

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MAR. 25 and APRIL 1, 1932.*

I.—GENERAL; PLANT; MACHINERY.

Corrosion in marine boilers and use of fresh water as remedy. H. JANSSEN (Korrosion u. Metallschutz, 1931, 7, 108—111; Chem. Zentr., 1931, ii, 1342).—Distillates of sea-water prepared on board ship always contain $MgCl_2$ and other salts; the condensate is acid. The evaporators are preferably heated with exhaust steam and the distillates neutralised with NaOH. The val. of $(NaOH + Na_2CO_3; \text{mg. per litre})/4.5$ should be 400—2000. A. A. ELDRIDGE.

Float valves with counter-weights. H. STAFFEHL (Chem.-Ztg., 1932, 56, 76—78).—Const.-level valves operating by means of a float either with direct lever action (pressures up to 4 atm.) or by a lever and screw (pressures up to 8 atm.) are described. Modifications suitable for various liquids are mentioned. A back-pressure float valve prevents condenser water returning to the steam chest of a turbine. C. IRWIN.

Manometers for low pressures. S. DUSHMAN (Ind. Eng. Chem. [Anal.], 1932, 4, 18).—The applications and limitations of different types are indicated. E. S. HEDGES.

Heat flow through granulated material. J. H. AWBERY (Phil. Mag., 1931, [vii], 12, 1152—1155).—Theoretical. H. J. EMELÉUS.

Bleaching earths. P. G. NUTTING (Ind. Eng. Chem. [Anal.], 1932, 4, 139—141).—Measurements of the filtering power and H_2O content of a number of clays are recorded. E. S. HEDGES.

Nomogram for reduction of gas volumes to 30 in. pressure and 60° F. D. G. BAGG (Gas J., 1931, 196, 730—731).—The nomogram described, for obtaining the "tabular number" for conversion of moist gas vols. into vols. at 30 in. pressure (saturated with water vapour) and 60° F., consists of 3 scales representing, respectively, tabular no. and temp., the tabular no. being also graduated with the logs. of the "gas-volume factors" (*i.e.*, 1/tabular no.). H. E. BLAYDEN.

Automatic remote control of boilers. J. M. YOUNG (J. Inst. Fuel, 1932, 5, 217—223).

High-Cr steels. Thermocouples.—See X. **Olier continuous diffusion.**—See XVIII. **Water-softening plant.**—See XXIII.

See also A., Feb., 115, CCl_2F_2 as refrigerant. 137, Lustre-meter and spectrophotometer. Automatic colorimetry. Measurement of temp. potentiometrically. 138, Automatic p_H recorder. Extraction apparatus for large quantities of solid material. Const.-pressure device for autoclaves. 139, Determining particle size.

PATENTS.

Furnace for sintering, roasting, and firing. VEREIN. STAHLWERKE A.-G. (B.P. 364,698, 14.10.30. Ger., 6.11.29).—A shaft furnace is provided with supply and delivery pipes so that it can be operated on down-draught with solid fuel or on updraught with gaseous fuel. Poke-holes in the wall and a bottom discharge are provided. B. M. VENABLES.

Furnaces. A. CHRISTENSEN (B.P. 364,920, 4.3.31).—The combustion of garbage or other feebly combustible material is started on a fixed or moving grate and completed in an adjacent rotating cylinder, without additional air. B. M. VENABLES.

Refractory structures of furnaces. L. S. LONGNECKER (B.P. 364,955, 27.3.31. U.S., 4.4.30).—Hooks are described by means of which firebricks may be suspended from a beam to form a roof. B. M. VENABLES.

Drying kilns. A. E. WHITE. From B. F. STURTEVANT Co. (B.P. 364,560, 28.8.30).—In a kiln having forced circulation, the supply and return ducts are provided with connexions and valves so that their functions can be reversed at intervals. Preferably there are two stacks of timber (or other goods being dried) with a wide inlet-outlet passage in the middle and a narrow one at each side. Also, to distribute the air evenly, the apertures leading from the ducts to the drying chambers are individually small and in the aggregate are less than half the area of the ducts. B. M. VENABLES.

Drying of road-making aggregates and other broken or granular material. J. F. WAKE (B.P. 364,762, 21.11.30).—A vertical casing surrounds a number of alternate upright and truncated inverted conical or pyramidal deflectors over which the material flows downwards. Passages for the upward flow of drying medium are provided by axial pipes and by louvre-like openings in the cones. B. M. VENABLES.

Steam superheaters and other tubular heat-exchange apparatus. SUPERHEATER Co., LTD., Assees. of COMP. DES SURCHAUFFEURS SOC. ANON. (B.P. 364,981, 30.4.31. Fr., 5.5.30).—A method of arranging the tubes of a superheater is described. B. M. VENABLES.

Apparatus for continuous treatment of solid substances under pressure [and heat]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 364,665, 4.10.30).—The reacting substances, at least one of which is preferably a fluid, are passed through a number of pressure-resisting tubes which are combined in a bundle and rotated. Heating by means of the

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

condensation of substances having a lower latent heat and higher b.p. than that of H_2O is excluded; other forms of external heat, preferably with preheating of any stable reagent, may be used. *E.g.*, in the hydrogenation of coal, the H_2 may be preheated to any desired extent, but the coal should not be much heated until subjected to the pressure and the action of H_2 .

B. M. VENABLES.

Materials suitable for use as insulators for heat or sound. A. MELVILLE, J. T. MILLAR, and R. S. T. HENRY (B.P. 364,314, 18.12.30).—Paper-mill sludge and kieselguhr separately have small binding qualities, but when mixed each constituent forms a binder for the other. The mixture must be allowed to dry without much disturbance because the insulating properties are largely due to the voids left by evaporation of water. For temp. below 176° the cellulosic machine sludge should be used, and for higher temp. the CaO sludge is preferable.

B. M. VENABLES.

Pulverisers for coal or other material. P. W. GRIFFIN (B.P. 364,954, 27.3.31).—The hammers of a disintegrator run closer to the casing at the bottom than at the top, leaving an open space in the upper part to which the material is fed.

B. M. VENABLES.

Mixing and kneading apparatus. E. EPPLER, and BAKER PERKINS, LTD. (B.P. 364,588, 3.10.30. Ger., 15.5.30).—A bowl is rotated at such a speed that considerable centrifugal force is developed, and inclined scrapers are provided which deflect the material towards the centre of the bowl, these being pivotally mounted so as to be set closer to or further from the side and bottom of the bowl according to the material treated.

B. M. VENABLES.

Centrifugal separators. T. BROADBENT & SONS, LTD., and W. HALLITT (B.P. 364,348, 12.1.31).—A centrifuge having a differentially driven helix for the continuous discharge of solid matter has the wall of the bowl divided into three zones, viz.: (b) a middle one, preferably conical, for the separation; (a) one sloping inwardly upon which the solids are scraped inside the liquid level and are thus drained; and, in the other direction, a zone (c) for clarification, preferably having a cylindrical wall.

B. M. VENABLES.

[Automatic regulation of the] concentration or thickening of liquids such as pulps and ore slimes. L. MELLERSH-JACKSON. From MERCOR CENTRIFUGAL SEPARATOR CO., LTD. (B.P. 364,567, 2.10.30).—With the object of obtaining pulp of const. thickness from a gravity or centrifugal thickener, some or all of the thick pulp is continuously passed through a counterpoised "testing" vessel, the outflow from which when the vessel is heavy passes to final delivery along with that portion (if any) of the thick pulp that does not pass through the vessel. When the vessel rises, owing to its being light, the outflow is diverted back to the thickener. At starting up, when the pulp is likely to be thin, it is preferable to pass all of it through the testing vessel, and thence (probably) back to the thickener, so that the net delivery is nil, until the pulp becomes thick enough; then a by-pass may be opened so as to get a large output with a comparatively small testing vessel.

B. M. VENABLES.

Regulating device for supply of flushing liquid to centrifuges. AKTEB. SEPARATOR (B.P. 364,506, 15.6.31. Swed., 21.6.30).—The supply of flushing liquid is held in strict proportion to the liquid to be separated by passing them both through vessels, having adjustable bottom outlets, suspended at opposite ends of a balance beam which operates a valve in the supply pipe for flushing liquid.

B. M. VENABLES.

Centrifugal dust separators and collectors. H. VAN TONGEREN (B.P. 364,449, 2.4.31).—The dusty gas is passed through a number of curved passages in series, a layer of conc. dust being sheared off at each stage. Also, in order to reduce the distance the dust has to travel to reach the outer wall, the curved passages may be subdivided in parallel. The angle (radians) subtended by the curve \div the greatest distance (m.) a particle has to travel transversely to reach the layer that is sheared off should be at least 500 in the first stage and progressively greater in the subsequent stages.

B. M. VENABLES.

Apparatus for homogenising liquids. H. CORBLIN (B.P. 364,921, 4.3.31).—The liquids are passed through grooves formed on the surface of a plate (access being obtained through holes in the plate) against which a flexible diaphragm is pressed by fluid pressure. A convenient source of the pressure is a bottle of liquefied gas. Projections extending into the grooves may be fastened to the diaphragm.

B. M. VENABLES.

Means of indicating the specific gravity of liquids. THREE STAR ACCUMULATORS, LTD., and W. H. DURANT (B.P. 364,858, 16.1.31).—Two or more balls, cylinders, or other elements are constructed of materials of slightly different sp. gr. and stuck together to form an indicator which, besides floating or sinking according to major changes in sp. gr., will capsize into a different position when floating owing to minor changes in sp. gr.

B. M. VENABLES.

Rotary-hearth furnaces. HEVI-DUTY ELECTRIC CO., ASSEES. OF E. L. SMALLEY (B.P. 366,409, 24.6.31. U.S., 10.7.30).—See U.S.P. 1,806,027; B., 1932, 83.

Production of high vacua. W. W. TRIGGS. From KEMET LABORATORIES CO. (B.P. 365,801, 28.1.31).—See U.S.P. 1,800,134; B., 1931, 1031.

Furnace walls. TAYLOR STOKER CO., LTD., and J. W. HITCHCOCK (B.P. 366,279, 3.2.31).

Production of [impregnated fibrous strip] frictional coverings [from armoured asbestos]. H. KATTWINKEL (B.P. 365,988, 21.8.31).

Org. compounds containing F [as refrigerants].—See III. **Heat-insulating substances.**—See VIII. **[Metal-]melting furnaces.**—See X. **Resistance material. Separating particles from gases.**—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Preparation of coal from the petrographic viewpoint. K. LEHMANN and E. HOFFMANN (Brennstoff-Chem., 1932, 13, 21—29).—The methods of the petrographic analysis of coal are outlined (cf. Stach, B., 1929, 382; Lehmann and Stach, Glückauf, 1930, 66,

289). A large-scale method of separating the petrographic constituents has been developed, based on their different behaviour towards crushing by elastic impact (cf. B.P. 363,618; B., 1932, 247). Removal of fusain from a coal increases its val. for briquetting by lowering the proportion of pitch required to produce briquettes of a given strength. Removal of fusain also lowers the P content of the coal, raises the m.p. of the ash, and often improves the coking properties. The fusain can be burned in pulverised-fuel installations. Other possible technical applications of the process are discussed. In one case the vitrain fraction of the coal proved more suitable for the production of metallurgical coke than the original coal, whilst the durain was more suitable for the production of domestic fuel by low-temp. carbonisation.

A. B. MANNING.

Genesis of coals. II. "Coalification" ("Inkohlung") of cellulose and lignin in neutral medium. III. "Coalification" of resins and waxes in neutral medium. IV. Carbonisation of artificial coals. E. BERL and A. SCHMIDT (*Annalen*, 1932, 493, 97—123, 124—135, 135—152).—II. Investigations on the production of artificial coals are reviewed briefly. The action of H_2O at 150—350° and saturated NaCl at 300—400° (under correspondingly high pressures) on cellulose (I) and lignin (II) is investigated by the method previously used (A., 1928, 742). Amorphous, dark-coloured products are obtained from (I) only above 250°; those from (II) retain the original structure even at 400°. The amount of insol. solids formed is smaller with (I) than with (II), but more H_2O -sol. and gaseous products result from (I). More solid is obtained from (I) with NaCl than with H_2O at 300—350°; this is ascribed to coagulation of dissolved colloidal org. matter by NaCl. The C content of the solids from (I) increases with rise in temp. (pronouncedly at 200—250°); with (II) there is a gradual increase for C, but the OMe content falls (above 250°). OMe-free products are obtained from (II) only with NaCl at 350—400°. The alkali-sol. humic acid content of the solid products from (I) diminishes with raised reaction temp. (particularly at 225—275°); those from (II) show first a slight increase (max. at 300°) and then a gradual decrease. The H_2O -sol. products formed are: HCO_2H , $AcOH$, $COMe_2$, and sugar-like reducing compounds (max. at 225°, then decreasing rapidly with rise in temp., especially in presence of NaCl) from (I); CH_2O , $MeOH$, HCO_2H , $AcOH$, $COMe_2$, (probably) higher fatty acids, and mono- and di-hydric phenols [including $o-C_6H_4(OH)_2$] from (II). The reducing power of the solution from (II) and H_2O increases with rise in temp., and is probably due to CH_2O . Fe is found in all the solutions and arises from the apparatus. The gaseous products formed (mainly CO_2 , with some H_2 , CO , CH_4 , and C_nH_n) increase with rise in temp. in both cases.

III. The resin content of different bitumens and montan waxes varies from 6.6 to 74%. When pine resin (III) and carnauba wax (IV) are heated with H_2O or NaCl (as above), "coalification" occurs. Tough, viscous products are obtained from (III) at 300—400° containing about 84.5% C; the acid and sap. vals. of the products both decrease with raised temp., but the ester no. remains more or less const. CO_2 is the main

gaseous product, but, compared with (I) and (II), increased amounts of H_2 and CH_4 result. Brittle substances (C about 82%) are produced from (IV); the sap. vals. of these decrease with rise in temp., whilst the acid vals. increase. In both cases the "coalification" gives products resembling natural lignite-bitumens.

IV. Compression of the "coalification" products from (I) and pine wood at 2000—3000 kg./sq. cm. gives compact solids (V and VI, respectively) having the appearance, and showing the conchoidal fracture, of coal. Similarly, (II) affords a duller, more brittle, and more friable product (VII). The % of coke, tar, aq. distillate, and gas (by difference) obtained when these artificial coals are carbonised at 500—550° are: (V) 67, 13.4, 9, 10.6; (VI) 66, 15.8, 10.5, 7.7; (VII) 77.5, 2.4, 14, 5.1; the primary tars contain phenols, phenolcarboxylic acids, aliphatic and aromatic hydrocarbons, and small amounts of bases. Conversion of aliphatic into aromatic substances must occur during the "coalification" of (I). The amounts of phenols in the primary tars from (V) and a young gas coal are similar; less phenols are produced from older coals. High-temp. carbonisation gives the following % of coke, tar, liquid hydrocarbons (mainly C_6H_6), and gas (including loss): (V) 61, 4, 0.6, 34.4; (VI) 56, 1.7, 1.1, 41.2; (VII) 65, 1.5, 0.3, 33.2. Anthracene and $C_{10}H_8$ are present in the tars from (VII) and a "coalification" product of (I) with 0.05N-NaOH.

H. BURTON.

Carbonisation and chemical utilisation of coal. C. BERTHELOT (*Chim. et Ind.*, 1931, 26, 763—780; 1932, 27, 18—30).—Processes for low-temp. carbonisation and artificial anthracite preparation in France, Germany, Great Britain, and America are reviewed and methods of treating the primary tar are outlined. The economics of the processes are discussed. Blending of coals of high volatile content with coke dust gave increased amounts of large-size metallurgical coke in the Ruhr and, taking into consideration coke-grinding costs, effected appreciable economies. Methods in use for the synthesis of NH_3 and N products from coke via water-gas are described and discussed in conjunction with methods of synthesis of MeOH. The present economic position is reviewed.

H. E. BLAYDEN.

Swelling and caking processes in the thermal treatment of coal. P. SCHLÄFFER and E. MÜLLER (*Eidgen. Materialprüf. E.T.H., Zürich*, 1931, Ber. No. 55, 47 pp.).—Hypotheses advanced to explain coke formation and caking capacity of coals are reviewed. Four German coals were investigated; the softening temp. (penetrometer method), degrees of swelling, swelling pressures and caking indices, and permeability to gas flow during carbonisation were determined and the gas composition and yields on heating two coals (those of max. and min. softening temp.) at a const. rate were measured. The softening temp., degree of swelling, swelling pressure, and caking power were greatly influenced by the rate of heating. Gas yields on heating showed a max. at 450°. The temp. of min. permeability to flow of N_2 through the heated coal is shown to be the temp. of semi-coke formation. The change in properties on preheating a coal in a vac. is attributed to intramol. transformation of the coal substance.

H. E. BLAYDEN.

Effect of catalysts on the liquefaction of Fushun coal by the Bergius process. R. ABE (J. Soc. Chem. Ind., Japan, 1931, 34, 500—510 B).—Paraffin oil was used to reduce the coal to a paste, an initial H_2 pressure of 75 kg./sq. cm. was employed, and the reaction temp. was 400° . As catalyst, Fe_2O_3 was employed in the ratio of 1 pt. of Fe to 10 pts. of coal. The max. oil yield was 21.2% on pure coal, and after treatment for 30 min. it rapidly diminished, perhaps through cracking, with corresponding increase of gaseous hydrocarbons. The b.p. range of the oil fell rapidly with time of treatment. X-Ray examination showed that the reduced catalyst consisted of α -Fe and Fe_3O_4 ; FeO was absent. Tests with an Fe_3O_4 catalyst, which gave a much more viscous paste in the same proportions of Fe to coal, gave an oil yield of up to 27%, but after 60 min. it fell rapidly; the yield of gaseous hydrocarbons was less. In neither case was any H_2S or NH_3 formed. The properties of the oils produced are tabulated in detail. C. IRWIN.

