Circulation of masseccute in the pan and velocity of flow in the tubes are considered, and means for increasing the efficiency of the calandria are discussed. J. H. L.

**Conductometric control of [sugar] boiling.** S. STARE (Z. Ver. deut. Zucker-Ind., 1933, 83, 807—821).—Improved boiling of refinery syrups resulted from control by means of Honig and Alewijn's apparatus (B., 1933, 361) modified to suit the low conductivities of the syrups. The electrode surface was increased and gilded, and by means of a variable resistance the initial reading for each boiling was adjusted to 100 milliamp. J. H. L.

**Control of sugar boiling by means of electrical conductivity.** III. O. SPENGLER, F. TÖDT, and J. WIGAND (Z. Ver. deut. Zucker-Ind., 1933, 83, 822—832; cf. B., 1933, 38, 87, 406).—Reference is made to recent papers by Smith (B., 1933, 361), Alewijn (Archief, 1933, 309, where errors due to stray currents from the electrodes to the pan walls are discussed), and Stare (preceding abstract). Adjustment of the initial current to a fixed val., as adopted by Stare for refinery syrups, is not recommended for raw syrups, which vary considerably in Brix. The circuits of electrical apparatus for pan control made by Siemens & Halske and by Wösthoff are described; the former indicates current in milliamp. and the latter conductivity in microsiemens. Control can be carried out with readings of current only, but to compare the working of different pans these readings must be calc. to conductivities, and a method



of calibrating the electrodes for this purpose is described. The electrodes designed by the authors are now made of Cr-Ni steel instead of Cu. J. H. L.

**Clarification of sugar solutions for polarisation, using the wet and dry methods.** K. ŠANDERA (Z. Zuckerind. Czechoslov., 1933, 58, 49—56).—Raw beet sugars were polarised after clarification with (a) basic Pb acetate (I) solution (2 c.c.  $\equiv$  0.7 g.) before making up to the mark; and (b) 2 g. of dry, powdered (I), added after completing to vol. (Horne's method). The averages of 14 determinations were: (a) 96.11, (b) 96.01. Polarisation of artificial raw sugars showed the dry method of clarification to give results very close to the actual sucrose content, and the wet method about 0.1% higher. The dry method is pronounced very practical, and more effective in clarification than the solution of the same Pb content. J. P. O.

**Sweetening-off the filter-presses.** H. A. SCHLOSSER (Centr. Zuckerind., 1933, 41, 424—425).—To attain a low % of sugar in the cake with min. consumption of wash- $H_2O$  the most satisfactory method is slow filtration ( $1\frac{1}{2}$  hr. per press) with separation of the rich from the dil. sweet-waters, the latter being used for the first washing of the next press, which separation can be effected automatically. Where the filtering capacity is limited, and filtration must be effected in short cycles, there is a tendency for the cake to crack. In these circumstances the Staněk method (patented) of following the juice with a suspension of filter mud in  $H_2O$  or dil. sweet- $H_2O$  should be used. It makes possible the reduction of the sugar content of the cake to < 0.5%. J. P. O.

**Affinability [of raw beet sugars].** J. DĚDEK and F. DOLÁK (Z. Zuckerind. Czechoslov., 1933, 58, 41—44).—Samples of artificial raw sugars (white crystals of known size of grain covered with molasses) were affined by the Berlin (A) and Prague (B) (Šandera) methods. Using method A, the no. of crystals ( $n$ ) remained almost unaltered, and about 90% of the white crystals remained unaltered in size, or practically so. On the other hand, in method B,  $n$  was 50% less and the sizes of the fractions had much diminished, due to the much greater amount of  $H_2O$  used for washing. J. P. O.

**Determination of invert sugar in sugar-factory products.** R. OFNER (Chim. et Ind., 1933, 29, Spec. No., 1125—1126).—The defects of Herzfeld's method are discussed and reference is made to several proposed substitutes, including the author's (B., 1929, 832; 1932, 279). J. H. L.

**Conditions for the most accurate and simple determination of invert sugar in presence of sucrose.** O. SPENGLER and F. TÖDT (Z. Ver. deut. Zucker-Ind., 1933, 83, 833—845).—A preliminary survey of the problem how best to determine invert sugar in raw and refined sugars. Replacement of Fehling's solution by a reagent less affected by sucrose is considered the most promising line of study, and some experiments with Ost's, Müller's, and Kraisy's solutions are described. J. H. L.

**Detection and determination of fructose in presence of glucose.** F. FISCHL (Chim. et Ind.,

1933, 29, Spec. No., 1123—1124).—10 c.c. of sugar solution containing < 0.5% of sugar are mixed with 30 c.c. of Ofner's Cu reagent (B., 1929, 832; 1932, 279) in a wide-necked flask, heated in a  $H_2O$ -bath at 60° for 5 min. with slow motion, and then cooled rapidly. Reduction indicates the presence of fructose, which is determined by adding HCl to dissolve  $Cu_2O$  and titrating with  $N/77-I$ ; 1 c.c.  $\equiv$  5 mg. of fructose. J. H. L.

**Herzfeld's method of determining reducing sugars compared with the iodometric method.** R. OFNER (Z. Zuckerind. Czechoslov., 1933, 58, 33—35).—Compared with the standard Herzfeld method of determining invert sugar, the author's iodometric procedure (B., 1932, 279) is claimed to be not only simpler and quicker, but much more accurate: Filtration of  $Cu_2O$  is avoided, the results are independent of the amount of sucrose present, and no special tables are required. J. P. O.

**Effect of certain preservatives on the determination of sucrose by the invertase method.** C. F. POE, M. COOLEY, and N. F. WIRT (Ind. Eng. Chem. [Anal.], 1933, 5, 309—310).— $CH_2O$  (I),  $NaHSO_3$  (II),  $Na_2B_4O_7$  (III), Na salicylate, NaOBz (IV), and  $H_3BO_3$  (V) have no effect on such determinations when present in amounts  $\nless$  those ordinarily used in food products. In amounts  $\gt$  these, (I), (IV), and (V) affect the action of invertase, (II) decreases the  $[\alpha]$  of glucose, and (III) that of fructose. E. C. S.

**Report of Sub-Committee on development of a volumetric copper reduction method for sugar determinations.** J. T. FLOHIL (Cereal Chem., 1933, 10, 471—476).—Details are given of a method for the determination of Lintner diastatic power based on Schoorl's volumetric Cu reduction method of determining sugar. E. A. F.

**Determination of viscosity coefficients of viscous liquids, and application of Le Chatelier's formula.** B. DERJAGUINE (J. Chim. phys., 1933, 30, 548—555).—The variation of viscosity of potato molasses and 42% sugar solution in glycerol has been determined as a function of temp. Le Chatelier's formula is valid. E. S. H.

**Boiler  $H_2O$  containing sugar.**—See I. Active carbons.—See II. Sugar beets.—See XVI. Starch and EtOH yields of cereals. Yeasts and EtOH yield.—See XVIII. Storage of beet slices. Artichoke syrup.—See XIX. Beet-sugar waste-waters.—See XXIII.

See also A., Nov., 1117, Peptisation of native starches. 1134, Tyndallmetric examination of [sugar] liquors. 1146, Micro-detection of fructose. 1205, Effect of ultra-violet rays on molasses fermentation. 1215, Aromatic constituent of maple sugar products. Latex of the lac tree of Cambodia. 1217, Determination of carbohydrates in leaves.

## PATENT.

Starch-degrading enzymes.—See XVIII.

## XVIII.—FERMENTATION INDUSTRIES.

**Selection of yeast.** M. SCHÜTZA (Woch. Brau., 1933, 50, 368—370).—From 12 different yeasts were selected 2 bottom-fermentation flocculating yeasts which gave



a beer with very pure taste and full, vinous aroma, and 3 bottom-fermentation powdery yeasts which gave a very mild beer with mild aroma. The same final attenuation was obtained with all 12 yeasts (including 2 top-fermentation yeasts), thus supporting the results of Isotti (B., 1933, 648). R. H. H.

**Flocculating substances produced from brewery yeast.** F. STOCKHAUSEN and K. SILBEREISEN (Woch. Brau., 1933, 50, 349—351, 357—362, 365—368).—When washed or alkali-treated yeast is stirred with H<sub>2</sub>O at 0° a type of autolysis occurs after 24 hr. which produces in the H<sub>2</sub>O various substances having a flocculating effect on yeast suspensions. The effectiveness of the "yeast-H<sub>2</sub>O" (I) depends on the time of prep. and the amount employed, and is due, not to substances adsorbed from the wort on the surface of the yeast, but mainly to glycogen (II) and yeast gum (III). (III) is less active but is produced in greater amounts than (II). Other constituents of (I) which do not contribute to the flocculating effect are coagulable proteins, nucleoproteins, NH<sub>2</sub>-acids, e.g., tyrosine, tryptophan, histidine, and proline, and adenine. R. H. H.

**Improvements in yield [of alcohol] by use of various yeasts.** R. PIQUE (Chim. et Ind., 1933, 29, Spec. No., 1168—1172).—In wine-making, suppression of undesirable organisms and improvement in bouquet and EtOH content may be attained by use of vigorously fermenting cultures of selected yeasts. Suggestions are made as to the choice of yeasts for different types of wine. Selected wine yeasts (*S. ellipsoideus*) are recommended for the making of cider and vinous beverages from honey (hydromel) and also for use in distilleries operating on apples, sugar beets, and molasses; apart from bouquet, very rapid fermentations and high yields of EtOH can be attained with them. J. H. L.

**Air content of ethyl alcohol.** K. R. DIETRICH and W. LOHRENGEL (Z. Spiritusind., 1933, 56, 232—233).—The solubility of air and O<sub>2</sub> in EtOH-H<sub>2</sub>O mixtures, in C<sub>6</sub>H<sub>6</sub>, and in benzine (d<sub>15</sub> 0.725, 90% boiling between 96.1° and 100.8°) was measured. Air is about 7.7 times, and O<sub>2</sub> about 7.1 times, as sol. in EtOH as in H<sub>2</sub>O at 19°. H. J. E.

**Sulphurous acid in wine manufacture. III. Dissociation of combined sulphur dioxide in grape musts and wines.** L. MOREAU and E. VINET (Ann. Falsif., 1933, 26, 454—463; cf. B., 1928, 462).—Part of the bound SO<sub>2</sub> in must and wine is capable of dissociating after removal of free SO<sub>2</sub> (I). The fraction available for maintaining antiseptics =  $(l-a)(100-R)/R$ , where  $l$ =(I),  $a$ =concn. of SO<sub>2</sub> necessary for antiseptics, and  $R$ =the index of partial combination. The rate and degree of dissociation increase with rise in temp. and with increased dilution and alkalinity. The latter fact is made use of in accelerating the determination of the  $R$  and  $T$  indices (cf. B., 1927, 665). E. C. S.

**Detection of caramel in sweet wines.** H. MASTBAUM (Z. Unters. Lebensm., 1933, 66, 254—258).—Jägerschmid's method (B., 1909, 376) is not applicable to the detection of adulteration with caramel of wines of the type of Moscatel de Setubal, the colour of which is due to natural caramel. E. C. S.