Pitch coke. A. SANDER (Chem.-Ztg., 1932, 56, 73—74).—During the war coal-tar pitch was coked in Germany in large cast-Fe retorts arranged in batteries. The life of each retort ranged from 22 to 56 distillations according to the amount of care in heating and cooling. They held 1.0—2.5 tons of pitch, and each cycle of operations required 24—36 hr. Distillation was *in vacuo*. The yield was 50—60% of coke, 30—40% of oil, 4.6% of resin, some combustible gas, and NH_3 (which was removed by washing). The coking of lignite pitch is carried out in order to obtain valuable distillation products of petroleum type. It is impossible to obtain a coke of uniform composition in cast-Fe stills, the product being layered. It should contain 1—2% of volatile matter and $\geq 1\%$ ash and have d 1.35—1.65. The ash content is important in regard to the use of coke for electrodes. In 1929 pitch coking was begun in an old battery of coke ovens at Bochum. These have top feed and machine discharge and are working regularly. It is expected that pitch coke made in this way will replace petroleum coke in Germany for electrodes, and it may also prove a substitute for wood charcoal in the production of special steels. C. IRWIN.

Mechanism of combustion in a coal-dust flame. H. SCHWIEDESSEN (Arch. Eisenhüttenw., 1931—2, 5, 291—298).—The process of combustion in a coal-dust flame in a wrought-Fe furnace has been investigated by temp. measurements and gas analysis; the results are reproduced in diagrams showing the distribution of temp., CO_2 , excess air, and suspended dust, and the rate at which combustion of fixed C and volatile matter takes place. Temp., gas content, and combustion loss of the coal particles at any point depend solely on the time taken by the burning particles to reach that point; the expulsion of volatile matter from the coal particles takes place over a comparatively long distance as a skin of fused slag forms over their surface which tends to retard the evolution of gas. The rate of combustion of the particles is dependent on the O_2 concn. and on the extent to which they become covered with a slag skin. A. R. POWELL.

Fuel-gas analysis for heating value and combustion calculations. K. M. WATSON and N. H. CEAGLSKE (Ind. Eng. Chem. [Anal.], 1932, 4, 70—72).—The triple-combustion scheme of fuel-gas analysis described enables the average compositions and vols. of both saturated and unsaturated hydrocarbons to be determined. Equations are developed for calculating the heating vals. of mixtures of hydrocarbon gases from their average compositions. The error is generally $< 2\%$. E. S. HEDGES.

Determination of total sulphur in gases. E. LIEBER and R. ROSEN (Ind. Eng. Chem. [Anal.], 1932, 4, 90—93).—A modified Drehschmidt apparatus for determining the total S content of gases is described. The analysis requires from 1 to 100 litres of gas, combustion is completed in 1 to 4 hr., and the S in the absorbent medium is determined gravimetrically as $BaSO_4$. The mean error is 0.3%, corresponding with a mean error of 2.4% on a basis of the actual S content of the gas. E. S. HEDGES.

Separation of bitumen from the asphaltic rocks of Ragusa by treatment with aqueous saline solutions. G. ROBERTI (Annali. Chim. Appl., 1931, 21, 269—581).—The separation can be effected by Na_2SiO_3 , Na_2CO_3 , Na_3PO_4 , and K_2CO_3 solutions. The activity increases with the concn., but after several treatments it diminishes rapidly. The technical application of the process depends on the possibility of regenerating the activity of the exhausted solutions. O. F. LUBATTI.

Distillation and cracking of oils from the calcareous rocks of Ragusa. G. ROBERTI (Annali. Chim., Appl., 1931, 21, 563—568).—Fe turnings employed as catalytic agent produce an increase of those products of the distillation which are sol. in H_2SO_4 and decrease the yield of the aromatic substances. Cu turnings undergo rapid sulphurisation, but, as in the case of Fe, the desulphurisation of the gases is incomplete. The catalytic activity of activated C decreases rapidly in the process. The employment of exhausted rock as catalyst produces a lower yield of volatile oils, but favours the increase of aromatic substances. The quantity of volatile oils obtained varies from 4 to 24% of the bitumen contained in the rock. The quantity of light aromatic hydrocarbons obtained corresponds with 10% of the bitumen. O. F. LUBATTI.

Myers' demulsification test for bituminous emulsions. P. R. SMITH (Ind. Eng. Chem. [Anal.], 1932, 4, 105—106).—During this test (A.S.T.M., D-244-28 τ [1928]) the temp. of the emulsion and of the $CaCl_2$ solution should be controlled, preferably at $25^\circ \pm 1^\circ$. A sieve made of wire that will not be attacked by alkali must be used. E. S. HEDGES.

Removal of sulphur from benzene and middle oil by hydrogenation. G. ROBERTI (Mem. R. Accad. d'Italia, 1930, 2, Chim. No. 5, 15 pp.).—Hydrogenation at 100 atm. and 340° in presence of a CoS catalyst reduced the thiophen-S content of a specimen of benzene from 1.513% to 0.073%; non-thiophen-S (0.133%) was not removed to any great extent. The S content of an oil derived from bituminous shale was reduced from 2.92%

to 0.32% by catalytic hydrogenation at 100 atm. and 500°.

H. F. GILLBE.

Phytosterols and abietic acid as origins of the optically active constituents of mineral oils. M. A. RAKUSIN (Petroleum, 1932, 28, No. 3, 9—12).—The production of optically active unsaturated products by the disruption of betulin and abietic acid by the action of AlCl_3 is adduced as evidence that phytosterols and abietic acid are the parent substances which give rise to optical activity in mineral oils; these are considered as of org. origin.

H. E. BLAYDEN.

Specific heats of mineral oils. H. KRAÜSSOLD (Petroleum, 1932, 28, No. 3, 1—7).—The relation between sp. heat, temp., and sp. gr. of mineral oils is shown to be expressed with an accuracy of $\pm 3\%$ by $c = a + b \times (t - 15)$, where b is the temp. coeff., t the temp. in °C., and $a = 0.937 - 0.56d^{15}$ when d^{15} is > 0.9 , or $a = 0.711 - 0.308d^{15}$, when d^{15} is < 0.9 .

H. E. BLAYDEN.

Explosion limits of mineral oil vapour-air mixtures. F. PACHTNER (Petroleum, 1932, 28, No. 4, 1—4).—The results of investigations of the explosive limits of hydrocarbon gases and vapours mixed with air are reviewed and discussed with reference to explosive properties of vapours present in oil tanks. The explosive limits of the vapour-air mixtures may lie between 1.1 and 12.5%.

H. E. BLAYDEN.

Calorimetric determination of ethylene, propylene, and butylene in gas mixtures. J. DUBOIS (Przemysl Chem., 1931, 15, 390—394).—The C_2H_4 , C_3H_6 , and C_4H_8 contents of gas obtained by cracking petroleum are calc. from the following data: heats of combustion of the mixture before and after absorption of unsaturated hydrocarbons by oleum, content of C_2H_4 homologues (by absorption in 87% H_2SO_4), and unsaturated hydrocarbon content.

R. TRUSZKOWSKI.

Identification of diolefines in lower fractions from vapour-phase cracked gasoline. S. F. BIRCH and W. D. SCOTT (Ind. Eng. Chem., 1932, 24, 49—50).—Butadiene, piperylene, isoprene, and cyclopentadiene are identified by isolation as their additive compounds with maleic anhydride.

H. A. PIGGOTT.

Determination of lead tetraethyl in ethyl gasoline. E. L. BALDESCHWIELER (Ind. Eng. Chem. [Anal.], 1932, 4, 101—102).— PbEt_4 is converted into $\text{Pb}(\text{NO}_3)_2$ by shaking the gasoline with conc. HNO_3 . Two liquid layers are formed; they are separated and Pb is determined in the lower layer as PbSO_4 . The accuracy is within 0.02 c.c. of PbEt_4 per gal. The method is applicable to the determination of many other organo-metallic compounds.

E. S. HEDGES.

Corrosion test on casing-head gasolines. A. W. TRUSTY (Ind. Eng. Chem. [Anal.], 1932, 4, 33).—To 10 c.c. of gasoline two drops of Hg are added; if the surface of the Hg remains bright the test is negative. Most casing-head gasolines and some straight-run gasolines contain practically no S.

E. S. HEDGES.

Incorporation of graphite with motor oils. E. KADMER (Petroleum, 1932, 28; Motorenbehr., 5, 2—5).—The advantageous effects of lubrication with graphitised oils (e.g., "Kollag," "Oildag") are discussed. It

is shown that reduction of running costs, increase of power, and greater adaptability to varying conditions result from admixture of graphitised oils to lubricating oils.

H. E. BLAYDEN.

Technical application of the deposition method. I. Paraffin and ozokerite. N. FUCHS (Z. angew. Chem., 1931, 44, 962—965).—Study of the type and behaviour of the deposit which readily forms on any polished surface frequently yields much information regarding the nature of the material which forms the surface. Thus the rate of evaporation and of agglomeration of drops of moisture condensed on a paraffin surface are closely related to the oil content of the paraffin. The phenomena observed during the crystallisation of paraffin from a solution in oil are described in detail and discussed. It is possible by this "deposition method" to determine approx. the oil content of a wax. The results obtained with various materials correspond closely with those determined refractometrically; even when the oil content is as low as 0.05% the oil is free, and not in the form of a solid solution, as has been reported. The method permits also very accurate determinations of the temp. at which a wax commences to decompose.

H. F. GILLBE.

Bleaching earths. Nomogram for gas vols.—See I. Dyes sol. in oils.—See IV. Road tar-bitumen.—See IX. Titration of oil acidity.—See XI. Sulphonated oils as emulsifiers.—See XII.

See also A., Feb., 127, Flame temp. of mixtures of air with CH_4 and O_2 , H_2 , and with C_2H_2 . 132, Activation of C by CO_2 . 152, Hydrogenative cracking of polynuclear hydrocarbons. 155, Cracking of phenols with H_2 .

PATENTS.

Dressing or grading of coal. K. LEHMANN, Assee. of RHEINISCHE STAHLWERKE (B.P. 363,618, 11.7.30. Ger., 31.7.29).—Coal is separated into its constituents, vitrain, durain, and fusain, by subjecting it to resilient disintegrating or shattering action for such time that the constituents, by reason of their different resistances to shattering impacts, are respectively broken up into granules of different sizes. These are then separated by screening or similar means. The treatment may be carried out in stages and may include cleaning processes for removing shale and other impurities from the coal.

A. B. MANNING.

Apparatus for destructive hydrogenation of solid fuels. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 363,626, 21.8.30).—An electrically-heated, vertical, cylindrical vessel has a number of horizontal, stationary trays supported therein. In the bottom of each tray is at least one aperture for the passage of the fuel, which is conveyed slowly over each tray until it falls through to the next, by means of rotating arms attached to a central axis. An annular space may be provided between the trays and the walls of the vessel to facilitate circulation of the H_2 and volatile reaction products. The fuel is introduced in powdered form into the top of the vessel; H_2 under pressure may be introduced at the bottom and passed countercurrent to the fuel, or may be blown in in such a manner as to

pick up the coal dust and maintain it in suspension in the upper part of the vessel for a short time before allowing it to fall to the trays.

A. B. MANNING.

Extinguishing [ignited] coke. STETTNER CHAMOTTE-FABR. A.-G. VORM. DIDIER (B.P. 364,236, 3.11.30. Ger., 25.11.29).—H₂O is sprayed on to the hot coke in a container so designed that the steam produced is forced by its own pressure down through the coke. Suitable apparatus is claimed.

A. B. MANNING.

Manufacture of carbon and hydrogen. N. GRÜNSTEIN (B.P. 363,735, 3.10.30. Ger., 3.10.29).—CH₄ or other hydrocarbon gas or vapour is passed over a catalyst, e.g., Fe or Ni supported on C black or on a compound, e.g., MgO, which can afterwards be removed by chemical dissolution, at a temp. below that at which the hydrocarbon decomposes in the absence of a catalyst. The metal may be deposited on the C black by decomp. of the carbonyl, and may subsequently be removed, again as the carbonyl, by treating the product with CO at a suitable temp.

A. B. MANNING.

Gas generators. C. MARISCHKA (B.P. 364,338, 6.1.31. Austr., 7.1.30).—The temp. and height of the clinker layer in gas generators with a stepped revolving grate are indicated by a device consisting of an annular chamber which surrounds the generator in the vicinity of the grate and through which a const. stream of H₂O is passed, the rise in temp. of the H₂O giving the required indication. It is advantageous to subdivide the cooling jacket into a number of independent segmental chambers and also to provide a similar device, e.g., a water-cooled grate-hood, for indicating the temp. in the centre of the generator. The information so obtained facilitates control of the working of the generator.

A. B. MANNING.

Gas producers for generating continuously a gas mixture for ammonia synthesis. K. KOLLER and M. LENGYEL (B.P. 363,812, 11.12.30).—The producer is provided with a superimposed retort wherein coal is distilled in a current of hot inert gases (700—1000°) from an external source. The incandescent coke descends to the producer shaft, wherein it is brought into contact with a preheated (300—700°) air-steam mixture, the steam content of which is regulated to give a producer gas in which the H₂:N₂ ratio is 3:1. The pressures in the apparatus are so regulated that no admixture of the producer gas with the retort heating gases takes place.

A. B. MANNING.

[Water]-gas-making process and apparatus. TRENT PROCESS CORP. (B.P. 363,931, 15.4.31. U.S., 16.4.30).—Coal-oil "amalgams," anthracite, bituminous coal, or other finely-pulverised fuels are passed through a preheater which, e.g., may comprise a series of externally-heated horizontal tubes connected to form a tortuous path for the material which is forced there-through by screw conveyors. Provision is made for withdrawing evolved volatile matter from the preheater. The material, at about 700°, is then mixed with superheated steam, also at 700°, and passed through a tubular gasifying apparatus wherein it is converted into water-gas. The combustion gases from the steam-generating and superheating furnace are utilised for heating the

tubes of the gasifying apparatus and thence pass through the preheater.

A. B. MANNING.

Production of gas of high calorific power, and by-products, by gasification with oxygen under elevated pressure. METALLGES. A.-G. (B.P. 364,407, 23.2.31. Ger., 10.3.30).—Solid fuels are gasified with O₂ and steam under pressures of 10—30 atm. or more in a producer having doubly-sealed lock chambers for the introduction of the fuel and the discharge of the ash, and a vertical rotating shaft to which are attached arms which discharge the ash into the lock chamber. The conditions of temp., pressure, and quantity of steam used are so selected that substantial amounts of CH₄ are formed. The gases may be purified from CO₂ etc. while still under pressure.

A. B. MANNING.

Low-temperature purification of combustible gases for distant supply. F. LENZE (B.P. 364,091, 22.9.30. Ger., 7.6.30).—The gas is cooled by means of non-congealable refrigerants to a temp. corresponding to the lowest temp. to be expected in the supply mains. The cooling is preferably carried out in stages; the wash oil for removing C₁₀H₈ may be used as the refrigerant in the first stage, and hygroscopic refrigerants, e.g., glycerin or aq. CaCl₂, in subsequent stages. The H₂O content of the gas is thereby sufficiently reduced to effect a considerable increase in the efficiency of the subsequent benzol recovery by adsorption with activated C, or similar means. Suitable plant is described.

A. B. MANNING.

Removal of ammonia and hydrogen sulphide from gases. H. LEITHAUSER (B.P. 363,848, 5.1.31. Ger., 6.1.30).—The gas is scrubbed with an Fe(OH)₃ suspension to remove part of the H₂S, the process being so regulated that sufficient H₂S remains to ensure an NH₃:H₂S ratio of 4:1 in the scrubbed gas. These constituents are then removed together by scrubbing with an NH₄ sulphite-bisulphite liquor.

A. B. MANNING.

Manufacture of asphalt. N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 363,972, 10.7.31. Holl., 13.8.30).—A petroleum residue (5% of asphaltenes) is "blown" so as to increase its asphaltene content to 7% or more, and is then distilled with or without steam, and under atm. or reduced pressure. A product is obtained which conforms to the specification for road asphalt.

A. B. MANNING.

Thermal decomposition of hydrocarbons. N. V. DE BATAAFSCHE PETROLEUM MAATS., Assees of R. W. MILLAR (B.P. 364,418, 6.3.31. U.S., 10.3.30).—When O₂ or a gas containing O₂ is burned in an atm. of a gaseous hydrocarbon or under the surface of a liquid hydrocarbon C₂H₂ is produced. More C₂H₂ is produced when the O₂ is burned in a jet of small diam. than when one of large diam. is used, and when the gas is supplied through several jets in one vessel than when use is made of the same number of jets each in a separate vessel. The ratio of hydrocarbon to O₂ and the shape of the vessel affect the yield.

D. K. MOORE.

Manufacture of hydrocarbons not saturated with hydrogen and hydrogen from gas mixtures containing hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 364,106, 20.8.30).—

Mixtures of gaseous paraffin hydrocarbons are subjected to thermal decomp., in the presence of steam and catalysts if desired, in stages at successively higher temp., *e.g.*, starting at 750° and rising to 1000°. The unsaturated and aromatic hydrocarbons produced may be condensed after each stage of the process by cooling the gases to -50°.

A. B. MANNING.

Production of hydrogen from hydrocarbon gases. STANDARD-I.G. Co., Assees. of G. H. FREYERMUTH, J. K. SMALL, and W. V. HANKS (B.P. 364,419, 7.3.31. U.S., 7.3.30).—The hydrocarbon gases, preferably freed from the greater part of their S impurities, *e.g.*, by washing with aq. NaOH, are mixed with steam and passed over a catalyst, *e.g.*, Ni or NiO, with or without the addition of Al₂O₃ or ThO₂ etc., at above 650°, for a prolonged period until there is evidence of a decrease in the activity of the catalyst, which is then reactivated by passing steam over it for 6–24 hr. Several reaction tubes may be used in parallel and the catalyst masses therein submitted to the reactivating process in rotation. The gases, now containing <3½% of hydrocarbons, are passed, with the addition of more steam if necessary, through a secondary reaction zone at a lower temp., wherein the CO is converted into CO₂ which is finally removed by washing the gases with H₂O under pressure.

A. B. MANNING.

Oxidation of hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 363,711, 19.9.30).—Crude petroleum or low-temp. tar fractions etc. are subjected to pressure hydrogenation in the presence of a suitable catalyst (metals of groups IV–VII, or their oxides, sulphides, etc.), preferably under conditions which remove the S compounds without markedly affecting the unsaturated hydrocarbons. The product is then converted into org. oxidation products, *e.g.*, alcohols, carboxylic acids, etc., preferably by blowing with air at about 150° in the presence of Mn stearate or similar catalyst.

A. B. MANNING.

Catalytic treatment of hydrocarbons at high temperatures. BRIT. & DOMINIONS FERALLOY, LTD., and J. W. BAMPFYLDE (B.P. 364,043, 17.7.30, 20.3. and 1.4.31).—Hydrocarbons are cracked in contact with surfaces consisting of Fe–Al alloy containing 9–30% (preferably 10–16%) Al, or this alloy with part of the Fe and/or Al replaced by 6% Cr; the Fe–Al alloy may be used as an electric resistance for internally heating the reaction chamber.

D. K. MOORE.

Conversion of methane into liquid hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 364,144, 22.9.30).—CH₄ or a gaseous mixture containing it is brought into contact with a catalyst consisting of or coated with Cr or graphite at 800–1200° to yield liquid hydrocarbons, *e.g.*, C₆H₆. The catalyst may be used as an element for internal electrical heating. [Stat. ref.]

D. K. MOORE.

Recovery of gasoline from [hydrocarbon] gas. F. A. HOWARD and N. E. LOOMIS, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,806,571, 19.5.31. Appl., 20.2.26).—Gas containing gasoline vapour, *e.g.*, natural gas, is scrubbed with a hydrocarbon oil and this oil stripped of its volatiles by passing through a number of

rectification towers in series so that the effluent from one becomes the feed to the next, the completely stripped oil being recirculated. Each tower in the series operates under a pressure less than the preceding one. The vapours from each tower are segregations of the desired gasoline components.

D. K. MOORE.

Production of low-boiling hydrocarbon oils from heavier, higher-boiling hydrocarbon oils by the action of hydrogen at elevated temperatures. STANDARD-I.G. Co., Assees. of R. P. RUSSELL (B.P. 364,403, 23.2.31. U.S., 24.3.30).—A hot mixture of H₂ and a heavy hydrocarbon oil is passed into a reaction chamber containing a catalyst immune from S poisoning, *e.g.*, oxide or sulphide of Cr, W, Mo, maintained at about 445° and under pressure of > 20 atm. (preferably > 100 atm.). The liquid and vapour pass into a separator the vapours from which are condensed (A) and the gas is purified and recirculated. The liquid is discharged into a flash tower from which the heavy oil which does not vaporise is withdrawn; the condenser vapours collected in a pan underneath the reflux plates in this tower are recirculated, whilst the vapours leaving the tower are condensed and the condensate is mixed with A to form a motor fuel.

D. K. MOORE.

Destructive hydrogenation of hydrocarbon oils. STANDARD-I.G. Co., Assees. of R. T. HASLAM (B.P. 364,401, 20.2.31. U.S., 13.3.30).—In the production of petrol with anti-knock properties, heavy oil, *e.g.*, kerosene, gas oil, with or without admixture of H₂, is heated to below 480°, *e.g.*, 455°, and led into a well-lagged, catalytic hydrogenation chamber in which the heat of reaction raises the temp. to above 480°, *e.g.*, 530°. The process is operated at > 20 atm.

D. K. MOORE.

Purification of petroleum and petroleum distillates. ANGLO-PERSIAN OIL Co., LTD., and A. E. DUNSTAN (B.P. 364,204, 10.10.30).—The sulphides and mercaptans remaining in petroleum oils after treatment with "doctor solution" are removed by treatment with Cl₂ or Cl compounds, *e.g.*, NaOCl.

D. K. MOORE.

Recovery of sulphur dioxide from products obtained in the treatment of mineral oils and tars or their distillates. BOLGAR OIL PROCESSES, LTD., P. A. MACKAY, and W. R. ORMANDY (B.P. 364,103, 16.7.30 and 15.5.31. Cf. B.P. 319,673; B., 1929, 1041).—By keeping the temp. below 180°, 80% of the S in the acid sludge from petroleum oil purification is driven off as a mixture of SO₂, CO, CO₂, hydrocarbon vapours, and steam, but without the formation of H₂S. After cooling and drying, the gases are compressed and led to a reservoir in which a layer of liquid hydrocarbons forms on a layer of liquid SO₂. The former is separated and the latter used for the manufacture of H₂SO₄.

D. K. MOORE.

Dewaxing of mineral oils. L. EDELEANU, Assr. to EDELEANU GES.M.B.H. (U.S.P. 1,809,777, 9.6.31. Appl., 11.1.30).—Mineral oils are saturated with liquid SO₂ (whereby the solubility of wax in the oil is reduced), cooled to below -15° by vaporisation of some of the liquid SO₂, and filtered from deposited wax.

D. K. MOORE.

Dewaxing oil. L. D. JONES, ASST. to SHARPLES SPECIALTY Co. (U.S.P. 1,806,570, 19.5.31. Appl., 25.5.28).—The wax pptd. by chilling diluted petroleum oil is removed by filtration through a medium of loosely-matted wool, cotton, steel, ramie, or asbestos fibres.

D. K. MOORE.

Production of lubricating oils. D. A. HOWES, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 363,846, 5.1.31).—In the polymerisation of olefines, *e.g.*, C_2H_4 , to form lubricating oils at 15–20°/10–50 atm., the yield is increased by using as catalyst $AlCl_3$ mixed with Al or Zn, preferably in finely-divided form. D. K. MOORE.

Manufacture of lubricating oils. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 363,823, 17.12.30).—Hydrocarbon oils containing paraffin wax, *e.g.*, brown-coal-tar oils, petroleum residues, are cracked at 450–600° with metals of group VI or their compounds, or Cu, as catalyst, or are dehydrogenated by first forming derivatives with substances capable of being split off as their H compounds, *e.g.*, Cl, and then heating with Al_2O_3 , $ZnCl_2$, etc. as catalyst. Compounds with < 14.5 pts. H per 100 pts. C. are removed from the products by suitable solvents and the residue is polymerised or condensed with hydrocarbons liquid between 50° and 300°, *e.g.*, C_6H_6 , anthracene oil, paraffins, at 25–150° in the presence of a condensing agent, *e.g.*, $AlCl_3$, BF_3 .

D. K. MOORE.

Manufacture of high-quality lubricating oils. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 364,110, 22.9.30).—Lubricating oils of low viscosity, either rich in H, *e.g.*, spindle oil, or hydrogenated at 390–430°/50–300 atm. with a compound of a metal of group VI as catalyst, or such oil split up into a fraction rich in H and one poor in H (this being hydrogenated) by means of differentiating solvents, *e.g.*, PhOH or liquid SO_2 , are cracked at temp. below 600°, preferably above 480°, with compounds of metals of group VI, with or without active C or Cu as catalyst; the unsaturated hydrocarbons formed are then polymerised and/or condensed, with or without the addition of liquid hydrocarbons, *e.g.*, mineral oil, $C_{10}H_8$, in the presence of $AlCl_3$, $ZnCl_2$, with or without the addition of bleaching earth or SiO_2 at temp. usually up to 150° but sometimes higher.

D. K. MOORE.

Treatment of lubricating oil in internal-combustion engines. ROHÖL-VERGASER-GES.M.B.H., and K. F. NÄGELE (B.P. 363,852, 8.1.31).—The volatiles which pass from behind the piston of internal-combustion engines, operated by heavy oil, into the lubricating oil are removed from the latter by heating it with the exhaust gas and drawing part of this through the oil, the temp. of which is thermostatically controlled.

D. K. MOORE.

Suction control apparatus for chamber ovens or retorts. C. STILL (B.P. 365,934, 18.5.31. Ger., 19.5.30).

Pulverisers for coal. Treatment of solids under pressure.—See I. Wetting etc. agents.—See III. NH_4 salts. S from ores etc.—See VII. Tarred macadam. Road-surfacing material.—See IX. Wax-like substances.—See XII. Gas from sewage.—See XXIII.

III.—ORGANIC INTERMEDIATES.

Determination of traces of carbon monoxide in ethylene. H. S. BOOTH and M. B. CAMPBELL (Ind. Eng. Chem. [Anal.], 1932, 4, 131–134).—The pyrotannic blood method (cf. A., 1926, 100) is recommended and a recalibration of the "pyrotannic detector" colour standard is given. The sensitivity may be increased 10–40 times by concentrating the CO by fractional distillation of the liquid mixture under reduced pressure.

E. S. HEDGES.

Methanol condensation as modified by alkalis catalysts. G. T. MORGAN, D. V. N. HARDY, and R. A. PROCTER (J.S.C.I., 1932, 51, 1–7 T).—Metallic oxides, prepared by ignition of the nitrates, are more efficient for the condensation of CO and H_2 at high pressures than when pptd. by aq. KOH, owing to adsorbed KOH in the latter case. Addition of alkali hydroxides to an equimol. mixture of Cr_2O_3 and MnO decreases the yield of MeOH obtainable, but does not lead to gaseous by-products. LiOH, NaOH, and KOH, but not RbOH or CsOH, decrease the yield of liquid product, RbOH and CsOH being thus the best catalysts for production of higher alcohols etc. A Cr_2O_3 -MnO catalyst containing 15% of Rb yielded: MeOH 42%; other alcohols 38%; aldehydes, acetals, and ketones (?) 15%; acids < 1%; CH_4 2%; CO_2 2%. The following substances were identified in the product: MeOH, EtOH (trace), PrⁿOH, BuⁿOH, β -methylbutanol, β -methylpentanol, $\beta\delta$ -dimethylpentanol, CH_2O , EtCHO, hydroxyaldehydes, and unsaturated aldehydes. The catalyst which normally produces EtOH gives also MeOH, PrⁿOH, BuⁿOH, BuⁿOH, *n*-amyl alcohol, β -methylbutanol, and β -methylpentanol. Theories of the formation of higher alcohols are discussed, the aldol theory being considered to be favoured by the above products. The hydroxy-aldehydes have the composition $C_6H_4(OH)\cdot CHO$, but are not hydroxybenzaldehydes. The following substances were synthesised: β -methylpentanol (from PrⁿOH and NaOPr at 220–240°), b.p. 148° (2:4:6-trinitrobenzoate, m.p. 126–127°; 1-hydrogen 3-nitrophthalate, m.p. 141°; 3:5-dinitrobenzoate, m.p. 49°; hydrogen tetrachlorophthalate, m.p. 103°; *p*-xenylcarbamate, m.p. 98–98.5°); δ -methylpentanol (from $MgBu^{\delta}Br$ and ethylene oxide), b.p. 148° (2:4:6-trinitrobenzoate, m.p. 118–118.5°; 1-hydrogen 3-nitrophthalate, m.p. 138.5–140°; 3:5-dinitrobenzoate, m.p. 69°); β -methylhexanol (from Et β -methylhexoate), b.p. 164–165° (*p*-xenylcarbamate, m.p. 88–88.5°; 1-hydrogen 3-nitrophthalate, m.p. 131–132°); β -ethylpentanol (from Et β -ethylvalerate), b.p. 164–166° (*p*-xenylcarbamate, m.p. 77–77.5°; 1-hydrogen 3-nitrophthalate, m.p. 127–128°); $\beta\delta$ -dimethylpentanol (from Et $\beta\delta$ -dimethylvalerate), b.p. 158–159° (*p*-xenylcarbamate, m.p. 74–75°; 1-hydrogen 3-nitrophthalate, m.p. 149°); β -ethylhexanol (from BuⁿOH and BuⁿONa at 220–240°), b.p. 180° (*p*-xenylcarbamate, m.p. 80°; 1-hydrogen 3-nitrophthalate, m.p. 107–108°). R. S. CAHN.

Determination of isopropyl alcohol in ethyl alcohol. F. M. ARCHIBALD and C. M. BEAMER (Ind. Eng. Chem. [Anal.], 1932, 4, 18–20).—The sample is agitated with a standard solution of NaOH at a definite temp., the supernatant alcohol is separated, and its alkalinity determined by titration. The result is compared with an empirical curve, which shows %

Pr^oOH plotted against alkalinity. Advantage is taken of the difference in solubility of NaOH in the two alcohols. The error is about 0.25% when <20% of Pr^oOH is present. More than 50% of Pr^oOH cannot be determined except by first diluting with a known amount of EtOH.

E. S. HEDGES.

Industrial analysis of glacial acetic acid. E. CHARLES (Ann. Chim. Analyt., 1932, [ii], 14, 5—13).—HCO₂H in glacial AcOH is determined by measuring the CO liberated on addition of Ac₂O or by titration with standard Br-water. H₂O is determined by boiling 100 g. of sample with a known amount of Ac₂O in small excess and a trace of conc. H₂SO₄ as catalyst, subtracting from that amount the Ac₂O remaining unchanged as found from the new m.p. and the Ac₂O equiv. to the HCO₂H present. The Ac₂O equiv. of the H₂O present is thus obtained. From the difference between the m.p. of the sample and that calc. for AcOH containing the amounts of H₂O and HCO₂H found, the content of higher homologues of AcOH may be calc.

W. J. BOYD.

An ether explosion. L. RAMBERG (Svensk Kem. Tidskr., 1931, 43, 305—309).—An account is given of a violent explosion which occurred during an extraction with Et₂O in a Soxhlet apparatus, due to the presence of peroxides in the Et₂O. The necessity for freeing Et₂O from these compounds is emphasised, and their nature and the methods for their detection and removal are discussed. MeCHO appears to act as a catalyst in promoting the formation of these compounds, and as this substance is produced when Et₂O is treated with FeSO₄ to remove peroxides an explanation is afforded of the fact that Et₂O which has been so treated is invariably found to contain peroxides again after a short interval.

H. F. HARWOOD.

Alkylamines as solvents. F. W. BERGSTROM, W. M. GILKEY, and P. E. LUNG (Ind. Eng. Chem., 1932, 24, 57—62).—Approx. determinations of the solubility at 20—30° of a large variety of compounds, mainly org., in NHEt₃, NEt₃, NHPr^o₂, NH₂Bu^o, NHBu^o₂, NBu^o₃, isoamylamine, and CH₂Ph-NH₂.

H. A. PIGGOTT.

Genesis of coals. Utilisation of coal. Solvents from the gas industry. Determining C₂H₄ etc. in gas mixtures.—See II. MeOH from alcoholic liquors. Prep. of EtOH. Determining fusel oil and aldehydes. Detection of BzOH etc. in wine.—See XVIII. Applications of lactic acid.—See XIX.

See also A., Feb., 129, Blanc's Al₂O₃ [for conversion of EtOH into Et₂O]. Catalytic oxidation of EtOH. Vapour-phase esterification. 141, Thermal polymerisation of C₂H₂. 142, Prep. of chloropicrin. 145, Prep. of acraldehyde and keten. 150, Purification of HCO-NH₂. 152, Hydrogenative cracking of polynuclear hydrocarbons. 156, Cracking of phenols with H₂. Prep. of 4-nitropyrocatechol. 159, Condensation of aldehydes with hydrazones. 182, Determination of phenanthrene in commercial anthracene.

PATENTS.

Manufacture of organic compounds containing fluorine. FRIGIDAIRE CORP., Assees. of T. MIDGLEY, JUN., A. L. HENNE, and R. R. McNARY (B.P. 359,997,

24.6.30. U.S., 5.4.30).—Cl, Br, or I in polyhalogeno-compounds, especially CCl₄, CBr₄, CHCl₃, CHI₃, or CHCl:CHCl, is partly or wholly replaced by F by the action, e.g., of SbF₃, the products being distilled off through a fractionating column as formed. CF₂Cl₂ is obtained in 90% yield from CCl₄ and SbF₃ in presence of SbCl₃ as catalyst. Suitable plant is described. The products are suitable as refrigerants, fire-extinguishing agents, anæsthetics, etc.