**Composition of grape and apple sweet musts and their arsenic, copper, and zinc contents.** C. VON DER HEIDE and K. HENNIG (Z. Unters. Lebensm., 1933, 66, 321—338).—Analytical data, particularly with respect to Zn, Cu, and As, are given for sterilised and pasteurised grape and fruit sweet musts. Legal upper limits are suggested for the Zn, Cu, and As contents. Clarification with K<sub>4</sub>Fe(CN)<sub>6</sub> is indispensable to the manufacturer, and should be allowed. In certain samples too high contents of EtOH and SO<sub>2</sub> were found. E. C. S.

**Determination of arsenic, phosphoric acid, copper, zinc, iron, and manganese in must and wine.** C. VON DER HEIDE and K. HENNIG (Z. Unters. Lebensm., 1933, 66, 341—349).—Simple colorimetric methods are described for the determination of As, Cu, Fe, Mn, and P<sub>2</sub>O<sub>5</sub>. The methods hitherto employed require larger vols. of material and are more laborious. For Zn a gravimetric method is retained since the appearance of the incandescent oxide is an indication of its purity. E. C. S.

**Tables for determining the alcohol and extract contents of wine from the specific gravity of alcohol-water mixtures and of sucrose solutions at 20°, referred to water at 4°.** C. VON DER HEIDE and H. MÄNDLEN (Z. Unters. Lebensm., 1933, 66, 338—341). E. C. S.

**Regulations for control of sweet fruit musts and fruit syrups.** A. BEYTHIEN (Z. Unters. Lebensm., 1933, 66, 101—118).—Legal definitions are proposed for "sweet fruit must" and "fruit syrup." E. C. S.

**Alcoholic standard for cider.** D. W. STEUART (Analyst, 1933, 58, 602—603).—Ciders on the market contain 2—10 vol.-% of EtOH. The quantity depends on (a) the sugar content, which must be kept high if a sweet cider is required; and (b) the N content (0.01% for cider apple juice and 0.018% for ordinary apple juice), which must be > 0.006% at the beginning of fermentation to avoid slow fermentation and cider sickness, but must subsequently be reduced to < 0.001% by removal of the yeast crops in order to obtain true stability in bottle after carbonating. J. G.

**Vitamins in fermented and artificial vinegars.** J. KRÍŽENECKÝ and M. NEVALONNYI (Z. Unters. Lebensm., 1933, 66, 278—288).—Fermented vinegar (I) contains vitamin-B<sub>1</sub> and -B<sub>2</sub>. Practically useful amounts of vitamin-C could not be detected. The activity of the vitamins was obscured by the harmful physiological effects of AcOH, particularly in the case of -B<sub>1</sub> and -D. The last two could not be detected in artificial (I). The differences in dietetic val. of fermented and artificial (I) are due to differences in content of nutritive substances rather than to differences in vitamin content. E. C. S.

**Sardinian fermented milk.** N. BAIARDO and E. LUCCHETTI (Annali Chim. Appl., 1933, 23, 396—404).—The prep. and composition of "mizzuraddu" made from sheep's milk are described, and analytical standards, for detecting adulteration or alteration, are suggested. T. H. P.

**Determination of alcohol yield of cereals and polarimetric determination of starch.** M. RÜDIGER



(Z. Unters. Lebensm., 1933, 66, 59—62).—The material (25 g.) is sterilised in the autoclave and then treated with successive quantities of kiln-dried malt, for 15—20 min. at 75—80° and 2—2½ hr. at 58—60°. 0.75 c.c. of 10% CH<sub>2</sub>O and 2 g. of purified pressed yeast are added, and fermentation is allowed to proceed to completion at 28°. For maize and dari the EtOH yield differs from that calc. from the starch content by only 0.3 litre per 100 kg. Rye gives too high (66 litres), and potato flakes give too low (60 litres), a yield under these conditions. E. C. S.

Treatment of H<sub>2</sub>O.—See XXIII.

See also A., Nov., 1201, Determining peroxidase. Separation of enzymes. 1202, Enzymes of myrosinase. 1203, Phosphamidase. 1204, Enzymes of lactose fermenters. Conversion of alcoholic into lactic fermentation. Transforming CH<sub>3</sub>Ac·CHO by yeast. Prep. of phosphoglyceric acid. Synthesis of alanine. 1205, Effect of ultra-violet rays on alcoholic fermentation of molasses. 1206, Gluconic acid fermentation.

#### PATENT.

Manufacture of preparations of starch-degrading enzymes. KALLE & Co. A.-G. (B.P. 399,998, 14.11.32. Ger., 14.11.31 and 1.4.32).—A salt of an acid of P, except H<sub>3</sub>PO<sub>4</sub>, is added to solutions or dry preps. of enzymes, e.g., for desizing, with or without activators, stabilisers, or buffer substances. Examples are NaPO<sub>3</sub>, Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub>, Na<sub>3</sub>HP<sub>2</sub>O<sub>7</sub>, "Candioline," Na glycerophosphate. C. H.

#### XIX.—FOODS.

Effect of winter exposure in the stook on quality of wheat. R. K. LARMOUR, J. G. MALLOCH, and W. F. GEDDES (Canad. J. Res., 1933, 9, 252—260).—Samples of wheat which had been exposed in the stook during winter and threshed in the spring were compared with corresponding samples threshed in the autumn. In about 50% of the cases the grade of wheat was lowered by winter exposure, and the wt. per bushel decreased in nearly all cases. Flour yield was generally increased. Baking quality showed evidence of damage in 40% of cases, and improvement in 22%. O. J. W.

Bean meal and malt flour in milling and baking. P. BRUÈRE and J. CHEVALIER (Ann. Falsif., 1933, 26, 464—469).—The action of bean meal in wheaten flour is due to amyolytic and proteolytic enzymes. An equal diastatic effect can be obtained by the addition of 0.5—1.0% of malt flour. E. C. S.

Determination of the degree of milling of flour in bread. K. EBLE and R. BRETSCHNEIDER (Z. Unters. Lebensm., 1933, 66, 314—317; cf. B., 1932, 655; 1933, 329).—The P<sub>2</sub>O<sub>5</sub> contents (I) of 34 samples of rye flour are given. The mean of these vals. enables the ash content of rye flour to be calc. from (I). The determination of P<sub>2</sub>O<sub>5</sub> in bread and the calculation of the degree of milling from (I) are described. E. C. S.

Report of Sub-Committee on the viscosity test for soft winter wheat flours. E. G. BAYFIELD (Cereal Chem., 1933, 10, 494—501).—Viscosimetric determinations were carried out on unbleached soft winter wheat

flours by different investigators, using (a) their own procedure, and (b) a standard procedure involving several increments of lactic acid. Using (b), duplicate determinations may be obtained in a single laboratory within 2° [MacMichael (M)] and in different laboratories within 5°M. Results indicate that small differences due to variety of wheat and variations in ash and protein content may be measured viscosimetrically. E. A. F.

Report of Sub-Committee on selenium as a Kjeldahl catalyst in the cereal laboratory. C. F. DAVIS and M. WISE (Cereal Chem., 1933, 10, 488—493; cf. A., 1931, 1384; B., 1932, 621).—Using Hg-Se or Hg-Cu-Se catalysts, the concn. of sulphate must be kept low to avoid low results, and a shorter digestion period is required than with other catalysts. Se, either alone or with other common catalysts, is not recommended for use in Kjeldahl determinations. E. A. F.

Evaluation of systematic and random errors in protein, moisture, and ash determinations. A. E. TRELOAR (Cereal Chem., 1933, 10, 477—487; cf. 1928, 909; 1930, 32; 1932, 1051).—The procedure used in evaluating the systematic (I) and random (II) errors of chemists who have been co-operating in an endeavour to attain concordance in the above determinations in wheat and its products is described. Consistency between collaborators for (II) is very much higher for protein than for ash and moisture. (I) are somewhat lower in the analyses of flour than in wheat and wheat-feed. E. A. F.

Measurement of colour in flour and bread by means of Maxwell discs. J. C. BAKER, H. K. PARKER, and F. B. FREESE (Cereal Chem., 1933, 10, 437—446).—The N.-A. disc colorimeter (C), which is described and figured, measures the 4 factors making up the colour of a flour, viz., the colours red, yellow, black, and white, each expressed as a % of the total colour of the flour, by means of Maxwell discs. Measurements were carried out on the dry, wet, and dried-out (using both oven- and air-drying) slicks (Pekar tests) and on circular discs of bread. An attempt was made to correlate each of the component colours with flour grade, bleaching, and with degree of extraction, using light petroleum, for hard and soft wheats, respectively, and to correlate bread colour with the colour of the wet and dried-out slicks, respectively. The C cannot demonstrate such fine differences as can the Pekar test, but it is able to define coarse differences numerically. E. A. F.

Biochemical characteristics of dough and bread [made] from sprouted grain. N. KOZMIN (Cereal Chem., 1933, 10, 420—436).—Samples of flour milled from wheat which had germinated for varying periods up to 102 hr. were fermented, the gas production being determined every 15 min. and the sugar content at the end of the fermentation period (4 or 5 hr.), and then baked into round loaves. The diastatic activity (D) and H<sub>2</sub>O-sol. extract (including the protein, reducing sugar, and other non-nitrogenous content) of flour and bread from normal and sprouted grain were determined. The stickiness and apparent dampness of the crumb of bread from sprouted wheat flour (I) is due, not to an excessive moisture content, but to a sharp increase in



H<sub>2</sub>O-sol. substances (reducing sugars and, particularly, dextrans); this reduces the H<sub>2</sub>O-retaining capacity of the starch gel and the elasticity of the crumb. The starch hydrolysis in the dough from (I) is stimulated to a greater degree in the baking process than in the dough from normal flour. Baking decreases the sol. protein content to a smaller extent than Neumann's data indicate. The use of 0.1N-HCl as doughing liquid for (I) or of a long sponge fermentation (18 hr.) gave loaves having a normal, dry, and elastic crumb and a H<sub>2</sub>O-sol. content approaching that of bread from normal flour and only very slightly above that of the unbaked dough. The inhibition of *D* increased with the concn. of acid. Flours which were strongly buffered required a higher HCl concn. (e.g., 0.2N) to produce a normal loaf, and in this case there was a sharp decrease in H<sub>2</sub>O-sol. substances in the bread, indicating total inhibition of *D* during baking. The decrease in H<sub>2</sub>O-sol. constituents on baking was due to the coagulation of some of the sol. protein and, probably, also to exhaustion of the sugars by fermentation. If the saccharogenic activity of a flour exceeds a certain min., then the abs. val. will have no direct influence on the rate of fermentation; in this case *D* cannot be identified with the gas-producing capacity.

E. A. F.

**Influence of humidity and carbon dioxide on development of moulds on bread.** O. SKOVHOLT and C. H. BAILEY (Cereal Chem., 1933, 10, 446—451).—Samples of bread inoculated with pure cultures of *Aspergillus niger*, *Rhizopus nigricans*, and *Penicillium expansum* were kept, respectively, in atm. of varying humidities for 7 days and of varying concn. of CO<sub>2</sub> for 4 days. No mould was discernible at humidities < 84%; appreciable amounts of mould developed only at 90% R.H. Bread containing 6% of skim-milk solids has, on an average, 1.3% more moisture in the crumb than bread containing no such addition. A 17% CO<sub>2</sub> atm. at high R.H. exerts a measurable retarding effect on mould growth in bread; 50% of CO<sub>2</sub> completely inhibited mould. Temporary exposures to 50—88% CO<sub>2</sub> atm. for periods varying from 20 min. to 18 hr. exerted no permanent effect on mould spores.