C. HOLLINS.

Preparation of absolute alcohol and commercially pure powdered calcium hydroxide. R. W. JAMES. From MERCK & Co., INC. (B.P. 364,111, 22.9.30).—Abs. EtOH and Ca(OH)₂ are simultaneously produced by heating and refluxing with agitation aq. EtOH with granular CaO in an excess of 5—20% over the amount required for dehydration. After the EtOH is distilled off, sufficient H₂O is added and the heating and agitation are continued until the original excess of CaO is completely hydrated. The agitation is of sufficient power to keep the contents in motion even when no liquid is present.

C. RANKEN.

Manufacture of alcohol and ether [from ethylene]. DISTILLERS' CO., LTD., W. P. JOSHUA, and H. M. STANLEY (B.P. 360,492, 9.8.30).—C₂H₄ is absorbed in 50—80% H₂SO₄ (preferably 60—75%) at 90°/50—100 atm. for EtOH, or above 100°/50 atm. for Et₂O and EtOH.

C. HOLLINS.

Manufacture of chloroethanesulphonic acid and the salts and chloride thereof. I. G. FARBENIND. A.-G. (B.P. 360,558, 5.9.30. Ger., 5.9.29).—Ethylene dichloride reacts with Na₂SO₃, K₂SO₃, ZnSO₃, etc. in dil. EtOH, MeOH, etc. to give salts of chloroethanesulphonic acid (only one Cl being replaced), from which the acid chloride may be obtained.

C. HOLLINS.

Manufacture of acrylic acid esters. ROHM & HAAS A.-G. (B.P. 361,153, 15.12.30. Ger., 20.2.30).—β-Hydroxypropionic esters or their alkyl ethers are treated, e.g., at 150—200°, with dehydrating agents (H₂SO₄ on SiO₂ gel, or H₃PO₄), with or without addition of sulphates, phosphates, borates, or metals such as Fe or Cu. Stabilisers (quinol, naphthols, S, Cu) may be added to prevent polymerisation of the product.

C. HOLLINS.

Manufacture of nitro-β-hydroxy-pyridine and -alkylpyridines. I. G. FARBENIND. A.-G. (B.P. 360,188, 3.11.30. Ger., 7.11.29).—3-Hydroxypyridines or homologues are nitrated in H₂SO₄ below 110°. Mononitro-3-hydroxy-derivatives of pyridine (m.p. 68—69°; amine, m.p. 163°), α-picoline (m.p. 107—108°; amine, m.p. 153°), γ-picoline (m.p. 88—89°), and 2-methyl-5-ethylpyridine (m.p. 162—165°) are described.

C. HOLLINS.

Manufacture of salts of a quinolinesulphonic acid readily soluble in water. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 360,501, 12.8.30).—The Li salts of 7-iodo-8-hydroxyquinoline-5-sulphonic acid are sol. in H₂O to the extent of 15%; the Li₂ salt has an alkaline reaction.

C. HOLLINS.

Production of molecular compounds of the alkali-metal, alkaline-earth-metal, and magnesium salts of 2-phenylquinoline-4-carboxylic

acid with 1-phenyl-2:3-dialkyl-5-pyrazolones or with 1-phenyl-2:3-dialkyl-4-dialkylamino-5-pyrazolones. R. and O. ADLER (B.P. 360,368, 4.7.30, Czechoslov., 14.11.29).—The ingredients in stoichiometric proportions are heated together with a little H₂O until a clear melt is obtained. C. HOLLINS.

Manufacture of heterocyclic compounds. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 360,027, 31.7.30).—A hydroaromatic compound containing suitable *o*-substituents (*e.g.*, an *o*-halogenoketone) is condensed with an aminopyridine or other heterocyclic NH₂ compound having a free position *ortho* to the NH₂, whereby hydrogenated pyridinoindoles or analogous compounds are produced. 2-Chlorocyclohexanone and sodio-2-aminopyridine in PhMe give an intermediate, m.p. 147°, which is converted by Ac₂O into 6:7:8:9-tetrahydro- $\alpha\beta$ -pyridinoindole, CH₂·CH₂·C—C·CH:CH

$\begin{array}{c} | \\ \text{CH}_2 \cdot \text{CH}_2 \cdot \text{C} \cdot \text{NH} \cdot \text{C} \cdot \text{N} \cdot \text{CH} \\ | \quad | \quad | \quad | \\ \text{CH}_2 \cdot \text{CH}_2 \cdot \text{C} \cdot \text{NH} \cdot \text{C} \cdot \text{N} \cdot \text{CH} \end{array}$, m.p. 95°, reduced by Na and EtOH to the dodecahydro-compound, m.p. 58–59°, b.p. 160–163° (methiodide, m.p. 248°); the 7-Me (m.p. 94°, b.p. 150–154°/3 mm.; methiodide, m.p. 270°), 7-CH₂Ph (b.p. 204–212°/1 mm.), 9-OMe (b.p. 140°/3 mm.), 6-Me-8-Pr⁺ (b.p. 155–160°/1 mm.), 2-NO₂ (m.p. 210°; amine, m.p. 195°), 2-I (m.p. 150°, b.p. 187–190°/3 mm.), and 3-Me (m.p. 56°, b.p. 156–160°/2 mm.; methiodide, m.p. 302°) derivatives are obtained by using 2-chloro-4-methyl- (b.p. 65–70°/3 mm.), 2-chloro-4-benzyl- (b.p. 150–155°/1 mm.), 2-chloro-6-methoxy-, and 2-chloro-3-methyl-5-*isopropyl*- (b.p. 95–100°/3 mm.)-cyclohexanones, and 5-nitro-, 5-iodo-, and 6-methyl-2-aminopyridines, respectively. 2-Aminopyridine and 1:2-dibromocyclohexane give 6:7:8:9:10:11-hexahydro- $\alpha\beta$ -pyridinoindole, m.p. 94°, b.p. 122–125°/3 mm.; the 5-Me derivative, from 2-methylaminopyridine and 2-chlorocyclohexanol, has b.p. 140–145°/3 mm. Analogous compounds are formed from 2-aminopyridine and 2-bromocyclopentanone (b.p. 145°/2.5 mm.) and 2-bromo-1-keto-1:2:3:4-tetrahydronaphthalene (m.p. 157°, b.p. 200–210°/1 mm.). 4-Aminopyridine and 2-chlorocyclohexanone give 6:7:8:9-tetrahydro- $\beta\gamma$ -pyridinoindole. 2-Chlorocyclohexanone is also condensed with 2-amino-6-methoxyquinoline (m.p. 153°; B,HCl, m.p. 265°), 1-aminoisoquinoline (b.p. 200–210°/3 mm.), 2-amino-5:6-dimethylpyridine (giving CH₂·CH₂·C—N·CMe:CMe

$\begin{array}{c} | \\ \text{CH}_2 \cdot \text{CH}_2 \cdot \text{C} \cdot \text{N} \cdot \text{C} \cdot \text{N} = \text{CH} \\ | \quad | \quad | \quad | \\ \text{CH}_2 \cdot \text{CH}_2 \cdot \text{C} \cdot \text{N} \cdot \text{C} \cdot \text{N} = \text{CH} \end{array}$, m.p. 186°, b.p. 190–200° (m.m.), and 5-amino-3-methyl-1:2:4-triazole (giving CH₂·CH₂·C—N—CMe

$\begin{array}{c} | \\ \text{CH}_2 \cdot \text{CH}_2 \cdot \text{C} \cdot \text{N} \cdot \text{C} \cdot \text{NH} \cdot \text{N} \\ | \quad | \quad | \quad | \\ \text{CH}_2 \cdot \text{CH}_2 \cdot \text{C} \cdot \text{N} \cdot \text{C} \cdot \text{NH} \cdot \text{N} \end{array}$, m.p. 229°, b.p. 180°/2 mm.).

C. HOLLINS.

Separation of alcohols and phenols from mixtures. A. A. KAUFMANN (B.P. 259,953, 24.7.30. Ger., 7.8.29).—Alcohols and/or phenols in mixtures are converted into borates by distillation (*e.g.*, in vac.) with boric esters, especially lower alkyl (Et and Bu) borates. The alcohols and/or phenols are regenerated from their borates, after recrystallisation if desired; camphor and camphor-free linalool are isolated from Shui oil; citronellal and terpene alcohols from Java citronella oil;

and *l*-borneol from fusel oil obtained from coniferous wood. C. HOLLINS.

Purification of dry-cleaners' solvent. AKTIEB. SEPARATOR, ASSEES. of R. G. WALKER (B.P. 361,690, 12.1.31. U.S., 13.1.30).—Used solvent from the washer is brought into contact with aq. alkali and/or charcoal in a main vessel from which it overflows into a centrifuge (with or without previous injection of H₂O) and thence returns to the washer. A portion of the solvent from the top of the main vessel or from the washer is bypassed to a still to remove high-boiling fractions, the low-boiling solvent being returned to the main vessel.

C. HOLLINS.

Manufacture of water-soluble, non-dyeing [tin] derivatives of sulphurised phenols [wool reserve salts]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 360,378, 2.8.30).—The non-dyeing sulphurised phenol derivatives of B.P. 173,313 (B., 1922, 139 A) are heated with a H₂O-sol. inorg. Sn compound in H₂O or aq. alkali. The product may be air-oxidised during isolation. C. HOLLINS.

Manufacture of wetting and emulsifying agents. I. G. FARBENIND. A.-G. (B.P. 361,732, 10.2.31. Ger., 10.2.30).—Ricinonic acid (dehydrated ricinoleic acid) or its glyceride or amide is treated with an aq. sulphite (NaHSO₃) at 80–100°. The products are stable to boiling dil. mineral acid. C. HOLLINS.

Manufacture of derivatives of [long-chain] ethers [wetting, emulsifying, cleansing, tanning, and softening agents]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 360,493, 7.7.30. Cf. B.P. 359,001; B., 1932, 173).—Ethers, obtained from aliphatic halides above C₉ (which may contain OH, CO, or CO₂H groups) and aliphatic polyhydric or alicyclic mono- or poly-hydric alcohols, or phenols, naphthols, or hydroxyethers, are treated with H₃PO₄, P₂O₅, or phosphoric halides or with sulphonating agents. Examples are: chlorinated petroleum fractions etherified with Na ethylene glycol and esterified with ClSO₃H at 40–50°; middle oil from brown-coal hydrogenation, chlorinated, etherified with NaOPh, C₁₀H₇·ONa or Na resorcinol, and sulphonated with oleum at 100–120°.

C. HOLLINS.

Manufacture of wetting, cleansing, dispersing, and like agents. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 360,539, 29.8.30).—Inorg. esters of non-carboxylated aliphatic alcohols above C₇ (preferably C₁₀–C₁₈) are heated with sulphites. Examples are: dodecanol, sulphated with ClSO₃H in Et₂O and the Na salt heated at 200° with cryst. NaHSO₃; Na octodecyl sulphate with aq. Na₂SO₃ at 250° or with aq.-alcoholic Na₂SO₃ at 200°; alcohols, b.p. 100–210°/18 mm., from reduction of coconut oil, sulphated with ClSO₃H and the ester heated with NaHSO₃ and NaOH at 200°; octodecyl bromide with aq. Na₂SO₃ at 130–160°; sulphated octodecylene glycol (from castor oil), neutralised with NaOH and Na₃PO₄, heated with aq. NaHSO₃ at 180°. C. HOLLINS.

[Manufacture of] wetting, penetrating, foaming, and dispersing agents. H. T. BÖHME A.-G. (B.P. 360,602, 8.10.30. Ger., 4.11.29).—Unsaturated aliphatic

hydrocarbons (C_{10} — C_{18}) or cracked petroleum fractions are converted by H_2SO_4 below 0° , in presence of a diluent if desired, e.g., C_2HCl_3 , C_6H_6 , or BuOH, into sulphuric esters of corresponding alcohols.

C. HOLLINS.

Chlorination of diphenyl. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 360,840, 10.6.30).—Resinous products are obtained by (hexa)chlorination of Ph_2 in presence of a carrier ($SbCl_3$) with cooling to the lowest temp. at which the mixture is stirrable (initially 70° , then 10° , finally 35°).

C. HOLLINS.

Manufacture of 3-hydroxythionaphthen- [thioindoxyl-] 7-carboxylic acid chlorides and carboxylic acids. I. G. FARBENIND. A.-G. (B.P. 360,349, 11.5.31. Ger., 10.5.30).—The dichlorides of 2-carboxy-arythioglycolic acids having the 6-position free are cyclised by $AlCl_3$ to thioindoxyl-7-carboxyl chlorides, which may be hydrolysed to the acid and oxidised by air to thioindigo-7:7-dicarboxylic acids. Products from 2-carboxyphenylthioglycolic acid and its 5-Cl-derivative are described.

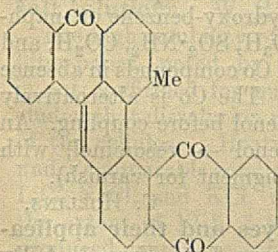
C. HOLLINS.

Manufacture of 4:5-dimethyl-7-chloro-oxythionaphthen [7-chloro-4:5-dimethylthioindoxyl] and of indigoid dyes derived therefrom. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 360,533, 22.8.30).—*o*-4-Xylidine-5-sulphonic acid is converted by way of the diazo compound into 4-chloro-*o*-xylene-5-sulphonic acid, the chloride of which is reduced to the 5-thiol compound, also obtainable from 5-chloro-*o*-4-xylidine by the xanthate method. The 4-chloro-*o*-5-xylidylthioglycolic acid, m.p. 70° , gives a thioindoxyl, m.p. 160° , which is oxidised to 7:7'-dichloro-4:4':5:5'-tetramethylthioindigo (red-violet), or is condensed with suitable components, e.g., 6-chloro-4-methylthioisatin α -anil (pink), 5:7-dibromoisatin α -chloride (violet), or 5-chloro-7-methoxy-4-methylisatin α -chloride (blue). The same dyes may be obtained from 7-chloro-4:5-dimethylthioisatin.

C. HOLLINS.

Manufacture of a condensation product of the anthraquinone series.

A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 360,102, 1.9.30).—2:2'-Dimethyl-1:1'-dianthraquinonyl is converted by KOH in a diluent (*cyclohexanol*, methyl*cyclohexanol*, or EtOH) at 75 – 80° into a yellow vat dye (annexed formula), together with a



little pyranthrone.

C. HOLLINS.

Manufacture of compounds of the dibenzopyrene-quinone series. I. G. FARBENIND. A.-G. (B.P. 360,215, 24.11.30. Ger., 22.11.29).—1:2:6:7-Dibenzopyrene-3:8- and 1:2:7:8-dibenzopyrene-3:6-quinones are converted by way of the disulphonic acids into $(OH)_2$ -derivatives, which may be halogenated and/or etherified (e.g., alkylated). The dihydroxy-1:2:6:7-compound gives by methylation a scarlet vat dye, and by bromination and methylation a violet-red. Methylation of the dihydroxy-1:2:7:8-compound gives a violet.

C. HOLLINS.

Conversion of CH_4 into liquid hydrocarbons. Thermal decomp. of hydrocarbons.—See II. **Halogenated vat-dye intermediates.**—See IV. **Alkaline-earth formates.** Alkali acetates.—See VII.

IV.—DYESTUFFS.

Substantivity of benzidine dyes: theory of direct cotton dyeing. W. SCHRAMEK and E. GÖTTE (Kolloid-Beih., 1931, 34, 218–269).—Chrysamine G (I), Congo Red (II), Bordeaux COV (III), Oxamine Violet B (IV), Diamine Blue BB (V), and Dianil Blue R (VI) are prepared from carefully purified intermediates and completely freed from electrolytes by dialysis through a collodion membrane. In the purification of (IV) and (V), which are prepared in alkaline solution, the presence of the isomerides normally obtained by acid coupling was observed. The pure dyes were characterised by conductivity measurements which indicated removal of some of the cation during dialysis; the dye salts have the normal dissociation of a binary salt, but the pure free acids are practically undissociated. In the pure state (I) and (IV) are strongly adsorbed by cotton, (II) and (III) only slightly, and (V) and (VI) not at all. Measurements of particle size by Fürth's method indicate that no connexion exists between substantivity and particle size. Addition of small amounts of electrolytes to (VI) in solution leads at first to homogenisation and reduction of particle size, often to dissolution: with Na_2SO_4 or NaCl this reaches a max. at 0.05 – $0.1N$, after which particle size increases until coagulation occurs; with $MgSO_4$ the initial stage is reached at $0.002N$. Affinity is also conferred on the dye by these electrolytes in concns. of 1×10^{-7} to 1×10^{-1} equiv. The effect of electrolytes on (III) is simply one of coagulation, nevertheless affinity is increased as with (VI). The free acid of (VI), which is homodisperse, has the same dyeing properties as its salts, and its affinity is likewise increased by small concns. of acid. It is concluded that the substantivity of the technical dyes is the result of the inorg. salts they contain, which as electrolytes can confer affinity on a "substantive" dye which in itself is without direct affinity. The affinity of the pure dye is correlated with constitution. H. A. PIGGOTT.

Direct electrometric titration of certain coal-tar food dyes. O. L. EVENSON and R. H. NAGEL (Ind. Eng. Chem. [Anal.], 1932, 4, 151–154).—A method is developed for the electrometric titration (with $TiCl_3$) of such dyes belonging to the azo, $CHPh_3$, NO_2 , and pyrazolone groups. A buffer salt (Na H tartrate or Na citrate) is used. The reducing action of Na H tartrate causes low results to be obtained with some dyes.

E. S. HEDGES.

Solubility in [hydrocarbon] oils of some indigoid dyes. H. LEDERER (Petroleum, 1932, 28, 5–6).—Dyes such as Indigo MLB 4B (tetrabromoindigo) and Thioindigo Red are soluble in hydrocarbon oils and may be used for giving a stable colour to such oils.

H. E. BLAYDEN.

See also A., Feb., 159, **Condensation of aldehydes with hydrazones.** 164, **Syntheses of purpurin and chryszin.** 170, **Addition of diazomethane and its derivatives to α -naphthaquinone.** 203, **Dye of red cabbage.**

PATENTS.

Manufacture of esters of leuco-compounds of vat dyes [indigosols]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 360,907 and 360,958, [A] 13.6., 11.8., 28.8., and 29.8.30, [B] 12.8. and 25.9.30 and 22.4.31).—(A) Vat dyes are treated with H_2S , preferably in presence of a *tert.* base (pyridine) at low temp., and, if desired, with addition of dry NH_3 or an amine capable of forming a salt with the H_2S , and the product, without isolation, is esterified with pyridine- SO_3 (etc.). The treatment with H_2S may take place in presence of the sulphating agent. (B) The process is applicable to nitro-vat dyes, the NO_2 group being retained. Examples of starting materials are: (A) indigo, 5:5'-dibromoindigo, indigoid dyes from β -naphthisatin chloride and α -anthrol and from chlorobromo- β - β -naphthisatin chloride and 4-methoxy- α -naphthol, thioindigo, 5:7-dibromo-2-indole-2'-thionaphthenindigo, tetrabromoindigo, 5:5'-dichloro-7:7'-dimethylthioindigo; (B) indigoid dyes from 5-nitroisatin chloride or 6:9-dichloro-5-nitro- α -naphthisatin chloride and α - or β -naphthathioindoxyl.

C. HOLLINS.

Manufacture of [thioindigoid] vat dyes. I. G. FARBENIND. A.-G. (B.P. 360,583, 25.9.30. Ger., 25.9.29).—A 6-alkoxythioindoxyl is condensed with 5-chloro-, 5-methyl-, 5:6- or 5:7-dichloro-, 5-chloro-7-methyl-, 6- or 7-chloro-5-methyl-, or 5:7-dimethyl-isatin, to give clear orange vat dyes.

C. HOLLINS.

Production of [halogenated] vat dyes and intermediates. C. SHAW, R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 360,355, 24.4.30).—Halogenation is effected in molten phthalic anhydride, preferably above 200°. Starting materials exemplified are dibenzanthrone, isodibenzanthrone, Caledon Red BN, indanthrone, benzanthrone, pyranthrone, anthraquinone, indigo, and thioindigo.

C. HOLLINS.

Manufacture of vat dyes [of the dibenzanthrone series]. IMPERIAL CHEM. INDUSTRIES, LTD., C. SHAW, and R. F. THOMSON (B.P. 359,937, 28.7.30).—Grey to black vat dyes are obtained by treatment of benzanthrone, preferably at moderate temp., with CrO_3 , followed by alkaline fusion.

C. HOLLINS.

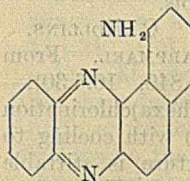
Manufacture of vat dyes [of the benzanthrone-pyrazolanthrone series]. I. G. FARBENIND. A.-G. (B.P. 360,776, 26.2.31. Ger., 26.2.30. Addn. to B.P. 298,284; B., 1928, 923).—Halogenated benzanthrone-pyrazolanthrones are oxidised and alkylated. The di- and tri-chlorodimethoxy-compounds are greenish-blue vat dyes.

C. HOLLINS.

Production of dyes [2-arylamino-1:4-naphthaquinones]. H. T. BUCHERER (B.P. 360,522, 19.8.30. Ger., 19.8.29).—Arylazo- α -naphthols are heated with arylamines in acid (AcOH). The reaction is: $HO \cdot C_{10}H_6 \cdot N_2 \cdot Ar + NH_2Ar' + H_2O = NH_2Ar + O:C_{10}H_6(NHAr) \cdot O$. Orange I, *e.g.*, is heated in heated in AcOH with NH_2Ph , *p*-aminodimethylaniline, *o*- and *p*-phenylenediamines, benzidine, or *p*-aminodimethylanilinesulphonic acid (giving a thiazinequinone). Alternatively, the azo dye may be heated first with AcOH (Orange I, *e.g.*, gives 2-*p*-sulphoanilino-1:4-naphthaquinone) and then with the arylamine to replace the 2-substituent.

C. HOLLINS.

Manufacture of monoazo dyes. I. G. FARBENIND. A.-G. (B.P. 360,798, 11.4.31. Ger., 11.4.30).—A 1-amino- α -naphthaphenazine (annexed formula) is diazotised and coupled with a suitable component in substance or on a substrate. Examples are: 1-amino- α -naphthaphenazine-4-sulphonic acid \rightarrow Schäffer acid (yellow-red on wool), acetyl-S-acid (bordeaux), Laurent acid (brick-red), resorcinol (yellow-brown), 1-*p*-sulphophenyl-3-methyl-



5-pyrazolone (yellow), β -naphthol (red Ba or Ca pigment), acetoacetanilide (yellow Ba or Ca pigment); the 4:10-disulphonic acid \rightarrow β -naphthol (yellow-red), 1-phenyl-3-methyl-5-pyrazolone (red-yellow), acetoacetanilide (yellow), 2:4-dihydroxyquinoline (yellow-red); the 4:6-disulphonic acid \rightarrow β -naphthol (brown-yellow); 10-chloro-1-amino- α -naphthaphenazine-4-sulphonic acid \rightarrow Schäffer acid (red); the 10- NO_2 compound \rightarrow Schäffer acid (red-brown); 1-amino- α -naphthaphenazine \rightarrow R-acid (bordeaux), acetoacet-*o*-toluidide (yellow pigment, insol. in linseed oil), β -naphthol (yellow-red pigment, sol. in stearin-ceresin), or on the fibre with Naphthol AS (red-brown).

C. HOLLINS.

Manufacture of azo dyes insoluble in water [ice colours and pigments]. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 361,097, 7.11.30).—2:3-Hydroxynaphthoic arylamides are coupled in substance or on the fibre with aminobenzotrifluorides. The presence of the CF_3 group improves the fastness to light. *m*-Aminobenzotrifluoride and its 6-OMe (m.p. 57–58°) and 6-Cl (b.p. 82–83°/9–10 mm.) derivatives are coupled with the *o*- and *p*-anisidides, 4- and 5-chloro-*o*-anisidides, 5-methoxy-*o*-toluidide, 4-chloro-2:5-dimethoxyanilide, anilide, *o*-toluidide, *o*-phenetidine, and 5-chloro-*o*-toluidide for orange to scarlet shades.

C. HOLLINS.

Manufacture of metalliferous azo dyes. SOC. CHEM. IND. IN BASLE (B.P. 360,356, 3.7.30. Switz., 3.7.29).—Dyes of the type: an *o*-aminophenol (except the 5-Cl-compound) \rightarrow a dihydroxy-benzene or -naphthalene, which are free from SO_3H , $SO_2 \cdot NH_2$, CO_2H , and pyrone groups, are treated with Co compounds in absence of caustic alkali or aq. NH_3 . The Co is alternatively introduced into the *o*-aminophenol before coupling. An example is 4-nitro-*o*-aminophenol \rightarrow resorcinol, with Co formate or $CoCl_2$ (scarlet pigment for varnish).

C. HOLLINS.

Manufacture of trisazo dyes and their application. IMPERIAL CHEM. INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & Co. (B.P. 360,124, 17.9.30).—Trisazo dyes of the type: arylaminesulphonic acid \rightarrow α -naphthylamine or Cleve acid \rightarrow α -naphthylamine or Cleve acid \rightarrow a *p*-aminobenzoyl or 4-*p*-aminobenzamidobenzoyl derivative of an aminonaphtholsulphonic acid, are prepared. Examples are: aniline-2:5-disulphonic acid \rightarrow Cleve acid \rightarrow α -naphthylamine \rightarrow *p*-aminobenzoyl-J-acid (blue on cotton; green by development with phenylmethylpyrazolone); metanilic acid \rightarrow Cleve acid \rightarrow Cleve acid \rightarrow *p*-aminobenzoyl-J-acid (blue; green with phenylmethylpyrazolone).

C. HOLLINS.

Indigoid dyes. Condensation product of the anthraquinone series. Dibenzopyrenequinone compounds.—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Pectic substances in retted flax fibre. E. PARISI and Z. TOMASSINI (Annali Chim. Appl., 1931, 21, 559—563).—Retting of flax has no selective action on the sugars which constitute the pectic substances, but an increase in the amount of pectic acid occurs. The hydrolysis of pectin of retted hemp produces only xylose, whilst from flax arabinose is also obtained. Maule's reaction of flax fibre is less intense than in hemp fibre. The SiO_2 content of the ash is lower in flax and the quality of the fibre is correlated to the CaO and SiO_2 contents.

O. F. LUBARTI.

Cellulose. II. Kaoliang cellulose. S. KOMATSU and M. SHIMADA (J. Soc. Chem. Ind., Japan, 1931, 34, 474—475 B).—Results of the analysis of cellulose pulps obtained from the culms and sheaths of Manchurian Kaoliang by the Cross and Bevan method, with and without pretreatment of the material with H_2O at 180° , indicate that during this pretreatment α -cellulose is converted into β -cellulose.

B. P. RIDGE.

Cellulose. I, III. Rice-straw cellulose. S. KOMATSU (J. Soc. Chem. Ind., Japan, 1931, 34, 474 B, 475—476 B).—I. [With Y. YAMANA.] The highest yield of cellulose (37%), with ash content 2.2%, furfuraldehyde yield 6.2%, and Cu no. 0.28, is obtained from rice straw by treatment with 3% H_2SO_4 ; superheated H_2O gives the lowest yield (32%) and 7% aq. NaOH an intermediate val. (35%), whilst the respective ash contents, furfuraldehyde yields, and Cu nos. are 7.1, 1.7%; 3.5, 7.0%; and 0.23, 0.14. In all cases the purification with the reagent stated was followed by treatment of the pulp with aq. Cl_2 .

III. [With Y. KINOSHITA.] The α -cellulose obtained from rice straw is similar in properties to that from wood and ramie.

B. P. RIDGE.

Insolubility of cellulose in concentrated neutral salt solutions. W. HÖNSCH (Textilber., 1932, 13, 84).—The high solubility of cellulose in the form of purified filter-paper, noted by von Weimarn (A., 1926, 576), is not confirmed for pure Egyptian cotton; it is concluded that dissolution must be preceded by degradation of the cellulose. Heating of Egyptian cotton samples under comparable conditions with conc. aq. CaCl_2 , KCNS , LiCl , NaCl , and KCl , whereby dissolution occurred to the extent of 12.5, 7.0, 5.0, 0, and 0%, respectively, produced degradation in the residual cotton as shown by increases in the Cu no. of 0.69, 0.58, 0.04, 0, and 0, respectively. Cotton may be completely separated from viscose silk by heating with aq. $\text{Ca}(\text{CNS})_2$ of such concn. that only the latter fibre is dissolved.

A. J. HALL.

Viscose. XXXVIII. Depolymerisation of cellulose during the ageing and xanthation of soda-cellulose. S. IWASAKI and E. SUGINO (J. Soc. Chem. Ind., Japan, 1931, 34, 498—500 B).—When soda-cellulose made from cotton paper is aged in the presence of air in a stoppered flask at 13° , a decrease of 20% in the viscosity of its solution in cuprammonium is shown after $5\frac{1}{2}$ hr., but only a slight decrease occurs on further prolonged treat-

ment. Its Cu no. shows a small increase, and its I val. no significant change. On xanthation with CS_2 , the viscosity shows only a small decrease, whilst the Cu no. and I val. increase slightly with increasing time of treatment.

B. P. RIDGE.

Swelling of cellulose acetate. I. SAKURADA and S. SUZUKI (J. Soc. Chem. Ind., Japan, 1931, 34, 490—493 B).—In the swelling of cellulose acetate it is considered that, besides the attraction between the dipoles of this substance and the liquid mols., the diffusion pressure of the liquid is very important; otherwise it is difficult to explain why so much liquid is sometimes absorbed by the cellulose acetate (e.g., 1 mol. of the latter takes up 43 mols. of CHCl_3 and 12 of EtOAc), whereas, when little swelling occurs, only a small amount of liquid is absorbed. Cellulose material acetylated for a long time and therefore depolymerised to a greater extent swells less and gives a much less viscous solution in CHCl_3 than that less drastically treated; hence high viscosity corresponds with great swelling. Since heat is liberated during swelling, rise of temp. should be accompanied by decreased swelling, whereas the reverse is often observed. This may be explained by the assumption that there are two stages of swelling: in the first, the attraction of the polar mols. is accompanied by liberation of heat, which hinders swelling; whilst in the second, where the effect of diffusion pressure is important, a rise of temp. assists the process. If this is so, liquids in which cellulose acetate does not swell much should show a decrease in the swelling with rise in temp., and this is actually found experimentally.

B. P. RIDGE.

Chemical and mechanical examination of paper pulp. Criticism of proposed analytical methods. Classification of paper for newspaper according to the breaking strength. M. ROTHKOPF (Anal. Assoc. Quím. Argentina, 1930, 18, 247—256).—Hälsé's formula for calculation of the % of mechanical pulp in a paper pulp yields accurate results only if the lignin contents of the mechanical and chemical pulps used are known for each sample of paper; the considerable influence of these factors is demonstrated. The lignin contents of a number of commercial pulps have been determined. Hälsé's method is recommended. The mean of the longitudinal and transverse breaking strengths of a paper is inversely proportional to the content of the mechanical pulp.

H. F. GILLBE.

Hydration measurement by the boiling method. T. R. LE COMPTE (Paper Trade J., 1931, 93, No. 14, 42—44).—Exposure of beaten stuff to boiling H_2O quantitatively depresses the wetness. An interpretation of Nakano's conception of the nature of wetness is offered.

CHEMICAL ABSTRACTS.

Production of paper from cornstalks. O. R. SWEENEY and L. K. ARNOLD (Iowa Eng. Exp. Sta. Bull., 1930, No. 100, 78 pp.).—The soda and sulphate processes were the most effective, a combination of H_2O digestion and cooking with NaOH giving good results. The optimal quantity of NaOH was 20% of the wt. of stalks; the yield of pulp was 40—50%. The sulphite process was unsatisfactory. Preliminary fermentation of the stalks reduced the NaOH consumption;

digestion with H_2O only or use of mechanical pulping processes produced inferior pulps. CHEMICAL ABSTRACTS.

Coalification of cellulose.—See II. Nitrocellulose.—See XXII.

See also A., Feb., 122, Solutions of cellulose [derivatives]. Swelling of pine-wood cellulose. 123, Prep. of artificial sponges of cellulose. 161, Pine-wood lignin. 184, Basic NH_2 -acids in wool.

PATENTS.

Extraction or treatment of vegetable fibres. E. V. HAYES-GRATZE (B.P. 363,651, 12.9.30).—After mechanically breaking up and removing the green skin or envelope from the leaves or stems, the latter are treated with a neutralised sulphonated oil, *e.g.*, castor oil diluted with 25 pts. of H_2O at 50–100°, the sulphonated oil having been previously ionised by electrolysis or by the action of a high-tension and/or high-frequency discharge, so that it has $p_H < 7$ when diluted with 10 vols. of H_2O ; a bleaching agent, *e.g.*, H_2O_2 , $NaOCl$, SO_2 , $NaHSO_3$, may also be added to the treating liquor. (Cf. B.P. 363,653; B., 1932, 270.) F. R. ENNOS.

Treatment of wool and other animal fibres. E. V. HAYES-GRATZE (B.P. 363,652, 12.9.30).—Wool is scoured by treatment at, *e.g.*, 43–60° with a 4–6.2 vol.-% aq. solution of a neutralised sulphonated oil which has been subjected to electrolysis or to a high-frequency discharge (cf. B.P. 363,653; B., 1932, 270) until its p_H val. after dilution with 10 vols. of H_2O is < 7 . Suitable bleaching agents, *e.g.*, H_2O_2 , O_3 (prepared *in situ* by a high-frequency discharge), SO_2 , etc. may also be present. D. J. NORMAN.

Cleansing of rags, fibres, cellulosic materials of a like kind and removal therefrom of rubber, gutta-percha, rosins, oils, fats, waxes, etc. J. KNAGGS, J. EASTMAN, and PORTALS, LTD. (B.P. 364,423, 10.3.31).—The material is extracted with a suitable volatile solvent, *e.g.*, C_6H_6 , solvent naphtha, $C_6H_6-CCl_4$, $C_6H_6-C_2HCl_3$, etc. Apparatus is described. D. J. NORMAN.

Manufacture of mixed esters of cellulose. KODAK, LTD., Assees. of C. S. WEBBER and C. J. STAUD (B.P. 360,417, 5.8.30. U.S., 2.8.29).—A cellulose ester is digested with an org. acid (except α -hydroxylated and α - and γ -keto-acids) in a solvent, *e.g.*, in dioxan at 100°; the free acid must have an ionisation const. greater than that of the acid corresponding with the ester, *e.g.*, above 1.82×10^{-5} . $COMe_2$ -sol. cellulose acetate is heated with hydrated oxalic acid, maleic acid, *m*-toluic acid (product, m.p. 215–232°), or salicylic acid (product, m.p. 248–271°). The oxalic and maleic acid products are H_2O -sol. C. HOLLINS.