E. A. F.

**Non-survival of red mould (*Monilia sitophila* group) at baking temperatures.** C. B. MORISON (Cereal Chem., 1933, 10, 462).—Conidia and ascospores of the mould were killed by exposure to 70° for 10 min., and to 60° for 20 min., but not to 50° for 5 min. No evidence of the mould was found in baked loaves kept in an incubator after 5 days.

E. A. F.

**Macaroni products.** J. A. LECLERC (Cereal Chem., 1933, 10, 383—419).—Data are given as to the United States production, consumption, imports, and exports of macaroni products (I). The types of (I) preferred, the cost of manufacture, and the types of wheat suitable for (I) are described. A brief account is given of semolina milling, with particular reference to the grades and composition of semolina or farina used. A detailed account is given of the manufacture of macaroni and of the forms in which the (I) are made. The characteristics of a good macaroni, the composition and food val. of macaroni, and the effect of storage on these (I) are

indicated. An account is given of the manufacture and composition of noodles, of (I) made from materials other than wheat, and of gluten macaroni.

E. A. F.

**Influence of agitation of milk before separation on fat loss in skim milk.** W. FINLAY and J. LYONS (Econ. Proc. Roy. Dublin Soc., 1933, 2, 501—513; cf. B., 1932, 784).—The loss (*L*) of fat in skim milk varies with the degree of agitation of the milk prior to separation. Heating in the plate pasteuriser (*P*) under gravity gives the best results. When the milk is pumped through *P*, *L* varies with the pump stroke. The vertical-type flash heater gives worst results; *L* varies with the speed of the beaters. *L* also depends on the no. of fat globules > 2 μ in diam. Agitation causes loss by increasing the no. of small globules. *L* caused by agitation may be > the saving due to preheating, particularly during summer.

E. C. S.

**Detection of pasteurisation [of milk] by the holding method.** M. F. BENGEL (Z. Unters. Lebensm., 1933, 66, 126—136).—Certain fractions (I) of milk-albumin coagulate when heated for several min. at 60—63°. If 20 g. of (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> are added to fresh milk a clear serum is obtained which contains (I) and becomes cloudy when heated to 65°, but the serum from pasteurised milk does not contain (I) and remains clear up to 70°.

E. C. S.

**Amyl alcohol for milk testing.** J. GOLDING (Analyst, 1933, 58, 531).—*n*-Amyl alcohol (I) gives Gerber tests 0.49% too high on milk containing 3% of fat. The b.-p. data for (I) given previously (cf. B., 1933, 569) are incorrect. 86.5% of the sample now in use distils between 130° and 131.2°.

E. C. S.

**Effects of freezing on physical and nutritional properties of milk.** R. C. MONKWITZ, M. H. BERRY, and W. C. BOYER (Maryland Agric. Exp. Sta. Bull., 1933, No. 344, 435—446).—Growth data for rats revealed no loss of nutritive val. in milk which had been frozen. Freezing causes a partial pptn. of milk solids, the amounts of constituents affected being in the descending order: albumin, lactose, total protein, ash, casein, total solids, fat. Except in the case of fat the amounts pptd. increase with the period of freezing. Fat globules, after freezing, tend to clump together and become irregular in size and shape.

A. G. P.

**Methods of investigation of preserved milk preparations.** F. E. NOTTBOHM (Z. Unters. Lebensm., 1933, 66, 254).—For sweetened preps. Schmid-Bondzynski's method is recommended; for unsweetened, Weibull's method (Staatsbl. Koninkl. der Nederlanden, 1925, No. 56).

E. C. S.

**Determination of the degree of concentration of tinned milk.** A. BECKEL (Z. Unters. Lebensm., 1933, 66, 177—182).—The *d*, fat content (*F*), Cl content (*C*), and *n* of the Cu serum of the milk are determined after dilution with an equal vol. of H<sub>2</sub>O. The degree of concn. is then given by either of the formulæ: 0.0444(*d* + *F* + 0.1*n*) or 0.067[*n* + 0.06(*C* — 100)].

E. C. S.

**Bacteriological examination of milk and its use in practical milk control.** T. and S. O. KOCH (Milch. Zentr., 1933, 62, 233—238, 245—248, 261—264, 273—275,



289—293).—The advisability of bacteriological standards for milk in Denmark similar to those in force in England and America, particularly for milk sold as “for children” or as “tuberculin-tested,” is discussed, and an account of a series of bacteriological investigations carried out by the authors on such milks, and extending over the years 1931 and 1932, is given, the milk being examined at various farms and then at the dairies supplied from the farms. The methods of bacteriological examination employed are described. Determinations of total plate count (I) and *B. coli* content (II) were made concurrently with reductase, sedimentation, and incubation tests, and acidity determinations. On the basis of the results obtained the authors recommend the adoption of bacteriological standards for milk in Denmark, based on determinations of (I) and (II). E. B. H.

**Fluorescence of milk and dairy products.** J. A. RADLEY (Analyst, 1933, 58, 527—529).—The fluorescence (*F*) in ultra-violet light mainly of wave-length 3650—3660 Å. of fresh cows' milk varies from white to yellow, becoming grey to blue on storing. Pasteurisation for 30 min. at 62.5° has no effect, but a reduction in intensity and a change in colour towards white take place in milk and cream heated for 30 min. at 100°. Reconstituted creams give colours varying with the type of dried milk used. The changes taking place during the process of cheese-making are described. The *F* of lactochrome (I) isolated from milk is an intense greenish-yellow, which changed to blue when reducing agents were used. *F* in milk is therefore probably due to the presence of (I). E. C. S.

**Technique of butter manufacture.** W. MOHR (Z. Unters. Lebensm., 1933, 66, 37—48).—The H<sub>2</sub>O content (I) and consistency of butter varies with the temp. of churning (II), *p<sub>H</sub>*, degree and temp. of washing and salting. Lecithin, which accumulates in the cream, passes entirely into the butter-milk. The loss of fat into the last-named is at a min. which depends on the feeding-stuffs employed, *p<sub>H</sub>*, and (II). Washing at 14—20° in winter gives brittle, and at 2—8° good, butter. Washing at 8—14° in summer gives good, and at 2° poor, soft butter. A nomogram is given for calculating (I) from evaporation data. E. C. S.

**Factors affecting the body or viscosity of cream and related matters.** J. LYONS and G. T. PYNE (Econ. Proc. Roy. Dubl. Soc., 1933, 2, 461—500).—The viscosity ( $\eta$ ) of rich, raw cream (I) varies with the separation temp. (*T*) and previous heat-treatment of the milk.  $\eta$  is at a max. when *T* is approx. 33°, but this max. is higher when the milk is chilled prior to separation.  $\eta$  is lower when the milk is stored at a high temp. before separation, the max. being ill-defined. Pasteurisation lowers  $\eta$  to an extent depending on the method of prep. of (I).  $\eta$  is lowered more by the flash method than by the holding method.  $\eta$  varies inversely as the fat-globule size, which is increased by pasteurisation. The agglutinin of milk does not influence  $\eta$ . By the reparation at 30° of chilled pasteurised cream (II) of low fat content, richer cream of normal  $\eta$  could be produced, except at certain times of the year, when the cream tended to churn. Passing (II) through a clarifier gave worse results. The  $\eta$  of (I) subjected to low-pressure

homogenisation was increased without marked injury to whipping and churning quality. The  $\eta$  of whipped (I) varies directly, and whipping time (III) inversely, as the homogenisation pressure and temp. (III) varies inversely as the fat content, increases on pasteurisation, but diminishes with reparation. The temp. coeff. of the  $\eta$  of (I) is —3% per 1° F. E. C. S.

**Formaldehyde titration of milk proteins and its use in the detection of reconstituted creams etc.** J. C. HARRAL (Analyst, 1933, 58, 605—606).—The ratio of the c.c. of 0.1N-NaOH required for determinations of the total N and for formol titration (by the method of Pyne, A., 1932, 1154) varied from 17.8 to 19.4 for 10 milks, and from 25.0 to 29.1 for 8 milk powders, and should therefore prove useful for the detection of reconstituted creams etc. J. G.

**Suggested regulations for control of melted cheese.** F. E. NOTTBOHM (Z. Unters. Lebensm., 1933, 66, 83—101).—A legal definition is suggested for “melted cheese,” particularly in respect of salt, butter, and H<sub>2</sub>O contents. E. C. S.

**Use of preparations containing phosphatides in the manufacture of foods.** F. E. NOTTBOHM and F. MAYER (Z. Unters. Lebensm., 1933, 66, 21—36).—The compositions of commercial preps. of animal and vegetable lecithins are given. Their use in the manufacture of cocoa, chocolate, margarine, macaroni etc., and dried milk is described. E. C. S.

**Methods of investigation of eggs.** K. BRAUNSDORF and H. BRINCKMEIER (Z. Unters. Lebensm., 1933, 66, 301—314).—Candling is the most informative test with respect to the age and condition of eggs. The proportions of white and yolk and their consistency in the egg when broken must also be taken into consideration. E. C. S.

**F.-p. determination of hens' and ducks' eggs.** P. WEINSTEIN (Z. Unters. Lebensm., 1933, 66, 48—59).—The difference (I) in f.p. between white and yolk of the fresh hen's and duck's egg is > 0.14°. The yolk is more nearly isotonic with the blood than is the white. When mixed with a solution of NaCl of f.p. —0.55° the observed rise in f.p. of the yolk is > the calc., so that (I) of equally diluted white and yolk is approx. 0.11°. When, as a result of storage, the f.p. of egg-yolk rises to —0.55°, the egg is dead. The *d* of eggs preserved in CaO or water-glass remains unchanged for long periods after removal from storage, but (I) falls below 0.10° in < 4 weeks. In ordinary cold-store eggs, however, a low *d* is associated with a small (I). E. C. S.

**Iodine content of hens' eggs as affected by the ration.** O. H. M. WILDER, R. M. BETHKE, and P. R. RECORD (J. Nutrition, 1933, 6, 407—412).—The I content of eggs increased with that of the ration and declined immediately the I feeding was discontinued. The I content was independent of the form in which it was supplied (dried kelp, iodised linseed meal, KI). A. G. P.

**Some factors affecting egg weight in the domestic fowl.** R. T. PARKHURST (Poultry Sci., 1933, 12, 97—111).—Inadequate supplies of vitamin-D gave lower



average egg wt. than where adequate amounts of ergosterol and cod-liver oil were supplied. An all-in-one ration gave larger egg size (*E*) than the same ration in mash form. Addition of green food and oyster shell increased *E* when supplementing all-in-one mash rations. Fish meal was superior to peanut meal. Minerals added to a meat meal ration with oyster shell *ad lib.* did not increase *E*, nor did extra amounts of dried skim milk added to a standard ration. Mixed proteins as compared with an individual protein, or variation in % protein, did not affect *E*. The substitution of fish meal for meat and bone meal, of dried butter-milk for dried skim milk, and of peanut meal for extracted soya-bean meal was without effect. It appears that protein and minerals are of no special val. in regard to egg wt.

E. M. C.

**Estimation of the extract of coffee and chicory.**

E. J. BUNCE (Analyst, 1933, 58, 532; cf. B., 1933, 570).—Determination of the H<sub>2</sub>O-sol. extract of 2 samples of Indian coffee gave by Jones' method 27.8 and 28.3%, and by Smetham's method 30.1 and 30.4%. E. C. S.