Manufacture of cellulose derivatives [containing nitrogen]. SOC. CHEM. IND. IN BASLE (B.P. 360,742, 12.1.31. Switz., 11.1.30).— γ -Chlorohydroxypropyl ethers obtained from cellulose or its derivatives and epichlorohydrin are converted by *tert.* amines into nitrogenous cellulose compounds having affinity for acid and direct dyes. Suitable amines are pyridine and NMe_3 . C. HOLLINS.

Production of highly etherified cellulose. A. LIEBSTER (B.P. 364,463, 13.4.31. Ger., 16.5.30).—

Cellulose ethers containing 49–50% OEt are obtained in a single operation by the interaction, *e.g.*, in an autoclave at $> 150^\circ$ for about 10–15 hr., of cellulose, dry $NaOH$, an ester of an inorg. acid, *e.g.*, EtCl, and an inert org. liquid, *e.g.*, petroleum, in the absence of added H_2O . D. J. NORMAN.

Manufacture [purification or separation] of benzylcellulose. IMPERIAL CHEM. INDUSTRIES, LTD., A. S. LEVESLEY, and F. C. RANDALL (B.P. 364,142, 19.9.30).—Up to 10% of a H_2O -sol. wetting agent, *e.g.*, a sulphonated oil or an alkylated naphthalenesulphonic acid, is thoroughly incorporated with the reaction product and this mixture is then agitated for some hr. with 4–20 times its wt. of H_2O . The resulting liquor containing most of the impurities in true or colloidal solution is then drawn off and the benzylcellulose washed with H_2O . This product may be further purified by washing it with org. solvents which have no coagulating action on benzylcellulose and only a very slight swelling action, *e.g.*, a mixture of aliphatic and aromatic hydrocarbons, containing 20–80 wt.-% of aromatic hydrocarbons, a 1:1 mixture being generally suitable. D. J. NORMAN.

Production of artificial silk. H. W. K. JENNINGS. From GLANZSTOFF-COURTAULDS GES.M.B.H. (B.P. 363,505, 15.12.30).—In the wet-spinning process, the thread is led through a perforated trough within the spinning bath, so that any individual filaments which become loosened are carried back to their own thread. F. R. ENNOS.

Production of artificial silk. VEREIN. GLANZSTOFF-FABR. A.-G. (B.P. 363,932, 15.4.31. Ger., 15.4.30).—Spun threads of viscose or cuprammonium silk are wound on light, perforated tubes, in which are inserted similarly perforated supporting tubes, *e.g.*, of Al, during the spinning, washing, after-treating, and, if necessary, drying processes; the spooled thread is subsequently supplied for direct working up without the supporting tube. F. R. ENNOS.

Production of viscose silk. H. A. GILL. From GLANZSTOFF-COURTAULDS GES.M.B.H. (B.P. 363,441, 3.10.30).—0.01–0.5 wt.-% of cholic acid or a bile acid derivative thereof is added to viscose, which is spun to give fibres of increased stretch. F. R. ENNOS.

Production of artificial silk from viscose. ALGEM. KUNSTZIJDE UNIE N.V. (B.P. 363,908, 5.3.31. Holl., 8.3.30).—Viscose is treated at any stage of its manufacture with < 2 wt.-% of a highly sulphonated fatty acid or fat, *e.g.*, 0.1% of "Praestabit" (a sulphurinate), together with small amounts of alkylated aromatic sulphonic acids, *e.g.*, isobutyl-naphthalenesulphonic acids; the proportion of non-sulphonated fat or fatty acid in the highly sulphonated material used should be so small that the latter shows no turbidity with excess of lye. F. R. ENNOS.

Preparation of artificial silk and other artificial products from acetylcellulose. R. E. ELLIS. From C. F. BOEHRINGER & SOEHNE, G.M.B.H. (B.P. 363,700, 14.8.30).—Cellulose is acetylated at a low uniform temp. (20–25°) without addition of those catalysts which may subsequently cause decomp. of the cellulose triacetate (H_2SO_4 , H_3PO_4 , HNO_3 , Fe, Cr, Mn, etc.,

or their compounds), but using as catalyst a halogen, a hydrogen halide, a perhalogen acid or a salt thereof, or a halogenated fatty acid, a swelling agent, *e.g.*, AcOH, being added before or during the reaction in order to retain the fibrous structure of the cellulose throughout the operation. The resulting primary cellulose triacetate is dissolved in CHCl_3 -EtOH or CH_2Cl_2 -EtOH mixtures and spun in the usual manner.

F. R. ENNOS.

Manufacture of artificial threads and other products from cellulose. L. LILIENFELD (B.P. 362,437, 28.5.30).—The etherification or esterification of cellulose xanthate by the processes described in B.P. 335,906, 357,526, 357,154, and 357,121 (B., 1931, 153; 1932, 60, 141) can be effected in the complete absence of H_2O or in the presence of only small quantities thereof. Thus dehydrated viscose or the raw product resulting from the action of CS_2 on alkali-cellulose may be used.

D. J. NORMAN.

Manufacture of [fine threads of] artificial silk. J. DELPECH and C. HEINRICH (B.P. 364,438, 23.3.31. Fr., 5.7.30).—Nitrocellulose is partly dehydrated by replacing its H_2O content by 70% aq. MeOH and draining until it contains about 27 wt.-% of MeOH, after which it is dissolved in anhyd. MeOH (free from EtOH or Et₂O) in the proportion of 12–13 kg. to 100 litres of MeOH. The resulting solution is spun at $\leq 25^\circ$ in 10–40% aq. MeOH, and the threads are treated with 2.5% soap solution before reeling.

F. R. ENNOS.

Production of fine threads from cellulose derivatives. H. SUTER (B.P. 363,929, 9.4.31. Ger., 30.4.30).—Small quantities of unsaturated org. substances (olein, linseed oil, olive oil) together with oils or fats are added to the swelling agent (halogenated hydrocarbons) with which the threads are treated before stretching.

F. R. ENNOS.

Manufacture of yarns with centrifugal spinning machines. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 363,706, 18.9.30. Ger., 12.2.30).—Better results are obtained if the thread pull is greater than has previously been considered suitable, *e.g.*, is equiv. to the centrifugal force exerted by a spinning pot of at least 140 mm. internal diam. revolving at ≤ 6000 r.p.m. Alternatively, a high thread pull may be obtained partly centrifugally and partly by passing a current of air through the guide tube. Suitable machinery is described.

D. J. NORMAN.

Manufacture of paper [for electrical condensers]. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of A. L. ALLEN (B.P. 363,191, 26.1.31. U.S., 25.1.30).—A close, uniform paper (< 0.0005 in. thick) suitable as a dielectric in condensers can be made from kraft pulp if the pulp is washed and then beaten under carefully controlled conditions of gradually increasing severity until the requisite degree of hydration is attained. The total time of beating may be about 36 hr.

D. J. NORMAN.

Manufacture of [water-resistant] paper. G. J. MANSON (B.P. 363,372, 6.6.30).—See U.S.P. 1,762,928—1,762,931; B., 1931, 154.

Preparation [weaving] of india-rubber textile fabrics. W. HALSTENBACH, H. PLUTTE, and C. ROSENKRANZ (HALSTENBACH & Co.) (B.P. 366,019, 25.10.30. Ger., 10.3.30).

Material composed of rubber, canvas, and other sheet material [for soles of footwear etc.]. C. A. BELDAM (B.P. 366,012, 20.10.30).

[Support for] treatment of artificial filaments, threads, bands, etc. [in cake form]. COURTAULDS, LTD., and E. A. MORTON (B.P. 365,810, 2.2.31).

Heat or sound insulation. Conc. of pulps.—See I. Separation of alcohols etc.—See III. Treatment etc. of cellulose materials.—See VI. Laminated glass. Non-splintering sheets.—See VIII. H_2O -sol. resins [for paper].—See XIII. Adhesive mountants.—See XV. Wound-treating materials.—See XX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Determination of substantivities of dyes. General method. W. P. COHOE and E. R. COHOE (Ind. Eng. Chem. [Anal.], 1932, 4, 112–116).—Determinations have been made of the wt. of dye taken up by cloth in dye baths of known concn. and of the corresponding colour vals. of the dyed cloths. The influence of temp., mercerisation, speed, and tightness of twist have also been examined. The results are plotted as standard graphs, from which it is possible to tell how much dye must be fed into the machine to compensate for that taken out by the cloth.

E. S. HEDGES.

Direct cotton dyeing.—See IV.

PATENTS.

Coloration of [cellulose ester and ether] materials. BRIT. CELANESE, LTD., G. H. ELLIS, T. OCKMAN, and H. C. OLPIN (B.P. 361,362 and Addn. B.P. 361,381, 15.5.30).—Aralkylated arylamines, *e.g.*, benzylethylaniline, are incorporated with cellulose ester and ether materials before, during, or after dyeing (etc.) with (A) aminoanthraquinones, (B) other dyes, in order to increase the fastness to light and acid.

C. HOLLINS.

Printing or colouring or colour-embossing of textile fabrics. G. R. HUDSON (B.P. 363,358–9, [A, B] 11.9.30).—Fabric is simultaneously printed and steamed by passage, while in contact with a wet blanket, between (A) pairs of rollers, the upper being a printed and the lower a heated roller, or (B) between an upper printing platen and a lower heated platen. The blanket is sprayed with H_2O at suitable stages.

A. J. HALL.

Cleansing of wool and other animal hair from spots of tar and paint stains. H. HAAKH (B.P. 363,026, 6.10.30).—Wool (etc.) is treated with a tar oil or petroleum distillate having a high b.p. and containing a fibre-lubricating product, *e.g.*, Turkey-red oil, glycerin, or wool grease, until the stain is removed, and is then washed with H_2O , the tar oil being afterwards separated from the wash water.

A. J. HALL.

Treatment [conditioning] of textile fabrics. A. W. BENNIS (B.P. 364,130, 28.6., 11.10., and 18.10.30).—The fabric is passed through a chamber where it is treated with a homogeneous gas stream charged with a volatile liquid to form a vapour, *e.g.*, air and steam, the liquid being deposited at a const. rate throughout the surface area of the material by a sudden reduction in temp. and/or pressure. Suitable apparatus is described.

F. R. ENNOS.

Treatment [with caustic alkalis] of fabrics, yarns, or other products composed of regenerated cellulose, or mixtures thereof with natural cellulose or other fibres, or fabrics impregnated or coated with regenerated cellulose. W. MARSHALL (B.P. 363,883, 7.2.31).—The protecting agents described previously (B.P. 323,307; B., 1930, 281) are replaced wholly or partly by Na_2CO_3 or NaHCO_3 , or the alkali in the mercerised material may be neutralised by exposure to CO_2 before washing. A. J. HALL.

Sizing of artificial silk and other textile threads. A. I. KOUSNETZOFF (B.P. 363,448, 15.10.30. Fr., 26.3.30).—Sizes are used containing the resinates of amino-alcohols (*e.g.*, a resinate obtained by heating triethanolamine with colophony) and any suitable gum or wax; they are easily removed by scouring before dyeing. A. J. HALL.

Improvement [strengthening] of fibres of cellulose esters. SOC. CHEM. IND. IN BASLE (B.P. 363,284, 2.6.31. Switz., 2.6.30).—Esterified fibres obtained without degradation of the cellulose from which they are derived (*cf.* B.P. 349,322 and 353,978; B., 1931, 714, 921) are treated with a swelling agent, *e.g.*, aq. AcOH , pyridine, and NH_4CNS , and then stretched up to 150%; the resulting fibres have increased softness and tensile strength (up to 50%) both in the dry and the wet condition. A. J. HALL.

Production and treatment of filaments, yarns, ribbons, films, and other spun products made of or containing cellulose esters or ethers. BRIT. CELANESE, LTD., and W. I. TAYLOR (B.P. 362,939, 8.9.30).—The silk is treated at relatively high temp. with reagents which are inert at room temp., but become solvents or plasticisers for cellulose esters or ethers at higher temp., *e.g.*, glycerol, glycols, or other polyhydric alcohols, either alone or in admixture with inert diluents, *e.g.*, H_2O , C_6H_6 , or with substances which are solvents or plasticisers for cellulose esters or ethers at room temp., *e.g.*, AcOH , lactic acid, Me or Et ethers of ethylene glycol, Et lactate, Et₂ tartrate, diacetone alcohol, etc. If the silk is stretched during this treatment the finished thread shows increased strength; if it is allowed to shrink the thread shows increased extensibility but no material increase in strength. D. J. NORMAN.

[Delustring] treatment of materials made of or containing cellulose derivatives. BRIT. CELANESE, LTD. (B.P. 363,426, 24.9.30. U.S., 5.10.29).—A mixture of a solvent and a less volatile non-solvent for the cellulose derivative is applied to cellulose acetate (*etc.*) silk and the mixture then removed by evaporation. A suitable mixture consists of xylene (30%) and COMe_2 (70%). A. J. HALL.

[Delustring] treatment of materials made of or containing cellulose derivatives. BRIT. CELANESE, LTD. (B.P. 364,020, 25.9.30. U.S., 8.10.29. Addn. to B.P. 363,426; preceding abstract).—In the process of the prior patent, a mixture of a large proportion of the solvent with a small proportion of the less volatile non-solvent (*e.g.*, COMe_2 80—95 pts., xylene 20—5 pts.) is employed in order to cause individual threads to coalesce. F. R. ENNOS.

Impregnating [textile, leather, and wood] materials. SOC. CHEM. IND. IN BASLE (B.P. 363,918, 23.3.31. Switz., 22.3.30).—Liquors used for softening, sizing, waterproofing, or otherwise treating fibrous materials are prepared with an aq. dispersion of a lipid-sol. substance made with the aid of salts of partly acylated diamines and their NH_4 bases described in B.P. 358,202 (B., 1932, 138). A. J. HALL.

Making fibrous substances impermeable to fluids. E. T. RYDBERG (B.P. 364,466, 14.4.31).—Leather, cardboard, *etc.* are soaked in a mixture of vulcanisable oils, *e.g.*, boiled linseed oil, and S_2Cl_2 (preferably diluted with benzine), the quantity of S_2Cl_2 employed at first being about 75% of that necessary to bring about complete vulcanisation; vulcanisation is then completed by adding the remainder of the S_2Cl_2 . Any HCl evolved is neutralised by washing with aq. NaOH . F. R. ENNOS.

Silicification of fibrous materials of vegetable origin. O. STINER, and LIGNAT WERKE A.-G. (B.P. 362,955, 6.8.30. Switz., 6.8.29).—The material (*e.g.*, straw) is opened up by the action of acid-forming bacteria (*e.g.*, *Bacillus lactis aërogenes*), then heated to arrest bacterial action, and while saturated with acid is treated with aq. Na_2SiO_3 . A. J. HALL.

Treatment of artificial silk. BRIT. BEMBERG, LTD. (B.P. 363,958, 9.6.31. Ger., 13.6.30. Addn. to B.P. 312,034; B., 1930, 505).—When the untwisted treated silk is dried in skein form the filaments tend to stick together. This is avoided by unwinding the silk from the wet skein and twisting it before drying. D. J. NORMAN.

[Rollers for] discharge printing of fabrics [in half-tones]. ROTOPHOT A.-G. F. GRAPHISCHE IND. (B.P. 362,957, 9.8.30. Ger., 10.8.29).

Hank-treating apparatus. [Slating machine.] J. COLEY, S. A. MAIN, and WILDT & Co., LTD. (B.P. 366,179, 19. and 26.11.30).

Wetting *etc.* agents. Purifying dry-cleaners' solvent. Sn derivatives of phenols.—See III. Application of trisazo dyes.—See IV. Cleansing rags *etc.*—See V. Cleansing compound.—See XII.

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

Petersen tower system [of sulphuric acid manufacture]. H. PETERSEN (Chem.-Ztg., 1932, 56, 53—54, 74—76).—Capital and working costs of the author's plant for use with SO_2 from metallurgical processes are discussed and compared with those of the normal Pb-chamber and contact processes. It is claimed that the tower system is the cheapest of the three for all concns. up to 95% acid, that it is capable of dealing with gases of fluctuating SO_2 content and with those in which the supply of SO_2 is temporarily interrupted, and that it can use economically gases down to 1.5% SO_2 content. C. IRWIN.

Ammonia-soda process. V. Calcination of sodium bicarbonate. N. F. JUSCHKIEVITSCH and A. V. AVDEEVA (J. Chem. Ind. Russ., 1931, 8, No. 17, 4—17).—Measurements of the total CO_2 and H_2O

pressure of NaHCO_3 heated so that decomp. proceeds at const. velocity indicate that the reaction consists of: $\text{NaHCO}_3 \rightarrow \text{Na}_2\text{CO}_3, 4\text{NaHCO}_3 \rightarrow \text{Na}_2\text{CO}_3$. The ratio of H_2O to CO_2 in the evolved gases is approx. 1 up to the moment when no more free NaHCO_3 remains, when it rises sharply to 1.8, thereafter again falling to 1. The process of conversion of NaHCO_3 into Na_2CO_3 proceeds very slowly at $<140^\circ$, in spite of the fact that the dissociation pressure attains 1 atm. at 101° . Variations from 0.3 to 3.2 in the ratio $\text{H}_2\text{O}:\text{CO}_2$ in the atm. in which the reaction takes place have only an insignificant effect on the velocity of decomp. NaHCO_3 should be dried by passing a current of CO_2 from the calcination ovens over the moist NaHCO_3 , which moves in a rotating drum in the same direction as the gas, the temp. of which should be at least 150° on entering the dryer, and 80° on leaving it; in this way the most rapid drying is effected with the least risk of decomp. of NaHCO_3 , and with the greatest economy of hot gas. Dried NaHCO_3 should then be calcined in rotatory ovens heated by fuel gas. In this way the number of ovens can be reduced by 50%, with an economy in fuel of 30% as compared with the ordinary process.