**Determination of caffeine and extract in coffee.**

E. HELBERG (Mitt. Lebensm. Hyg., 1933, 24, 54—79; Chem. Zentr., 1933, i, 3813).—Results obtained by Juckenack, Hilger, and Wimmer's (I) method are too high, and a determination of N is necessary. Lendrich and Nottbohm's (II) and Fendler and Stüber's methods give results which are in accord (gravimetric and N determination) with these N vals. In (II) long drying of the residue followed by extraction with CHCl<sub>3</sub> gives low vals. for caffeine; extraction of the solution after saturation with NaCl gives quant. results. Preferably the extract is triturated with CaO and quartz sand and then extracted with CHCl<sub>3</sub>. CCl<sub>4</sub> needs 10 hr. extraction in a Soxhlet apparatus. The results then accord with those of (I) in which extraction is continued for 10 hr.

A. A. E.

**Chlorogenic acid content of raw and roasted coffee.** W. HOEFFNER (Z. Unters. Lebensm., 1933, 66, 238—251; cf. B., 1933, 650).—The chlorogenic acid (I) content of unroasted coffee was between 6.3 and 7.7%, and of roasted coffee between 3.2 and 4.5%, varying with the type of coffee and the degree of roasting. (I) is present in raw beans as the K and K caffeine salts. The former is destroyed first during roasting. E. C. S.

**Determination of chlorogenic and caffeic acids.**

W. PLÜCKER and W. KEILHOLZ (Z. Unters. Lebensm., 1933, 66, 200—238).—Chlorogenic (I) and caffeic (II) acids are determined by electrometric titration, using the quinhydrone electrode. (I) is decomposed by prolonged boiling in H<sub>2</sub>O solution. It occurs in two forms, the ordinary form having  $[\alpha]_D -35.45^\circ$  and the second having  $[\alpha]_D -47.90^\circ$ . Lendrich's process causes a loss of (I), which varies with the type of coffee. (II) occurs in traces only. Dihydroxystyrene could not be detected. The breakdown processes which occur during the treatment of coffee by Lendrich's process are probably less drastic than those suggested by him.

E. C. S.

**Caffeine content of maté.** F. KRAUSS, E. KLEUCKER, and A. KOLLATH (Z. Unters. Lebensm., 1933, 66, 348—354).—Maté contains 0.3—1.5% of caffeine (I). (I) was

identified by micro-analysis and a mixed m.p. with pure (I). It is incorrect to assume the presence in maté of a sp. alkaloid "matein," or to refer to "caffeine-poor" or "caffeine-free" maté. The Lendrich-Grossfeld method of determination of (I) is recommended, but treatment with KMnO<sub>4</sub> and with petroleum cannot be omitted, though such omission is possible with tea. E. C. S.

**Unification of methods of investigation of cacao products.** F. HÄRTEL (Z. Unters. Lebensm., 1933, 66, 251). E. C. S.

**Adulteration of cacao butter. Determination of the index of azelaic acidity.** G. SCHUSTER (Compt. rend., 1933, 197, 760—762).—The index of azelaic acidity (*A* = mg. of KOH to neutralise 1 g. of insol. acid glycerides obtained by KMnO<sub>4</sub> oxidation of the fat) is determined by conversion of the oxidation products into Mg salts, Mg pelargonate being separated by its solubility (30—40% at 15°) in 80% EtOH, whereas the Mg salts of the azelaic glycerides are insol. in this medium. Determination of the Mg in 1 g. of the latter permits the calculation of *A*. For cacao butter the mean val. of *A* is 98.7, but for karité butter it is 131.1, hence evaluation of *A* permits the detection and determination of the latter as an adulterant in the former. J. W. B.

**Definitions of meat salad [and herring salad] and mayonnaise.** A. BEHRE (Z. Unters. Lebensm., 1933, 66, 118—126). E. C. S.

**Effect of hydrogen cyanide on fruit.** F. BERAN (Z. Unters. Lebensm., 1933, 66, 317—321).—1 hr. after exposure to 6 mg. of HCN per litre at 17° for ½—3 hr., apples and pears contained (mg. HCN per kg. of fruit): 7; after 2 hr., < 1 mg.; after 6 hr., nil. The fruit was not damaged by exposure for 1—2 hr. to HCN at this concn., but 1.2 mg. per litre caused considerable spoilage. E. C. S.

**Effect of metals on colour of raspberries and strawberries.** J. M. BRYAN and T. N. MORRIS (Canning Trade J., 1933, 3, 252).—The quantity of metals causing discoloration in fruits and jams was investigated. 40 p.p.m. of Al, Cu, Ni, Cr, Zn, Pb, and Ag had no immediate effect on fruits at room temp. or above; 2 p.p.m. of Fe was sufficient to discolour strawberries, whilst blackcurrants are more sensitive to Sn. Figures are given for the corrosion of various metals by a 1% citric acid solution in presence and in absence of air. The results indicate that Al is suitable for fruit and fruit juices in the cold, whether air be present or not, but stainless steels should be carefully tested for use in air-free conditions, though most are suitable. E. B. H.

**Determination of formic acid in fruit juices.** C. ZÄCH (Mitt. Lebensm. Hyg., 1933, 24, 35—42; Chem. Zentr., 1933, i, 3813).—Fruit juice (10 c.c.) and tartaric acid (0.2 g.) are distilled with steam in a manner described; the distillate (300 c.c.) is rendered slightly alkaline with NaOH, evaporated, and acidified with HCl (0.2 c.c. of *N*-acid in excess). The solution is heated for 1 hr. at 100° with 10 c.c. of a reagent prepared from HgCl<sub>2</sub> 10 g., NaCl 4 g., NaOAc crystals 10 g. in 100 c.c., this being heated at 100° for 1 hr. and filtered. The pptd. HgCl (≧ 0.5 g.) is weighed. Genuine fruit juices afforded 4—9 mg. HCO<sub>2</sub>H in 100 c.c. A. A. E.



**Effect of carbon dioxide and sodium benzoate on vitamin-C content of orange juice.** A. F. MORGAN, C. I. LANGSTON, and A. FIELD (Ind. Eng. Chem., 1933, 25, 1174—1176).—The vitamin-C content of orange juice (I) was unchanged after storing for 18 months in the frozen state (cf. B., 1932, 815; 1933, 282). A commercial sweetened (I) was found to have 67% of the potency of fresh juice, the addition of NaOBz having no effect. (I) diluted with H<sub>2</sub>O containing CO<sub>2</sub> at a pressure of 2.5 kg. per sq. cm. and then pasteurised retained its potency; (I) diluted with ordinary H<sub>2</sub>O and pasteurised lost > 50% of its potency. E. C. S.

**Effect of storage on the palatability and vitamin content of rutabagas.** J. E. RICHARDSON and H. L. MAYFIELD (Montana Agric. Exp. Sta. Bull., 1933, No. 277, 12 pp.).—Rutabagas form an excellent source of vitamins-B<sub>1</sub> and -C and may be stored in cellars without loss of vitamin activity. Cooking causes greater loss of vitamin in the autumn than after the roots have been stored through the winter. A. G. P.

**Effect of storage and canning on the vitamin content of carrots.** D. D. LANGLEY, J. E. RICHARDSON, and E. J. ANDES (Montana Agric. Exp. Sta. Bull., 1933, No. 276, 32 pp.).—Storage in cellars for 4 months does not reduce the vitamin-A, -B<sub>1</sub>, or -C content of carrots. Some increase in -C potency is recorded. The cooking of carrots in autumn causes some loss of -A and -B<sub>1</sub>, but not of -C. Cooking after storage produces the same loss of -B<sub>1</sub> as autumn cooking, and some loss of -C, whilst the -A potency shows a small increase over that of the raw carrot. Canning by various processes involves little or no loss of -A or -B<sub>1</sub>, but a definite loss of -C. Subsequent storage of canned carrots causes marked losses of all three vitamins. A. G. P.

**Composition of canning tomatoes.** L. G. SAYWELL and W. V. CRUESS (Calif. Agric. Exp. Sta. Bull., 1932, No. 545, 32 pp.; cf. B., 1933, 409).—In general, the total solid content of tomatoes increased from September to October and declined markedly in November. Locular material had higher total acid and lower total solid, reducing sugar, acid-hydrolysable matter, and protein contents than the cores and walls. Nearly the whole of the sugar content consists of reducing sugars. Both sucrose and starch contents were < 0.05%. Detailed analyses of a no. of varieties are recorded. A. G. P.

**Changes occurring during freezing, storage, and thawing of fruits and vegetables.** M. A. JOSLYN and G. L. MARSH (Calif. Agric. Exp. Sta. Bull., 1933, No. 551, 40 pp.; cf. B., 1933, 443, 603).—Physical changes in fruit are largely conditioned by ice formation and osmotic action. During thawing enzymic hydrolysis of pectin was small but there was appreciable inversion of sucrose. Exposure of fruit to air during freezing or thawing or after thawing results in deterioration of colour and flavour if active oxidases are present. The latter cannot be inactivated by heat or treatment with acid or reducing agents without adverse effects on flavour. A. G. P.

**Effect of nature of container on quality of preserves.** A. MACHEBEUF, H. CHEFTEL, and (MME.) J. BLASS (Ann. Falsif., 1933, 26, 470—474).—Overcooking is more easily avoided in tins (I) than in glass containers

(II) since (I) may be rapidly cooled by immersion in cold H<sub>2</sub>O. Deterioration due to the chemical action of the container is most marked in ordinary (II), and may, in certain cases, mask the effects of overcooking.

E. C. S.

**Canning fruits and vegetables.** G. CADBURY (Chem. & Ind., 1933, 885—891).

**Tunny fish canned in Great Britain.** C. W. BANKS (Canning Trade J., 1933, 3, 315).—Tunny caught off the Yorkshire coast have been successfully canned. The general method of processing is given, together with the estimated yield per fish. E. B. H.

**Canning of cockles.** ANON. (Canning Trade J., 1933, 3, 314—315).—According to the Campden Research Station's report, the canning of cockles (method given) seems a commercial proposition on a small scale; figures are given for the probable yield and cost. E. B. H.

**Production of a palatable artichoke syrup. II. Hydrolysis of the polysaccharide material.** F. A. DYKINS and D. T. ENGLIS (Ind. Eng. Chem., 1933, 25, 1165—1168; cf. B., 1933, 1032).—Hydrolysis with HCl is optimal at 130.5° and *p*<sub>H</sub> 4.2, and requires 20 min. The salt formed by neutralisation amounts to < 1% of the syrup. The rate of hydrolysis does not vary with the salt content of the extract. The buffering capacity of the extract varies with the total solids. Other acids, the anions of which can be removed by pptn. or electro-dialysis, can be used, at a concn. determined by the optimum *p*<sub>H</sub> for hydrolysis. E. C. S.

**Determination of dry matter in pulpy materials rich in carbohydrates, e.g., beet, potatoes, chicory.** G. SESSOUS and M. ROHWEDER (Arch. Pflanzenbau, 1933, 10, 191—202).—A no. of methods are compared. The use of vac. desiccators during the cooling of dried samples is recommended. At 105° oxidation of the materials is considerable and volatile products may be lost. Interrupted periods of drying proved suitable. A. G. P.

**Dried potato preparations.** G. KAPPELLER (Z. Unters. Lebensm., 1933, 66, 136—142).—Methods of analysis of potato flour (I), flakes, and roller meal are described. Physical and chemical characteristics of 4 grades, and of 4 commercial preps. of (I) are given. E. C. S.

**Estimation of total and bound (D) gossypol in cottonseed meal.** F. H. SMITH and J. O. HALVERSON (Ind. Eng. Chem. [Anal.], 1933, 5, 319—320).—The method, a modification of Carruth's (A., 1918, i, 266; B., 1918, 319), gives results with an accuracy of 95.9%. E. C. S.

**Relation of moisture to extraction of gossypol from cottonseed meal with ether.** J. O. HALVERSON and F. H. SMITH (Ind. Eng. Chem. [Anal.], 1933, 5, 320—322).—The presence of H<sub>2</sub>O increases the amount of gossypol (I) extractable from cottonseed meal with Et<sub>2</sub>O, suggesting that there is no definite limit between bound and Et<sub>2</sub>O-extractable (I). E. C. S.

**Vitamin-B (B<sub>1</sub>) and -G (B<sub>2</sub>) content of cottonseed products.** M. L. WHITSITT (Ind. Eng. Chem., 1933, 25, 1169—1171).—Cottonseed meal (I) is rich in vitamin-B<sub>1</sub>, and both (I) and cottonseed hulls (II) are good sources



of  $B_2$ . Cottonseed oil contains neither vitamin. Feeding with extract of (II) and insufficient (I) for complete protection against polyneuritis (A) results in a much less rapid decline in wt. and a much longer survival period than feeding with (I) alone, but in each case A develops. Since (II) contains no appreciable amount of  $-B_1$ , a third factor of the B complex must therefore be present.

E. C. S.

**Grass silage.** C. BOYLE and J. J. RYAN (Econ. Proc. Roy. Dublin Soc., 1933, 2, 515—528).—Grass approaching maturity, and aftermath (I), can be converted into good silage by the pit method. Silage made from (I) can replace part or all of the hay (II) or roots (III). One stone of (II) may be replaced by 45 lb. of (I), 4 stones of (III) by 24 lb. of (I),  $\frac{1}{2}$  stone of (II) and 2 stones of (III) by 35 lb. of (I). Losses of dry matter amount to 18.5% or, calc. on the starch equiv., to approx. 25%.

E. C. S.

**Loss of food value during storage of fresh beet slices in silos.** J. VONDRÁK (Z. Zuckerind. Czechoslov., 1933, 58, 25—27).—The fresh slices at the end of 4½ months' storage had lost 57% of the ash-free dry substance. Only 10% of the original amount of sugars remained at the end of this time, so that this method of preserving a surplus of beets cannot be economically recommended.

J. P. O.

**Vitamin content of Eviunis (Vitophos).** A SCHEUNERT, M. SCHIEBLICH, and J. RESCHKE (Z. Unters. Lebensm., 1933, 66, 271—278).—Vitamin-A and -D could not be detected, and vitamin-B complex was present in very small amount, thus confirming van Esveld's results (Verslagen en meded. betr. de Volksgezondheid, 1932.)

E. C. S.

**Detection of "sionon" in diabetic foods.** R. VIOLLIER and E. ISELIN (Mitt. Lebensm. Hyg., 1933, 24, 104; Chem. Zentr., 1933, i, 3813).—Werder's procedure is used for the detection of synthetic sorbitol.

A. A. E.

**Application of statistical methods to food chemistry.** A. BECKEL (Z. Unters. Lebensm., 1933, 66, 158—177).

E. C. S.

**Determining moisture.**—See I. **Shortening by plastic fats.** **Test for butter fats.**—See XII. **Casein paints.**—See XIII. **Hide etc. fleshings [as feed].**—See XV. **Fodder beets.**—See XVI. **Sucrose in potato slices.** **Sugar determinations.**—See XVII. **Vitamins in vinegar.** **Fermented milk.** **EtOH yield of cereals.**—See XVIII.

See also A., Nov., 1117, **Starch and bread-making.** 1143, **Synthesis of vitamin-C.** 1178, [Fractionation of] casein. 1187, **Micro-determination of Ca in milk.** 1200, **Metal containers and foodstuffs.** 1211—3, **Vitamins (various).**

## PATENTS.

**Manufacture of a food [dried-egg] product.** S. TRANIN (B.P. 399,492, 4.4.32).—An easily sol. product with good beating properties and baking qualities is obtained by dissolving in liquid egg a protective substance (e.g., sugar or NaCl) in absence of air and spray-drying in a current of  $\text{CO}_2$  at  $> 48.9^\circ$ . Suitable apparatus is claimed.

E. B. H.

**Preservation of (A) fruits, vegetables, and other organic, especially medicinal, substances, (B) preliminarily dried fruits.** K. HESSEL (B.P. [A] 399,823 and [B] 399,831, 4.4.32).—(A) Atomised raw fruits, vegetables, or herbs are mixed with carrier substances such as flour and sugar, both substances being sprayed through separate nozzles, with compressed air, into an enclosed shaft. The raw substance becomes saturated and coated with the carrier substance and the fresh fruity flavour and vitamins are retained.  $\text{O}_2$ ,  $\text{O}_3$ , or  $\text{CO}_2$  may be used with the air for sp. purposes. (B) Cut-up, dried fruit is coated with a dry mixture of fruit acid and sugar. The products of (A) or (B) may be later compressed into shapes and mixed with fats, chocolate, sugar, etc. [Stat. ref. to (B)].

E. B. H.

**Protecting meat and other perishable commodities from deterioration during storage.** J. BREWIS (B.P. 399,648, 26.9.32).—Meat in cold storage is covered with a film of hardened edible fat applied directly or as an impregnated closely adhering paper or fabric wrapping.

E. B. H.

**Antioxidant for fats etc.** **Oil from fish press-cake.**—See XII.

## XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Preservatives in cosmetics.** E. BÖHM (Riechstoffind., 1933, 8, 7—9, 22—25; Chem. Zentr., 1933, i, 3805).—The use of preps. containing *p*-hydroxybenzoic esters, especially the benzyl ester, is discussed.

A. A. E.

**Effect of sodium perborate on glycerol.** L. RYBÁČEK (Časopis českoslov. Lék., 1933, 13, 41—46; Chem. Zentr., 1933, i, 3261).—The detection of  $\text{CH}_2\text{O}$  in presence of  $\text{H}_2\text{O}_2$  and perborate with guaiacolsulphonic acid (I) and other reagents has been studied in relation to the detection of glycerol (II) in preps. containing (II) and perborates. After removal of  $\text{H}_2\text{O}_2$  by filtration through  $\text{MnO}_2$ ,  $\text{CH}_2\text{O}$  can be detected with (I); a 4%, but not a 0.4% solution of  $\text{CH}_2\text{O}$  in presence of perborate gives a characteristic reaction. Hehner's reagent detects  $\text{CH}_2\text{O}$  in  $< 0.4\%$  concn. in presence of perborate. The reaction with morphine is similarly affected. Lebbin's reaction is normal at  $< 0.4\%$ . To avoid the formation of  $\text{CH}_2\text{O}$ , the mixture must be prepared in the cold; the stability can be increased by addition of phenacetin.

A. A. E.

**Properties of Psyllium seed.** R. HANSCH and E. U. STILL (Amer. J. Pharm., 1933, 105, 433—435).—Results of determinations of  $\text{H}_2\text{O}$ , ash, N, P, lipins, hexoses, and pentoses in the whole seeds, the hulls, and in a gum prepared from the hulls are given. (Cf. A., 1931, 131.)

W. S.

**Beam's colour test for hashish.** A. LUCAS (Analyst, 1933, 58, 602).—Beam's statement that the test is presumptive and not sp. is emphasised. J. G.

**Tobacco smoke.** III. E. WASSER and M. STÄHLI (Z. Unters. Lebensm., 1933, 66, 354—362; cf. B., 1933, 332).—None of the known basic constituents of tobacco is pptd. by silicotungstic or picric acid when present at the concn. usual in smoking tests, and these, therefore, do not interfere with the determination of nicotine (I)



by Pfl and Schmitt's method. When (I) in cigarette tobacco is  $< 1\%$ , (I) in the smoke varies with the (I) in the tobacco. Certain cotton-wool and cellulose plugs reputed to absorb (I) from the smoke were found to remove only 33% as a max., the effect being purely mechanical filtration. E. C. S.

**Determination of nicotine in tobacco and in parasiticides containing nicotine.** W. MOHR (Z. Unters. Lebensm., 1933, 66, 362—365).—The nicotine (I) is distilled in steam from alkaline solution, the distillate passing directly into saturated picric acid solution, by which (I) is pptd. E. C. S.

**Determination of arbutin in *Folia uvae ursi* and its preparations.** C. GRIMME (Pharm. Zentr., 1933, 74, 669—670).—The finely-ground drug (8 g.) is extracted with 50 c.c. of boiling  $H_2O$  (4 times) and the solution pptd. with  $Pb(OAc)_2$ . The filtrate is freed from Pb with  $NaHCO_3$ , extracted with  $Et_2O$  to remove  $p-C_6H_4(OH)_2$ , boiled with dil.  $H_2SO_4$  to hydrolyse the glucoside, and decolorised by Zn dust (1 g.). Arbutin is then determined in the final neutralised solution by titration with 0.1N-I. S. C.

**Determination of alkaloids in hydrastis  $\theta$ , hydrastis fluid extract, and other homœopathic tinctures.** H. NEUGEBAUER (Pharm. Ztg., 1933, 78, 1077).—The alkaline (aq.  $NH_3$ ), EtOH-free, aq. liquid is extracted with  $Et_2O$  and the  $Et_2O$ -free, aq. extract is acidified with 0.1N-HCl and titrated with 0.1N-NaOH. Adaptations for various tinctures are given. E. H. S.

**Microscopical identification of heroin.** G. D. WILLIAMS and C. C. FULTON (Amer. J. Pharm., 1933, 105, 436—440).—The sensitivity of a large no. of alkaloidal reagents in detecting heroin is indicated in a table, and those ppts. which are cryst. are described in detail.  $HgI_2 \cdot HCl$ ,  $HAuCl_4$ , and Na picrate are at once sensitive and give characteristic crystals. W. S.

**Collaborative comparison of five opium assay procedures.** A. R. BLISS, JUN., E. D. DAVY, J. ROSIN, W. H. BLOME, and R. W. MORRISON (Amer. J. Pharm., 1933, 105, 458—466).—The B.P. 1932 and U.S.P. X (modified) methods are, on the whole, accounted the best. W. S.

**Essential oil of maritime pine needles.** J. DE FAYARD (Bull. Inst. Pin, 1933, 215).—The oil distilled at various seasons was obtained in yields of 0.68—0.79% containing 3.68—3.76% of bornyl acetate. It had  $[\alpha]_D^{20} + 31.5^\circ$  to  $+34.2^\circ$ . A. A. L.