R. TRUSZKOWSKI.

Salt manufacture and salt biology. L. G. M. BAAS BECKING (Chem. Weekblad, 1932, 29, 98—100).—A lecture, dealing principally with the influence of bacteria, protozoa, and other organisms on the prep. of NaCl from brine, and especially from sea- H_2O .

H. F. GILLBE.

Equilibrium of potassium, sodium, and magnesium chlorides in water at high temperatures. E. I. ACHUMOV and B. B. VASILIEV (J. Chem. Ind. Russ., 1931, 8, No. 17, 17—23).—The solubility diagrams of the system $\text{NaCl-KCl-H}_2\text{O}$ at $0-200^\circ$ indicate that the best method of prep. of KCl from Solikamsk sylvinitite consists in treating not more than 2.34 tons of sylvinitite with 1 ton of H_2O at 150° , when 1.234 tons of NaCl containing 14 kg. of KCl remain undissolved, whilst the solution deposits large crystals of KCl on cooling.

R. TRUSZKOWSKI.

Origin of, and transformation processes in, the German potash beds. M. RÓZSA (Kali, 1931, 25, 136—139, 153—155; Chem. Zentr., 1931, ii, 834—835).—A summary.

A. A. ELDRIDGE.

Crystallisation of ammonium oxalate from aqueous solutions containing potassium ions. W. DOMINIK and J. BARTKIEWICZÓWNA (Rocz. Chem., 1931, 11, 795—804).—K-free $(\text{NH}_4)_2\text{C}_2\text{O}_4$ cannot be obtained from aq. solutions containing K, the content K, of $\text{K}_2\text{C}_2\text{O}_4, \text{H}_2\text{O}$ in the solid phase being given by $K/AK_1=C$, where A is the $(\text{NH}_4)_2\text{C}_2\text{O}_4$ content of the solid phase, K, the mean [K], and C a const. (0.045 at 20°). The K content of the crystals is smaller at higher than at lower temp. of crystallisation. At least one recrystallisation is necessary to obtain $(\text{NH}_4)_2\text{C}_2\text{O}_4$ containing $<1\%$ K.

R. TRUSZKOWSKI.

Preparation of phosphoric acid and phosphates. A. P. DUNAIEV (J. Chem. Ind. Russ., 1931, 8, No. 14, 35—38).—Decomp. of Ca phosphite commences at 300° and is complete at 600° , the reactions being: $\text{Ca}_2\text{H}_4\text{P}_2\text{O}_7, 2\text{H}_2\text{O} = 2\text{CaHPO}_4 + \text{H}_2\text{O} + 2\text{H}_2$,

$2\text{CaHPO}_4 \rightarrow \text{Ca}_2\text{P}_2\text{O}_7$, and $6\text{CaHPO}_4 + 5\text{H}_2 = 2\text{Ca}_3(\text{PO}_4)_2 + \text{P}_2 + 8\text{H}_2\text{O}$. The production of P commences at 460° , and rises to a max. of 2% at 580° . Max. evolution of PH_3 is 0.05% of total P.

R. TRUSZKOWSKI.

Citrate-insoluble phosphoric acid in di- and tricalcium phosphates. Some factors affecting its determination. K. D. JACOB, L. F. RADER, JUN., H. L. MARSHALL, and K. C. BEESON (Ind. Eng. Chem. [Anal.], 1932, 4, 25—31).—The solubility of CaHPO_4 and $\text{Ca}_3(\text{PO}_4)_2$ in NH_4 citrate solutions varies with the wt. of sample taken, with the p_{H} of the solution, and with the presence of other salts. The solubility increases as the particle size decreases and decreases with temp. of ignition up to 600° .

E. S. HEDGES.

Solubilisation of tricalcium phosphate [for fertilisers]. P. JOLIBOIS and G. CHAUDRON (Sci. Agric., 1932, 12, 317—318).— PbCl_2 is added to an aq. suspension of $\text{Ca}_3(\text{PO}_4)_2$ rendered faintly acid with HCl . The resulting Pb chlorophosphate is treated with HNO_3 of requisite concn. and at a suitable temp. to cause the separation of most of the $\text{Pb}(\text{NO}_3)_2$ (which is subsequently reconverted into PbCl_2 by treatment with HCl). The mother-liquor is neutralised with aq. NH_3 , the small ppt. of Pb phosphate removed, and the solution of NH_4 salts concn.

A. G. POLLARD.

Determination of lead peroxide. III Bromo-potentiometric method. A. V. PAMFILOV and E. G. IVANČEVA (J. Gen. Chem. Russ., 1931, 1, 760—764; cf. B., 1930, 142).—The results obtained by Rupp and Siebler's method (B., 1924, 466) are of the same order of accuracy as are those given by the iodometric method.

R. TRUSZKOWSKI.

Is the determination of hydrolytic acidity of fuller's earth a new way of estimating its decolorising power? O. ECKART (Chem.-Ztg., 1932, 56, 67).—Experiments show that hydrolytic activity cannot be taken as a criterion of relative bleaching power (cf. Utermöhlen, B., 1931, 881). CaCO_3 present in natural fuller's earth affects the acidity and not the bleaching power, whilst in chemically-treated earths an accurate correction for retained acid cannot be made.

E. LEWKOWITSCH.

Production of hydrogen and oxygen by electrolysis at high pressures. D. M. NEWITT and H. K. SEN (Inst. Chem. Eng., Jan. 20, 1932. Advance proof, 10 pp.).—Following the presentation of the theoretical aspect of increasing the pressure during electrolysis up to 200 atm., experimental results showed that the conductivity of solutions of NaOH and HCl (with the exception of concn. NaOH) increases to a max. val. and then progressively decreases. There was little pressure effect up to 100 atm. at <2 milliamp. per sq. mm., and a quant. estimate of the saving in energy is made varying between 5.8 and 11.5% for c.d. vals. of 0.5—3.5 milliamp. A small-capacity, high-pressure cell (designed for the Imperial College of Science and Technology) and the Noegerrath cell are described.

C. A. KING.

Distillation methods for determination of sulphur dioxide. P. F. NICHOLS and H. M. REED (Ind. Eng. Chem. [Anal.], 1932, 4, 79—84).—Rapid heating with

electrical heaters is recommended in the distillation of SO_2 from dried fruits. HCl liberates SO_2 more rapidly and completely than does H_3PO_4 of the same equiv. concn., and better results are obtained with the more conc. acid. The addition of NaHCO_3 increases the yield of SO_2 from dried fruits, but makes no difference to the yield from H_2SO_3 solutions. The Monier-Williams gravimetric method gave the highest consistent recovery of SO_2 from H_2SO_3 solutions.

E. S. HEDGES.

Absorption of sulphur trioxide by water vapour. I. E. ADADUROV and D. V. GERNET (J. Chem. Ind. Russ., 1931, 8, No. 18, 12–16).—6–8% of moist SO_3 , obtained by the contact process from moist SO_2 , remains unabsorbed by H_2O under the usual conditions; 99% absorption is obtained by heating SO_3 with H_2O for ≤ 6 sec. at 338–450° and condensing the H_2SO_4 so formed.

R. TRUSZKOWSKI.

Bleaching earths.—See I. **Utilisation of coal. Determining S in gases.**—See II. **Synthesis of Ca silicates.**—See VIII. **Pt [for synthesis of NH_3].**—See X. **Determining PO_4^{3-} in fertilisers.**—See XVI. **Analysis of Ca glycerophosphate.**—See XX.

See also A., Feb., 110, **Luminescence of Zn compounds.** 113, **Cryst. $\text{Pb}(\text{NO}_3)_2$.** 120, **Alkali Al silicates.** 121, **$\text{Al}(\text{OH})_3$ and Fe_2O_3 sols.** 122, **Hydrogels of SiO_2 and SnO_2 .** 124, **Pb-chamber process.** 129, **Blanc's Al_2O_3 . Catalysts (various).** **Electrolysis of Be compounds.** 130, **Production of NO in the electric arc. Photochemical formation of O_3 .** 131, **Explosion of alkali chlorates by shock. Fluoroberyllates.** 132, **TiN. PbS_2 . Ferromagnetic Fe_2O_3 .** 135, **Salicylic acid in alkalimetry.** 140, **Ultramarines.**

PATENTS.

Manufacture of nitric acid from ammonia. H. FRISCHER (B.P. 363,436, 1.10.30. Ger., 3.10.29. Cf. B.P. 221,513; B., 1925, 713).—Conc. HNO_3 (d 1.32 or over) is formed solely by the reaction of NH_3 oxidation products at atm. pressure in a chamber cooled indirectly to above 0° by a cooling-tube system. Part of the NO is converted into HNO_3 in the presence of HNO_3 mist. Excess H_2O vapour is eliminated by cooling before admittance of the gaseous products to the oxidation chamber. The chamber is provided with inlet and outlet pipes arranged tangentially to its wall, and with means for causing the reagents to travel downwards.

F. YEATES.

Manufacture of anhydrous hydrofluoric acid and anhydrous fluorides. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 363,424, 24.9.30).—F gas is passed through HF, or fused fluorides, or layers of solid fluorides, containing H_2O , at room temp. or above, the reaction effecting dehydration.

F. YEATES.

Elimination of sodium carbonate from technical solutions of potassium carbonate. I. G. FARBENIND. A.-G. (B.P. 363,971, 9.7.31. Ger., 20.8.30).—A solution saturated with respect to K_2CO_3 , and having d 1.52, is cooled to at least -15° and separated from the deposited Na_2CO_3 .

F. YEATES.

Manufacture of shaped calcium oxide. A.-G. F. STICKSTOFFDÜNGER (B.P. 363,526, 19.1.31. Ger., 17.1.30).

—Substantially dry CaO powder, or $\text{Ca}(\text{OH})_2$ powder, is pressed into shape without the addition of any binding agent; a hard, coherent mass is formed. The pressing operation may be followed by calcination.

F. YEATES.

Purification and, particularly, elimination of acids from hydrogen peroxide solutions by electrolysis. OESTERR. CHEM. WERKE G.M.B.H. (B.P. 364,457, 8.4.31. Austr., 24.7.30).—A diaphragm cell is employed, the H_2O_2 solutions serving as catholyte or anolyte in any order of succession, and an H_2O_2 solution of the same concn. as that to be purified being used as second electrolyte to prevent loss of concn. by diffusion. Substances desirable in the purified solution are incorporated during anodic operation by ionic migration, for which purpose dil. H_3PO_4 or a solution of a phosphate or a pyrophosphate is used as catholyte. The electrolyte taking up the impurities is supplemented only by diffusion and electrolytic transport so that the separated impurities will accumulate in that electrolyte, and, in the last cell, such a voltage is maintained that back-diffusion of the impurities into the solution is prevented. Several cells connected in series may be used. Suitable cations or additional anions are introduced into the electrolyte, under certain conditions of the cell, in small amounts and in the form of compounds the counter ion of which does not contaminate the solution.

F. YEATES.

Production of ammonium salts. GUTEHOFFNUNGSHÜTTE OBERHAUSEN A.-G. (B.P. 364,201, 10.10.30. Ger., 12.10.29).— $(\text{NH}_4)_2\text{SO}_4$ and $(\text{NH}_4)_2\text{SO}_3$ are prepared by passing NH_3 , O-free S compounds (H_2S), and O_2 or gases containing it at $\geq 400^\circ$ and at the usual or raised pressure over a catalyst comprising Co and/or Ni oxides mixed with excess of H_2O -insol. compounds of Ca, Ba, Mg, or Si. The NH_3 and H_2S may be generated from entirely separate sources, or coke-oven gases etc. may, e.g., be treated to convert S into FeS and then with H_2O to extract NH_3 , the FeS and aq. NH_3 serving as sources for the H_2S and NH_3 , respectively.

L. A. COLES.

Manufacture of alkali acetates. C. ARNOLD. From Dow CHEM. Co. (B.P. 363,948, 18.5.31).— NaOH , KOH , or a mixture of the two is treated with C_2H_2 and H_2O (1:1–4 mols.) at, e.g., 200–350°/1 atm.; the product is recovered as $\text{NaOAc}\cdot 3\text{H}_2\text{O}$ by crystallisation from H_2O after addition of NaOH equiv. to any KOH used in the main reaction.

L. A. COLES.

Manufacture of alkali chromates. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 363,423, 24.9.30).—Cr ores are worked up in the presence of basic filling or thinning materials with a mixture of 25–75% of alkali carbonate and 75–25% of sulphate.

F. YEATES.

Manufacture of chromates and dichromates. BOZEL MALETRA, Soc. Industr. de Prod. Chim. (B.P. 364,361, 23.1.31. Fr., 9.5.30).—Well-pulverised Cr metal, or a substance containing it (e.g., ferrochrome), is oxidised by heating for 4–6 hr. in an autoclave with NaOH lye (40%) or slaked lime in the presence of O_2 or an oxidising gas (air). A chromate is used instead of an alkali in making dichromates. When CO_2 is produced during the reaction it is bound chemically or removed.

F. YEATES.

Production of alumina and alkali phosphates. CHEM. WERKE, VORM. H. & E. ALBERT (B.P. 363,909, 11.3.31. Ger., 22.3.30).—Crude AlPO_4 is heated with aq. NaOH , the liquor is cooled to crystallise $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$, and the mother-liquor, after addition of more NaOH , is used for the treatment of fresh AlPO_4 ; the process is repeated until the liquor is sufficiently rich in NaAlO_2 to yield pure Al(OH)_3 without concn., preferably after residual Na_3PO_4 has been pptd. as $\text{Ca}_3(\text{PO}_4)_2$. L. A. COLES.

Manufacture of barium sulphate of definite granule size. SACHTLEBEN A.-G. F. BERGBAU U. CHEM. IND. (B.P. 363,936, 28.4.31. Ger., 28.4.30).—Pptd. BaSO_4 is mixed with $\gt 10\%$ of a fusion agent, then ignited at $300\text{--}1000^\circ$ or above (according to the desired degree of fineness of the crystals), and chilled in H_2O . The H_2O -sol. salts formed during pptn. of the BaSO_4 are used as fusion agents. F. YEATES.

Preparation of [anhydrous, finely-divided] calcium sulphate. S. F. W. CRUNDALL, I. P. LLEWELLYN, and P. SPENCE & SONS, LTD. (B.P. 363,347, 12.7.30).—Ca compounds, other than the sulphate, and particularly CaCO_3 , CaO , and Ca(OH)_2 , are treated with oleum, H_2SO_4 , or mixtures of H_2SO_4 and sulphates in such quantities that an excess of free acid remains after completion of the reaction, the concn. of this acid being below that required to effect appreciable dissolution of the CaSO_4 formed and high enough to ensure the formation of anhyd. CaSO_4 in extremely finely-divided form. F. YEATES.

Production of dicalcium or dimagnesium phosphate and of mixed fertilisers containing the same. KALI-FORSCHUNGS-ANSTALT, G.M.B.H. (B.P. 364,033, 26.9.30. Ger., 1.11.29).—Solutions prepared by dissolving crude phosphates in HCl or HNO_3 are added to boiling CaCl_2 or $\text{Ca(NO}_3)_2$ solution of concn. such that the solvent acid is expelled and CaHPO_4 is pptd. and removed. Alternatively, MgHPO_4 is pptd. by using solutions containing Mg instead of Ca salts, or K_2SO_4 is added to the original solutions and, after removal of gypsum, the liquor is added to boiling aq. $\text{Ca(NO}_3)_2$ to yield a mixed fertiliser on evaporation. L. A. COLES.

Production of alkaline-earth formates. R. KOEPP & Co., E. ELÖD, and M. ENDERLI (B.P. 363,788, 14.11.30).— Ca(OH)_2 (100 pts.), or an aq. suspension of it, is treated at raised temp. and pressure with CO in the presence of, e.g., 5—15 pts. of an alkali (preferably K) formate, hydroxide, phenoxide, acetate, carbonate, sulphide, sulphate, ethoxide, ferrocyanide, or borate. L. A. COLES.

Manufacture of [bivalent] metal cyanamides. H. WITTEK (B.P. 364,225, 24.10.30).—A bivalent metal oxide, e.g., CaO produced by calcining lime sludge from the decomp. of CaC_2 etc., is treated at dull to bright redness and at 1 atm. with a mixture of NH_3 and CO_2 (which, if desired, has been passed over imide-forming or H_2O -removing catalysts) under conditions such that the H_2O content of the furnace gases is $\gt 4\%$ (preferably 1.5—2%). Catalysts (halides, cyanamides, etc.) may be added to the oxides; hydrocarbons may be mixed

with the reacting gases, and the product may be treated with CO_2 to yield a weakly alkaline fertiliser. L. A. COLES.