**Salvia oils.** B. N. RUTOVSKI and K. SABRODINA (Riechstoffind., 1932, 7, 174—175; Chem. Zentr., 1933, i, 3804—3805).—Essential oil of wild *Salvia sclarea*, L. (0.1—0.3%), had  $d_{20}^{20}$  0.9001—0.9142,  $\alpha_D$   $+9.25^\circ$  to  $-14.39^\circ$ ,  $n_D^{20}$  1.4593—1.4650, acid val. 0.1—0.89, ester val. 171.06—196 (after acetylation 202.8—215.0). Vals. for cultivated plants were:  $d_{20}^{20}$  0.8888,  $\alpha_D$   $-7.24^\circ$ ,  $n_D^{20}$  1.4579, acid val. 0.58, ester val. 163.2 (after acetylation 219.8). Oil of *S. nemorosa*, L. (0.01%), had  $d_{20}^{20}$  0.8996,  $n_D^{20}$  1.4890, acid val. 2.07, ester val. 45.05 (after acetylation 80.86). Oil of *S. spinosa*, L. (0.06%), had  $d_{20}^{20}$  0.8888,  $\alpha_D$   $+12.45^\circ$ ,  $n_D^{20}$  1.4548, acid val. 21.0, ester val. 57.75 (after acetylation 188.86). Oil of *S. Korolkovi*, Rgl. et Schmalh. (0.35%), had

$d_{20}^{20}$  0.9133,  $\alpha_D$   $-14.85^\circ$ ,  $n_D^{20}$  1.4760, acid val. 0.69, ester val. 2.73 (after acetylation 38.39) and contained *l*- $\beta$ -pinene (25), cineole (34), *l*-camphor (3), borneol (10), bornyl acetate (1), caryophyllene (20), and a sesquiterpene alcohol (0.6%). A. A. E.

**Colour reaction of geranium oil and commercial rhodinol.** S. SABETAY (Riechstoffind., 1933, 8, 26—27; Chem. Zentr., 1933, i, 3806).—On addition of 0.5—1 c.c. of 10% Br in  $CHCl_3$  to 5 drops of geranium oil in 2 c.c. of  $CHCl_3$ , without cooling, an intense bluish-green colour appears. It is due to an unrecognised constituent of the oil. A. A. E.

**Brazilian bois de rose oil.** W. H. SIMMONS (Perf. & Essent. Oil Rec., 1933, 24, 338).—The method of Glichitch (A., 1923, ii, 661) gives too high vals. for linalool in the above oil owing to the presence of other alcohols. The distillation method is to be preferred. A. A. L.

**Caffeine in coffee and maté.** Vitophos. Diabetic foods.—See XIX.

See also A., Nov., 1144, Synthesis of vitamin-C. 1159 and 1171, Local anæsthetics. 1168, Heterocyclic bases from coumarans and phenyldioxan. 1171, Nitration of phenobarbital. 1175—6, Derivatives and reactions of alkaloids. 1179, Colorimetric determination of acetylsalicylic acid. 1180, Micro-detection of coumarin. Identification of cocaine and novocaine. 1183, Prep. of intact total phosphatide-cerebroside. 1184, Choline in ophthalmic powders. 1197, Tinctures of *Digitalis* as medicaments. Influence of Fe salts on tobacco smoke. 1208, Purification of secretin. Prep. of blood-Ca-raising substance. 1209, Determining thyroxine in thyroid. 1211, Male hormone from pigs' testicles. 1211—3, Vitamins (various). 1216, Prep. of dihydrorotenone. Active constituents of *Urtica dioica*. Indian medical plants. 1217, Alkaloids of *Artabotrys*.

#### PATENTS.

**Manufacture of ethanolamine [ $\beta$ -hydroxyethylamine] salts of theophylline.** H. A. SHONLE, Assr. to E. LILLY & Co. (U.S.P. 1,867,332, 12.7.32. Appl., 8.11.30).—Theophylline is dissolved in  $H_2O$  with rather  $> 1$  mol. of the amine and the solution is conc. to crystallisation in vac. The mono- (m.p. 255—260°) and tri- (m.p. 235—245°) (hydroxyethyl)amine salts are described. C. H.

**$\beta$ -Dimethylaminoethyl ester of *p*-butylamino-benzoic acid [local anæsthetic].** O. EISLEB, Assr. to WINTHROP CHEM. Co., INC. (U.S.P. 1,889,645, 29.11.32. Appl., 22.6.32. Switz., 22.12.30).— $\beta$ -Dimethylaminoethyl *p*-butylaminobenzoate, m.p. 43°, b.p. 210°/4 mm. (hydrochloride, m.p. 147—148°; nitrate, m.p. 131—132°; picrate, m.p. 120°; oxalate; *H* oxalate), is prepared from *p*-butylaminobenzoic acid (I), m.p. 153—154°, and  $NMe_2 \cdot CH_2 \cdot CH_2 \cdot OH, HCl$  in boiling PhMe in a stream of dry HCl, from the *Et* ester, m.p. 69—70°, of (I) and  $NMe_2 \cdot CH_2 \cdot CH_2 \cdot OH$  in presence of a little NaOEt, from the  $\beta$ -chloroethyl ester, m.p. 64—65°, of (I) and  $NHMe_2$  in  $C_6H_6$  at 100°, or from *p*- $NMe_2 \cdot CH_2 \cdot CH_2 \cdot O \cdot CO \cdot C_6H_4 \cdot NH_2$ , Bu<sup>n</sup>Br, and BuOH at the b.p. H. A. P.



**Manufacture of quinoline-8-azo compounds of the quinine series.** I. G. FARBENIND. A.-G. (B.P. 399,818, 11.4.32. Ger., 8.8.31).—An 8-aminoquinoline is diazotised and coupled with hydrocupreine (I) or a substituted (I) to give compounds effective against malaria. Examples are: (I)  $\leftarrow$  8-amino- (m.p. 168°), 8-amino-6-methoxy- (m.p. 180°; B, 2HCl, m.p. 218°), 8-amino-6-ethoxy- (B, 2HCl, m.p. 208°), and 8-amino-6-amyloxy- (B, 2HCl, m.p. 218°)-quinolines; hydrocupreinesulphonic acid  $\leftarrow$  8-amino-6-methoxyquinoline, hydrolysed. C. H.

**Preparation of parathyrin.** L. BERMAN (U.S.P. 1,865,164, 28.6.32. Appl., 21.6.27).—The proteins in fat-free parathyroid gland are first coagulated, e.g., with powdered  $H_2Cl_2$ , and the mixture is extracted with 5%  $H_2SO_4$  in 85–90% EtOH. C. H.

**Manufacture of bismuth-dithioglycollic acid and its salts.** K. STREITWOLF, A. FEHRLE, W. HERRMANN, and P. FRITZSCHE, ASSRS. to WINTHROP CHEM. CO., INC. (U.S.P. 1,864,679, 28.6.32. Appl., 30.10.30. Ger., 5.6.30).—Bi salts are treated with  $SH \cdot CH_2 \cdot CO_2H$  in acid solution, or  $Bi(S \cdot CH_2 \cdot CO_2H)_3$  is treated with acid, to give  $CO_2H \cdot CH_2 \cdot S \cdot Bi \left\langle \begin{array}{l} S \cdot CH_2 \\ O \cdot CO \end{array} \right.$  or its complexes. Alkali salts are described. C. H.

**Metal complex compound[s]. [Medicinals.]** H. SCHMIDT, ASSR. to WINTHROP CHEM. CO., INC. (U.S.P. 1,889,383, 29.11.32. Appl., 4.6.31. Ger., 3.6.30. Cf. B.P. 351,971; B., 1931, 1041).—Neutral complex compounds of a metal of at. no. > 21, e.g., Sb, Bi, Cu, Pb, Sn, Fe, Zn, Co, and Th, and of an alkaline-earth metal (Ca, Sr, Mg) with sulphonic acids of *o*-dihydroxyaromatic compounds [1 : 2 : 3 : 5- $C_6H_2(OH)_2(SO_3H)_2$ ] which may contain further substituents are claimed. An alkali metal may also be present. They are prepared by interaction of the org. compounds with the hydroxides of the metals, or with their salts in presence of alkalis, a neutral end-reaction being essential. The simplest compounds are probably of the type  $C_6H_2[(SO_3)_2Ca] \left\langle \begin{array}{l} O \\ O \end{array} \right\rangle Sb \left\langle \begin{array}{l} O \\ OCa_4 \end{array} \right\rangle C_6H_2[(SO_3)_2Ca]$ , but compounds of high mol. wt. are also claimed. H. A. P.

**Cryst. Al salts.**  $(CH_2)_6N_4$  derivative.—See III.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Sensitivity of [photographic] emulsions; method of expression suitable for direct application to exposure tables.** G. CORDONNIER (Bull. Soc. Franç. Phot., 1933, 20, 201–209).—In conjunction with a figure for the light val., obtained from tables allowing for various types of subject, sky conditions, time, and season, it is proposed to use, not a single figure for the speed of a given emulsion, but a range from min. to max. exposure for the shadows, together with an indication of the range of contrast obtainable. This range is indicated by a scale with sections of increasing depth of blackening. J. L.

**Advances in the field of sensitisers for [the photography of] the far red and infra-red in Ger-**

many since the year 1918. W. DIETERLE, H. DÜRR, and W. ZEH (Z. wiss. Phot., 1933, 32, 145–156).—The compositions and properties of the di- and tri-carbo-cyanines (= penta- and hepta-carbo-cyanines) are critically reviewed. J. L.

**Measurements of the electrostatic properties of photographic films.** D. C. ROSE (Canad. J. Res., 1933, 9, 197–216).—Experiments in which camera films were rubbed with wool and brass under varying atm. conditions of pressure, temp., and humidity indicate that under practically any conditions the celluloid is liable to acquire a considerable electrostatic charge. The charging is particularly sensitive to changes in the physical conditions of the film, and is more pronounced in a moist than in a dry atm. Methods of eliminating the trouble caused by static electricity in aerial photography are discussed. O. J. W.

See also A., Nov., 1127,  $Ag_2CrO_4$  in emulsions. **Effect of X- and  $\alpha$ -rays on emulsions.** 1134, **Camera for Laue spectrograms.** 1135, **Spectrophotometry.**

## PATENTS.

**Anti-halation layers for photographic plates and films.** I. G. FARBENIND. A.-G. (B.P. 399,387 and 399,713, [A] 21.2.33, [B] 1.3.33. Ger., [A] 24.2.32, [B] 1.3.32. Addn. [A] to B.P. 369,721; B., 1932, 704).—(A) A heterocyclic  $NH_4$  compound with a reactive Me group is condensed with an aromatic aldehyde, the reactants being chosen so as to give a product having affinity for cotton. When used in anti-halation layers the products are completely decolorised by the alkaline developer. Examples are: quinaldine Me methosulphate (2 mols.) with diphenyl-4 : 4'-dialdehyde or azoxybenzylideneaniline (yellow); 2 : 2'-dimethyl-6 : 6'-bisbenzoxazole Me methosulphate (red) or 6-phenylquinaldine Me methosulphate (violet) with *p*-dimethylaminobenzaldehyde. (B) As binding-agent there is used an alkali-sol. artificial resin, e.g., the resin from salicylic acid and paraldehyde, from  $PhO \cdot CH_2 \cdot CO_2H$  and  $CH_2O$ , from *o*- $MeO \cdot C_6H_4 \cdot CO_2H$  and  $CH_2O$ , or from  $PhO \cdot CH_2 \cdot CO_2H$  and 3 : 5-di(hydroxymethyl)-*p*-cresol. C. H.

**Production of pictures in colours.** E. D. COOPER and J. DAVIES (B.P. 399,786, 10.2.32).—Pictures are taken on a film, alternately through red and green filters (or other combination of colours). The completed positive printed from it is treated with a solution of æsculin (I) or  $\beta$ - $C_{10}H_5(OH)(SO_3H)_2$ , rose bengal, patent blue, and a trace of  $NH_3$ . The "red" image only is projected through a red filter (or dyed red), and is preferably combined with the "green" image in projection. The (I) solution may be applied to the "green" image only. J. L.

## XXII.—EXPLOSIVES; MATCHES.

**Cause of progressive disappearance of diphenylamine in colloidal powders.** H. MURAOUR (Compt. rend., 1933, 197, 758–760).—The disappearance of the stabiliser  $NHPh_2$  (I) from colloidal nitrocellulose powders is due to the oxidation and nitration or nitrosation of (I) by the nitrocellulose. The catalytic action of Cu salts is increased by alkali or alkaline-earth chlorides (which



themselves have no influence), and diminished by the presence of oxalates, tartrates, or  $K_4Fe(CN)_6$ . No similar action occurs when (I) is replaced by  $CO(NPhEt)_2$ , the use of which is recommended. J. W. B.

**The Schulze-Tiemann and Lunge methods for determining nitrogen in nitrocellulose.** E. RUBENS (Z. ges. Schiess- u. Sprengstoffw., 1933, 28, 172—176).—On shaking NO with  $H_2O$  the loss was about 8.5%, with 26.8—29.4% KOH it was 0.8—0.9%, and with 32.8% NaOH only 0.2%. Samples of  $KNO_3$  and nitrocellulose-A and -B, giving 13.84, 13.86, and 13.75% N by the Lunge (L) method, gave 13.79—13.81, 13.83—13.88, and 13.74% in the Schulze-Tiemann method when the gas was measured over 45—50% and 30% NaOH solution, and 13.66, 13.73, 13.62% when read over  $H_2O$ . With nitrocellulose, the Lunge-Lubarsch method gives results that are 0.17% higher than those obtained by method L. This is due to  $CO_2$  evolved from the nitrocellulose during its dissolution in the nitrometer. Samples of guncotton, dissolved and kept in closed weighing bottles, gave by method L, when tested immediately 13.43%, after 20 hr. 13.36%, and after 4 days 13.33% N. Samples kept in open bottles gave 13.37, 12.61, 10.32%. Corresponding figures for collodion were: (in closed bottles) 10.86, 10.75, 10.74%; (in open bottles) 10.82, 10.33, 9.4%. Method L is recommended, but samples should be tested at once or kept in well-stoppered bottles. W. J. W.

**Determination of o-nitrotoluene in nitrocellulose powder by the immersion refractometer.** J. A. O'CALLAGHAN and S. G. COOK (Ind. Eng. Chem. [Anal.], 1933, 5, 333—334).—The powder is extracted with  $Et_2O$ , the extract evaporated to dryness, and the  $o-C_6H_4MeNO_2$  (I) distilled with steam into pure  $Et_2O$ . (I) is then determined by the  $n$  of the  $Et_2O$  solution. E. C. S.

**Determination of diphenylamine by bromination, especially in the analysis of smokeless powders.** L. GALATIS and J. MEGALOIKONOMOS (Z. ges. Schiess- u. Sprengstoffw., 1933, 28, 273—277).—A red coloration of the pptd.  $NH(C_6H_3Br_2)_2$  (I) is avoided by diluting the Br with 3—5 vols. of EtOH. To obtain a pure product of satisfactory m.p., EtOH-Br must be used in such amount that the final colour matches that of 0.1N- $K_2Cr_2O_7$ . Corrections are given for the solubility of (I) in MeOH and EtOH at 5°, 15°, and 30°, and the method of analysis is detailed. W. J. W.

**Determination of stabilisers in smokeless powders.** F. BECKER and G. A. HUNOLD (Z. ges. Schiess- u. Sprengstoffw., 1933, 28, 233—237, 284—286).—To determine  $NHPh_2$  (I), the powder is decomposed with 25% KOH and the (I) distilled in steam into NaCl. The distillate is extracted with  $Et_2O$ , EtOH is added, and after evaporation of  $Et_2O$  the residue is treated with  $H_2O$ . While it is cooling, Br is added till a red colour persists, and the pptd.  $NH(C_6H_3Br_2)_2$  is collected, washed with EtOH, and dried. When centralite (C) is present dibromocentralite remains dissolved during the Br pptn. Its amount is determined in a separate sample by treating the distillate with  $Et_2O$  and obtaining the total (I) + C, provided no camphor or  $NO_2$ -compound is present. If they are present, the  $Et_2O$  solution is evaporated, the residue dissolved in  $CCl_4$ , the (I) pptd.

with HCl, the filtrate evaporated to remove excess of HCl, and the C determined by known methods. To separate (I) from akardite (A; diphenylurea), the powder is extracted with  $CHCl_3$ , and the solution evaporated under vac. The residue after treatment with  $H_2O$  is distilled, only (I) passing over. This is extracted with  $Et_2O$ , and the solution evaporated under vac., the residual (I) being dissolved in  $CCl_4$  and titrated. The residue in the flask is evaporated to dryness, dissolved in  $CHCl_3$ , transferred to a titrating flask, the  $CHCl_3$  evaporated under vac., and the residue treated with  $CCl_4$  and titrated. C in presence of A is determined by extracting the powder with  $CHCl_3$ , evaporating the solvent, treating the residue with pure  $CCl_4$ , and titrating with Br. A second sample, after decomp. with alkali, is distilled in steam. Part of the A is converted into (I) and distils over with the C. The distillate is treated with  $Et_2O$  and, after evaporation, dissolved in  $CCl_4$ . (I) is removed by treatment with HCl, and the C determined with Br, the result, subtracted from that obtained with the first sample, giving the amount of A. W. J. W.

**Heat of formation of nitrocellulose, nitroglycerin, and other constituents of propellant powders.** A. SCHMIDT and F. BECKER (Z. ges. Schiess- u. Sprengstoffw., 1933, 28, 280—282).—Vals. in the lit. for heat of combustion of various explosives are more correctly those for heat of explosion, as they do not relate to complete combustion to  $CO_2$  and  $H_2O$ . Using Prettre's formula (A., 1932, 229) for determining the composition of nitrocellulose from the N content, and 94.5 and 67.5 kg.-cal./mol. for  $CO_2$  and  $H_2O$  (liquid), respectively, the heats of combustion of nitrocelluloses of 11.05—14.12% N ranged from 2580 to 2208 g.-cal./g. Heats of formation fell with increasing N from 754 to 500 g.-cal./g., and showed an almost linear relationship. The heat of combustion of nitroglycerin was 1639 g.-cal./g., agreeing with Rinckenbach's 1631 g.-cal./g., whose val. of 362 g.-cal./g. for the heat of formation is therefore the most reliable (cf. B., 1927, 27). Heats of combustion and of formation of various stabilisers are tabulated. W. J. W.

**Detonation of explosives.** A. SCHMIDT (Z. ges. Schiess- u. Sprengstoffw., 1933, 28, 209—213, 243—244, 247).—Polemical. Friederich (B., 1933, 493) confuses the velocity of directed explosion gases with that of directed atoms or mols. and seeks to explain hydrodynamic phenomena from the viewpoint of the kinetic gas theory. Detonation velocity is not the same as velocity of the explosion products, nor is the latter identical with mean mol. velocity. Friederich's contention that at highest  $d$  the last-named val., or the detonation velocity, is linearly related to sp. pressure is unsound. At the highest  $d$  the former velocity is < 2000, and the latter 5000—8000 m./sec. W. J. W.

**Detonation of explosives.** W. FRIEDERICH (Z. ges. Schiess- u. Sprengstoffw., 1933, 28, 213—215, 244—247).—A rejoinder to Schmidt (cf. preceding abstract). The relationship between detonation velocity and velocity of the explosion gases is justified. The author's formula is an application and extension of Muraour's formula for the combustion velocity of propellant powders. Schmidt's criticisms are of a general nature and do not apply to the special conditions in the explosion front. W. J. W.



**Composition of the waste acid as a factor in the manufacture of nitroglycerin.** G. WALLERIUS (Z. ges. Schiess- u. Sprengstoffw., 1933, 28, 169—172, 203—206, 247—250, 282—284).—The highest yields are not obtainable when the waste acid (I) has the lowest solvent capacity. Yield is a function of the amount of (I) and its solvent capacity for nitroglycerin (II), and the best results are obtained when the product of these, the "loss factor," is lowest, the min. amount of (II) being then dissolved. This occurs when the  $H_2O$  content of (I) is  $>$  that of (I) having the min. solvent capacity. Reduction of the amount of (I) without appreciably increasing the solubility of (II), in order to promote high yields, may be effected by increasing the  $HNO_3$  at the expense of the  $H_2SO_4$ . Incidentally, this enables the ratio of mixed acid to glycerin to be decreased. The solubility of (II) in (I) was determined with four series of mixed acids, having  $H_2O$  contents of 19.10, 17.05, 15.20, and 12.95%, respectively, and the results are plotted for various compositions. To obtain the highest yield from mixed acid containing 2%  $H_2O$ , the acid must contain 45.65%  $HNO_3$  and the ratio of mixed acid to glycerin must be 6.20:1. The gross yield will be 234.9%, the (I) containing 14%  $HNO_3$  and 16.75%  $H_2O$  + glycerin esters, the ultimate net yield being 232.6%. The substitution of recovered  $H_2SO_4$  for oleum is discussed. W. J. W.

See also A., Nov., 1106,  $Cd(N_3)_2$ . 1119, System  $NHPh_2$ -centralite. 1123, Thermal decomp. of trinitroazidobenzene and other polynitropolyazidobenzenes. 1129, Prep. of  $NH_4N_3$ . 1149, Action of Na on aliphatic diazo compounds.

## PATENT.

**Manufacture of [low-density] blasting explosives.** H. W. CLAPHAM, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 399,553, 7.4.32).—The carbonaceous material consists of peat, having  $d > 0.22$  g./c.c. when under a pressure of 25 lb./sq. in., so that the  $d$  of the explosive may be as low as 0.6 g./c.c. W. J. W.

## XXIII.—SANITATION; WATER PURIFICATION.

**Control of atmospheric pollution.** G. BATA and E. LECLERC (Bul. Soc. Chim. România, 1933, 15, 13—38).—Daily outputs of  $CO_2$ ,  $SO_3$ , and other gases from domestic chimneys, power stations, limekilns, and glass works are calc. These figures, however, have little val. unless dilution, gas temp., and the height of chimneys are stated. In particular, the evolution of  $SO_3$  and  $H_2SiF_6$  from the concn. of dil.  $H_3PO_4$  varies rapidly as concn. progresses, and these products may possibly be entrained with soot. An experiment on the quenching of coke showed that 7.8 g.  $H_2S$  per ton of coke were produced. Dust deposited from the atm. has been studied by the microscopical examination of plates coated with vaseline and exposed for a month or less. Wind does not affect such plates, but they are spoilt by heavy rain. Other apparatus is reviewed. Various known methods for determining the gaseous impurities in air are outlined. C. I.

**Gram-specificity of disinfection processes.** M. GUTSTEIN (Arch. Mikrobiol., 1933, 4, 248—256).—Difference in the toxic action of dyes toward bacteria

are ascribed to the internal  $p_H$  of the organisms concerned. Thus the high toxicity of basic dyes to Gram-positive bacteria (I) is due to the positive  $p_H$  gradient between the organism and the dye solution, penetration of which is thereby favoured. The resistance of Gram-negative bacteria (II) to basic dyes, the inverse effects of picric acid on (I) and (II), and the influence of neutral salts on the toxicity of dyes are explicable on this basis. A. G. P.

**Industrial preparation of doubly-distilled water. Physico-chemical characteristics of water.** R. CLIQUET, J. GUILBERT, and H. PÉNAU (J. Pharm. Chim., 1933, [viii], 18, 321—334).— $H_2O$  is distilled in a Cu still and redistilled in an all-glass (Pyrex) apparatus (capacity 50 litres) at a rate of 3 litres/hr. It then contains  $CO_2$  about 0.5 mg./litre,  $Pb < 5 \times 10^{-8}$ ,  $Cu < 5 \times 10^{-8}$ ,  $B < 2 \times 10^{-8}$ ,  $NH_3 < 2 \times 10^{-7}$ , org. matter nil, dry residue 5 mg./litre, and had the following const.:  $\kappa < 2-3 \times 10^{-6}$  (originally  $4 \times 10^{-4}$ , after first distillation  $1-2 \times 10^{-4}$ ),  $\gamma$  72—74 dynes,  $p_H$  5.5 due to traces of  $CO_2$ . S. C.

**Water of crystallisation in total solids of water analysis.** V. P. SOKOLOFF (Ind. Eng. Chem. [Anal.], 1933, 5, 336—337).—Direct determination of  $H_2O$  of crystallisation is advocated. E. S. H.

**Lactose-fermenting organisms in Philadelphia's drinking water.** G. G. SCHAUT (J. Amer. Water Works' Assoc., 1933, 25, 1287—1294).—The raw  $H_2O$  is drawn from the rivers Delaware and Schuylkill, which have both previously received raw and treated sewage. Lactose-fermenting organisms (I) are present in the finished  $H_2O$  but have no sanitary significance so far as the health of the city is concerned. Their occurrence is governed neither by climatic conditions nor by the mineral characteristics of the  $H_2O$ ;  $H_2O$  temp., however, by affecting filter efficiencies, is important. For the removal of (I) a process involving a long sedimentation period and slow sand filtration is the most effective, whilst prechlorination and post-chlorination in conjunction with rapid sand filters are without avail. C. J.

**Coagulation [of water supplies] with aluminium sulphate.** A. G. NOLTE and W. A. KRAMER (J. Amer. Water Works' Assoc., 1933, 25, 1263—1278).—All waters have an optimum dosage for  $Al_2(SO_4)_3$  and a min. period of mixing to secure complete coagulation. Laboratory and plant trials, using  $\frac{1}{2}$  grain of  $Al_2(SO_4)_3$  per gal., show that good coagulation is obtained by mixing for 30 min. at 0.5—1.5 ft. per sec.; higher speeds and long mixing periods tend to break up the floc. An initial mix for 1—3 min. at high velocity is not harmful, and if followed by a mix at 1.0 ft. per sec. for the remainder of the period good results are obtained. In general, mixing at decreasing velocities has no advantage over const. velocities and there is no advantage to be gained by adding the coagulant to half the  $H_2O$  and mixing with the remainder after the floc has formed. C. J.

**Ammonia-chlorine treatment [of water] at Champaign-Urbana, Ill.** F. C. AMSBARY, JUN. (J. Amer. Water Works' Assoc., 1933, 25, 1251—125).—6 Filter troubles caused by Fe bacteria have been controlled by prechlorination at the rate of 20 lb. of  $Cl_2$



per 10<sup>6</sup> gals. after a preliminary sterilisation with NH<sub>2</sub>Cl. The distribution system has been freed from extensive growths of *Crenothrix* and *Spirophyllum* by treating the filter effluent with 1.0 p.p.m. of NH<sub>2</sub>Cl and continued flushing of the mains at night. C. J.

**Ammonia-chlorine treatment [of water] in Danville, Ill.** H. M. ELY (J. Amer. Water Works' Assoc., 1933, 25, 1257—1259).—By substituting NH<sub>2</sub>-Cl<sub>2</sub> treatment for plain chlorination operating costs have been materially reduced, algal growths eliminated, and complete sterilisation was obtained throughout the distribution system. C. J.

**Ammonia-chlorine treatment [of water] yields nitrites in effluent.** R. HULBERT (Eng. News-Rec., 1933, 111, 315—316).—The H<sub>2</sub>O at Detroit, Mich., is prechlorinated, coagulated with alum in presence of (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, filtered, and finally post-chlorinated. As a result of the summer Cl<sub>2</sub> demand of the final effluent being unusually heavy, investigations disclosed the presence of 0.05 p.p.m. of nitrite in the filter effluent, due to the oxidation of NH<sub>3</sub>. This not only absorbed Cl<sub>2</sub> but gave falsely high vals. for residual Cl<sub>2</sub> by the o-tolidine method on taking readings after 30 min. in daylight. Waste of Cl<sub>2</sub> by oxidising NO<sub>2</sub>' to NO<sub>3</sub>' may be avoided by permitting sufficient NH<sub>3</sub> to pass the filters to fix all the post-Cl<sub>2</sub> as NH<sub>2</sub>Cl, and correct Cl<sub>2</sub> vals. can be obtained as above in presence of nitrites by reading quickly after keeping for 10 min. in darkness. C. J.

**Chlorination [of water] in presence of traces of ammonia.** L. B. HARRISON (J. Amer. Water Works' Assoc., 1933, 25, 1260—1262).—When raw H<sub>2</sub>O is treated with NH<sub>3</sub> and Cl<sub>2</sub> and the dose so regulated that the filter effluent is free from Cl<sub>2</sub>, as determined by the o-tolidine test, the NH<sub>3</sub> remaining is available for the formation of NH<sub>2</sub>Cl in the finished H<sub>2</sub>O. A period of contact should be allowed before distribution, as NH<sub>2</sub>Cl requires 2—2.5 hr. to produce its max. effect. It is advisable that the Cl<sub>2</sub> should be completely utilised in that period as residuals > 0.05 p.p.m. may produce NH<sub>2</sub>Cl odours. C. J.

**Chlorination and dechlorination of drinking water.** W. PLÜCKER and H. GAUTSCH (Z. Unters. Lebensm., 1933, 66, 62—81).—0.05—0.20 mg. of free Cl<sub>2</sub> per litre acting for 1 hr. is a more effective disinfectant against *B. coli* than either NaOCl or NH<sub>2</sub>Cl at equiv. concn. In the presence of 0.01 mg. of Ag (as AgNO<sub>3</sub>) per litre, 0.05 mg. of Cl<sub>2</sub> per litre is effective in 15 min. Similar results are obtained with 0.02 mg. of Cu (as CuSO<sub>4</sub>) per litre. Neither Ag nor Cu is effective unless the H<sub>2</sub>O is filtered. Cl<sub>2</sub> adsorbed on charcoal filters is best removed by aq. NH<sub>3</sub>. Peat can replace charcoal in filters for removing Cl<sub>2</sub>. Mn in the peat catalyses Cl<sub>2</sub> → HCl. Little infection or disinfection takes place during filtration of H<sub>2</sub>O through peat. E. C. S.

**Determination of nitrates in water [by the indigo method].** O. MAYER (Z. Unters. Lebensm., 1933, 66, 193—200).—5 c.c. of H<sub>2</sub>O are treated with 1 drop of a solution containing 5% each of HgCl<sub>2</sub> and NaCl, 6 c.c. of H<sub>2</sub>SO<sub>4</sub> are added, and the mixture is titrated with 0.032% indigotin (I) in 10% H<sub>2</sub>SO<sub>4</sub> until a blue or green tint persists for 5 min. If > 3 c.c. of (I) are required, the H<sub>2</sub>O is appropriately diluted. E. C. S.

**Treatment of water for industrial purposes.** J. O. MEADOWS (J. Amer. Water Works' Assoc., 1933, 25, 1216—1220).—A discussion of the most suitable type of H<sub>2</sub>O and means for obtaining supplies, for many industries including paper, textiles, brewing, aerated waters, cellophane, and leather. Attention is called to the use of NH<sub>2</sub>Cl for preventing slimy growths of capsulated bacteria in paper-mill equipment. C. J.

**Oil-field water analysis. III. Estimation of sodium by dihydroxytartaric acid.** A. H. K. RADJY and C. E. WOOD (J. Inst. Petroleum Tech., 1933, 19, 845—862; cf. B., 1933, 366).—Dihydroxytartaric acid (I) or its Na salt may be titrated with aq. KMnO<sub>4</sub> in the presence of V<sub>2</sub>O<sub>5</sub> as catalyst or with aq. Ce(SO<sub>4</sub>)<sub>2</sub>. Details are given for the determination of Na by pptn. with the K salt of (I). The presence of NH<sub>4</sub> causes low, and of Mg high, results. D. K. M.

**Composition of waste waters from [beet-]sugar factories.** J. VONDRÁK (Chim. et Ind., 1933, 29, Spec. No., 1141—1142).—Effluents from various Czechoslovakian sugar factories were conc. to syrup and analysed. Apart from those which had fermented, their composition, calc. on total solids, approx. resembled that of beet molasses in respect of polarisation, sol. ash, and org. non-sugars. The total N was only 1/4—1/2 that in molasses and the amount of betaine was very low, especially in pulp waters, but the protein-N was much > in molasses. J. H. L.

**Chlorination of [beet-sugar] factory waste water.** E. SAILLARD (Suppl. Circ. Hebd., 1933, No. 2308).—In solutions containing 0.3—10.0% of sucrose (I) and having purities from 62° to 91° there is no destruction of sucrose by 10 g. of Cl<sub>2</sub> per cu. m. In pure solutions, however, the (I) is perceptibly attacked. Provided the solution be well buffered, the use of Cl<sub>2</sub> for deodorising and purifying beet-factory waste waters is permissible, even when the H<sub>2</sub>O is returned to the diffusion battery. J. P. O.

**HF and toxic fog.**—See VII.

See also A., Nov., 1132, Determining NH<sub>3</sub> in sea-H<sub>2</sub>O. 1208, Oligodynamic action of Ag.

#### PATENTS.

**Germicidal and detergent substances.** A. R. CADE and H. O. HALVORSON (U.S.P. 1,901,434, 14.3.33. Appl., 15.10.28).—The germicidal action of solutions of alkalis (e.g., hydroxides, carbonates, phosphates, etc. at p<sub>H</sub> 10—12.6) is increased by the addition of 0.1—5% of unsaturated soap (Na oleate, resinate, etc.). E. L.

**Preparations for care of the teeth and mouth.** HENKEL & Co., G.M.B.H. (B.P. 399,917, 18.5.32. Ger., 3.9.31).—The preps. contain H<sub>2</sub>SO<sub>4</sub> esters of higher aliphatic alcohols, e.g., lauryl or myristyl, or the mixture obtained by the reduction of coconut oil acids, incorporated with vegetable mucilages, org. acids, glycerin, oxidising agents, abrasives, etc., as desired. L. A. C.

**Depilatory compound [for toilet use].** R. H. MCKEE and E. H. MORSE, Assrs. to A. MERZ (U.S.P. 1,899,707, 28.2.33. Appl., 4.1.32).—A sol. stannite containing about 3.5% SnO is prepared by pptg. Sn(OH)<sub>2</sub> from SnCl<sub>2</sub>, washing, and stabilising the product with an alkali salt of a polybasic org. OH-acid and an excess of buffered alkali. D. W.



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P

322 / 33 / 1