Purification of zinc chloride. A. E. WHITE. From AMER. SMELTING & REFINING CO. (B.P. 364,049, 24.9.30).— ZnCl_2 containing impurities such as Pb, PbCl_2 , and Pb_2OCl (from the Parkes process of Pb refining) is melted in the presence of excess Zn metal, portions of the molten chloride being recirculated through the underlying metal to effect removal of the lead impurities and clarification of the ZnCl_2 , which latter forms a slag on the surface of the bath and is tapped off at a point above the level of the impurities. The Pb containing Zn as an impurity is caused to react with Cl_2 to form ZnCl_2 , which is treated as above. F. YEATES.

Manufacture of [dehydrogenation] catalysts. HOWARDS & SONS, LTD., J. W. BLAGDEN, and G. C. H. CLARK (B.P. 363,776, 6.11.30).—A suitable support (such as pumice stone or coke) is soaked in a solution of a mixture of salts of the metallic oxides capable of being easily decomposed into oxides, with elimination of acid radicals, by heating at $< 500^\circ$, and then heating the mass to the decomp. temp. F. YEATES.

Treatment [chlorination] of metallic oxy-compounds. L. MELLERSH-JACKSON. From INTERMETAL CORP. (B.P. 363,622, 18.8.30).—The gas mixture obtained by bubbling Cl_2 through SCl_2 (or a mixture of SCl_2 and S_2Cl_2) at 50° is passed over the oxy-compounds at a temp. such that one of the constituents is chlorinated and volatilised whereas the other is unattacked; e.g., on treating a mixture of Ta_2O_5 and Nb_2O_5 at 200° , NbCl_5 volatilises and the Ta_2O_5 is unattacked. In some cases double chlorides are formed, e.g., TiO_2 yields $\text{TiCl}_4 \cdot \text{SCl}_2$. L. A. COLES.

Conversion of liquid carbon dioxide into solid carbon dioxide. DRY ICE CORP. OF AMERICA, Assees. of J. D. SMALL (B.P. 363,827, 19.12.30. U.S., 19.12.29).—A mixture of solid and liquid CO_2 (capable of flowing) is treated in a centrifuge equipped with block moulds, the gas pressure being maintained at or above the triple point. The mixture is dried by boiling off the liquid, or the liquid may be frozen, during the operation, by means other than the cooling effect of its own expansion. F. YEATES.

Manufacture of solid carbon dioxide or equivalent substance. P. BROTHERHOOD, LTD., H. M. DUNKERLEY, and CARBON DIOXIDE CO., LTD. (B.P. 364,322, 23.12.30).— CO_2 (etc.), after being pressed through a compressor and a condenser, is collected as liquid in a receiver and passes thence through a nozzle into a mould in which it is converted into a solid block without any extraneous influence. Suitable apparatus is described. F. YEATES.

Purification of oxygen and hydrogen resulting from electrolytic decomposition of water. A. T. STUART (B.P. 364,462, 13.4.31. Can., 22.4.30).—The gas from an electrolytic cell is led into a purifying chamber, contained in a scrubber, and enters above the level of a liquid formed by the condensation and refrigeration of the caustic spray and evaporated H_2O given off with the gas, the condensate being interposed between the

corresponding gas offtake of the cell and the manifold connecting the offtakes of a battery of cells. The gas is deflected downwards by a baffle through the liquid and is taken off at the top of the scrubber in a dry, cooled condition. Excess condensate is returned to the cell from which it issued. F. YEATES.

Recovery of sulphur from sulphur ores. A. H. VERRILL (B.P. 360,899, 11.8.30).—Low-grade ores containing free S or asphalt, or waste materials from gas works, are heated with steam at 55–60 lb. pressure in a closed vessel filled with a solution containing 1 lb. of Na_2SiO_3 , 2 lb. of $\text{Ca}(\text{OH})_2$, 2 lb. of $\text{Na}_2\text{B}_4\text{O}_7$, 2 lb. of NaCl , and 1 lb. of MgCl_2 per 100 gals. of H_2O . The ore is placed on a perforated plate in the lower part of the vessel and the purified molten S is withdrawn from the collecting chamber below this plate. A. R. POWELL.

Recovery of sulphur from roaster gases. R. F. BACON (B.P. 364,351, 12.1.31).—Air, preheated to 300–600° by hot reduction gases, is admitted to the roasting chamber to enrich the roaster gases in SO_2 from which the S is obtained by means of a carbonaceous reducing agent. Heat from the reduction process is utilised to preheat the air for the roasting. F. YEATES.

C and H_2 . NH_3 and H_2S from gases. Gas mixture for NH_3 synthesis. H_2 from hydrocarbons. SO_2 from oil distillates.—See II. Powdered $\text{Ca}(\text{OH})_2$ and abs. EtOH.—See III. Treating materials containing Fe etc.—See X. Pigments.—See XIII. Hg fulminate. See XXII.

VIII.—GLASS; CERAMICS.

Synthesis of calcium silicates. S. NAGAI and K. AKIYAMA (J. Soc. Chem. Ind., Japan, 1931, 34, 471–473 B).—If equimol. mixtures of CaCO_3 and SiO_2 are ignited, the compound $3\text{CaO}\cdot 2\text{SiO}_2$ is formed at 900–1100°. At 1200–1400° this is converted into $2\text{CaO}\cdot\text{SiO}_2$ and at 1450° or above into $\text{CaO}\cdot\text{SiO}_2$ (m.p. 1540°). Prolonged heating (5 hr.) at 1300° gives a product which is almost entirely $\text{CaO}\cdot\text{SiO}_2$. This compound has very slight hydraulic properties compared with Ca aluminates. C. IRWIN.

Production of glass using sodium silicate. I. J. KITAGORODSKI and N. SOLOMIN (Glastech. Ber., 1931, 9, 349–354; Chem. Zentr., 1931, ii, 892).—Formation of silicate in mixtures into which all the Na has been introduced as silicate takes place more rapidly than in similar mixtures employing other salts; sulphate is particularly disadvantageous. A. A. ELDRIDGE.

Determination of alkali in glass. R. D. SMITH and P. CORBIN (Ind. Eng. Chem. [Anal.], 1932, 4, 137–139).—Sullivan and Taylor's oxalate method (B., 1914, 1154) with the additional use of 8-hydroxyquinoline is recommended for alkali determinations in borosilicate glasses, but not in glasses containing >5% Al_2O_3 or materials of the felspar class. Double pptn. of the oxalates and quinoline salts is recommended. E. S. HEDGES.

Micro-analysis of glass. W. GEILMANN (Glastech. Ber., 1931, 8, 404–408; Chem. Zentr., 1931, ii, 1742).—Zn is pptd. as the mercurithiocyanate. Fragments of glass are heated to fuming with HF and H_2SO_4 , or direct pptn. is effected on glass plates. The reagent is

composed of 3 g. of HgCl_2 and 3.3 g. of NH_4CNS in 5 c.c. of H_2O . 0.1% of ZnO can be detected in 10 mg. of glass. A. A. ELDRIDGE.

Works and laboratory control of the moisture content of ceramic pastes. P. P. BUDNIKOV and V. J. ENDOVITZKY (Chem.-Ztg., 1932, 56, 47–48).—The relation of electrical conductivity of ceramic pastes to moisture content can be used to determine the latter and to provide automatic warning of change of moisture content of pastes in the plant. An arrangement is described whereby automatic control of the moisture content of plastic masses in the pug mill may be obtained. W. J. BOYD.

Refractories. M. NAKAMOTO and K. KAWAI (J. Soc. Chem. Ind., Japan, 1931, 34, 470–471 B).—“Mullox” is a mixture of mullite with up to 20% Al_2O_3 or having as addition only an org. binder. Physical properties of varying compositions are tabulated. Al_2O_3 diminishes the strength and the temp. at which softening under a load of 2 kg./sq. cm. begins. This, however, occurs in any case above 1200°. C. IRWIN.

Slag and spalling tests on firebricks. J. F. HYSLOP and H. C. BIGGS (Trans. Ceram. Soc., 1931, 30, 288–294).—The slagging test is carried out in a crucible built up of standard $9\frac{1}{2} \times 4\frac{1}{2} \times 3$ in. bricks of the material to be tested. The crucible containing the molten slag is rotated on a vertical spindle projecting through the bottom of an oil-fired furnace. The spalling-test furnace is a tunnel divided into a central heating section and two outer cooling sections. The bricks under test are mounted on two trucks (5 bricks each) which are pushed into the heating and cooling zones alternately. The hot zone is heated to 1250° by Silit resistance rods and the cooling zones are cooled by an air fan. The disintegration of the three bricks in the middle of the truck is observed after a number of cycles. J. A. SUGDEN.

Glass for testing spirituous liquors.—See XVIII.

See also A., Feb., 124, Fused products of Cr_2O_3 – SiO_2 system. 131, Hydrothermal synthesis of Ca silicates. Decomp. of Ca_3 silicate at 1000–1300°. 136, Determination of MgO and alkalis after decomp. by HF.

PATENTS.

Manufacture of non-splintering glass, so-called safety glass. I. G. FARBENIND. A.-G. (B.P. 363,933 and Addn. B.P. 363,940, [A] 17.4.31, [B] 5.5.31. Ger., [A] 19.4.30, [B] 5.5.30).—The glass sheets are coated with (A) a condensation product of maleic anhydride with castor oil, butylene glycol, or a high-mol. unsaturated alcohol, or (B) a mixture of a cellulose derivative with the product used in (A), and are united by heat and pressure. L. A. COLES.

Manufacture of non-splintering compound transparent sheets. J. A. NYE and W. W. STAINER (B.P. 364,277, 27.11.30).—Flat or curved compound sheets comprise two glass sheets, one of which is approx. 0.01 in. thick, with an interposed celluloid sheet approx. 0.015 in. thick cemented to them. L. A. COLES.

Manufacture of laminated glass. O. C. HAWKES, LTD., and B. G. T. HAWKES (B.P. 365,371, 19.2.31).—

Sheets of glass and of cellulose acetate are immersed in a bath composed of 60–90% of glyceryl triacetate, 20–50% of EtOH, and 0.5–2% of camphor until the cellulose acetate is sufficiently plastic, and the sheets are then pressed together, drained, and subjected in an autoclave to liquid or gaseous (*e.g.*, steam) pressure of approx. 250 lb./sq. in. at 93–115° for 15 min.

L. A. COLES.

Manufacture of glass electric insulators. CORNING GLASS WORKS, Assees. of G. S. FULCHER (B.P. 363,934, 17.4.31. U.S., 7.5.30).—The part of the insulator to be united to a metal support is fortified, *e.g.*, by immersion in a mixture of HF and H₂SO₄, and then provided with a heat-resisting layer by, *e.g.*, coating it with "Ault & Wiborg No. 1222" varnish, heating at 130°, and spraying with a colloidal suspension of graphite in H₂O.

L. A. COLES.

Dense refractories. C. A. JENSEN. From GEN. REFRACTORIES Co. (B.P. 364,131, 2.7.30).—Refractory bricks etc. are formed by firing mixtures of 45–65% of particles of 3–30-mesh with 55–35% passing 50-mesh, with the addition, if desired, of small quantities of non-plastic material (SiO₂, chrome ore, diaspore, kaolin, etc.) of intermediate mesh, moulded in the moist state at > 1000 lb./sq. in. (preferably 5000–15,000 lb./sq. in.).

L. A. COLES.

Production of porous heat-insulating substances. L., R., and E. WURM and P. GÜTIG (B.P. 364,819, 23.12.30. Czechoslov., 23.12.29).—Light, porous bricks or shapes which are non-absorptive of H₂O may be formed from a mixture of basic MgCO₃ 85 pts., asbestos 15, paraffin wax 2, and H₂O 8 pts., the last two being in the form of an emulsion. The mixture is pressed into moulds, dried, and heated to above the m.p. of the wax (*e.g.*, 80°), preferably in an atm. containing < 21% of O₂. Other substances, *e.g.*, kieselguhr, cement, sawdust, may be used.

B. M. VENABLES.

Circulating the glass in glass-furnace gathering basins. J. MONCRIEFF, LTD., and A. F. McNISH (B.P. 365,514, 19.9.30).

Firing furnace.—See I. **Resistance material.**
Contact points of resistors.—See XI.

IX.—BUILDING MATERIALS.

Colloidal hardening of cement. II. H. KÜHL [with W. BÜSSEM and F. THILO] (Zement, 1931, 20, 650–651; Chem. Zentr., 1931, ii, 1619).—A vitreous mass from CaO 50.2, Al₂O₃ 12.9, SiO₂ 26.9% with 20% KOH (free from CO₂) was stored in absence of CO₂. The tensile strength was 28.8–23.4 kg. after 28 days. Not more than 2% of cryst. material was present.

A. A. ELDRIDGE.

Determination of lime in relation to the properties of cement. V. SEVIERI (Annali Chim. Appl., 1931, 21, 545–552).—From considerations of analytical data the new formula: CaO — [free CaO + 0.75 × (2Al₂O₃ + Fe₂O₃ + Mn₂O₃ + SO₃)] : sol. SiO₂ > 3, is considered most suitable for the evaluation of Portland cement of high basicity.

O. F. LUBATTI.

Properties of porous building materials. I. **Absolute porosity and saturation coefficient.** II. **Flow of air through porous bodies.** E. MADGWICK (Phil. Mag.,

1931, [vii], 12, 1155–1160, 1160–1168).—I. The amount of H₂O adsorbed per c.c. of material (porosity), the total pore space per c.c. of material (abs. porosity), and the ratio of these (saturation coeff.) were determined for various building materials.

II. Direct measurements were made of the flow of air through slabs of limestone, sandstone, brick, and plaster, and the increased resistance due to coated surfaces was measured.

H. J. EMELÉUS.

Analysis of mixtures of road tar and asphalt bitumen. H. MALLISON (Asphalt u. Teer, 1931, 31, 598–600; Chem. Zentr., 1931, ii, 1799).—Irregularities of results obtained by the usual methods are discussed. In the distillation method a temp. > 300° is recommended.

A. A. ELDRIDGE.

Test for bituminous emulsions.—See II. **Ca silicates.**—See VIII. **Slate powder.**—See XIII.

PATENTS.

[Light] mortar, concrete, and the like. F. E. MARECHAL (B.P. 363,746, 9.10.30. Belg., 12.10.29. Addn. to B.P. 349,957; B., 1931, 761).—The distribution of bubbles and texture of the mass are improved by using several agitators operating in opposite directions.

C. A. KING.

De-aëration of hydraulic binding agents and mixtures for use in connexion with the manufacture of concrete, agglomerates, and artificial stone. E. FREYSSINET and J. SEAILLES (B.P. 365,290, 30.12.30. Fr., 30.12.29).—The concrete etc., after it has been mixed with H₂O, is drawn by vac. through jets in the bottom of a hopper leading into a receptacle below; de-aëration may be facilitated by the provision of centrifugal apparatus etc. in the receptacle, and by vibrating the apparatus.

L. A. COLES.

Production of porous masses and objects. DEUTS. GOLD- u. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 363,058 and Addn. B.P. 363,192, [A] 28.10.30, [B] 27.1.31. Ger., [A] 28.10.29, [B] 1.2.30).—(A) Compounds from which the evolution of O₂ is controlled by decomposing catalysts are incorporated in a cement mixture which may also contain colloids to increase the homogeneity of the gas bubbles. Thus, 2.1 kg. of calcined gypsum are mixed with 2.6 litres of H₂O and 30 c.c. of 0.5% soap solution. When the consistency is uniform 100 c.c. of 6% H₂O₂ solution are stirred in and a suspension of 20 g. of MnO₂ in 50 c.c. of H₂O is added. The mass is then cast. (B) Non-setting substances, *e.g.*, kaolin, quartz, kieselguhr, are treated similarly and the mass formed is solidified by drying and burning in a kiln.

C. A. KING.

Manufacture of blocks, slabs, and other shaped bodies having a cellular structure. A. F. BERRY (B.P. 365,154, 10.10.30).—Mixtures comprising substances for producing a gas by chemical action so as to yield cellular products, *e.g.*, such as are described in B.P. 329,291 and 355,278 (B., 1930, 715; 1931, 990), after working to a dough with hot water and moulding, are subjected to a partial vac. in a heated chamber until they have set. The products may subsequently be dried by exposure to air under pressure (1 atm. or above), treated with, *e.g.*, MgSiF₆ or Na₂SiO₃ solution to harden

the surface, or impregnated with paint, varnish, etc. or with a fertiliser. L. A. COLES.

Manufacture of tarred macadam and the like. J. F. WAKE (B.P. 363,655, 18.9.30).—Road material is coated firstly with a heavy bituminous compound, then with one of lower viscosity, and lastly with a crude petroleum or tar oil. The coatings may be applied successively before discharging the material from the mixer. C. A. KING.

Making cold-flowing material for bituminous road surfacing, which hardens after being applied, and road-surfacing therewith. A. BREUER (B.P. 363,698, 21.7.30).—Tar, pitch, or asphalt, alone or mixed, is dissolved by heat in chlorinated hydrocarbons under pressure and applied cold. C. A. KING.

Impregnating wood with corrosive sublimate. K. BUBLA (B.P. 363,760, 21.10.30).—To reduce the unnecessary high absorption of HgCl_2 , wood is impregnated first with other solutions of high capillarity, e.g., alkali, NH_4 , and Al salts, esters, and chlorinated org. compounds. C. A. KING.

Preservation of wood. H. BAKER (B.P. 364,387, 13.2.31. Addn. to B.P. 352,650; B., 1930, 885).—Turnbull's blue, Prussian blue, Ca ferro- or ferri-cyanide, or Mn ferrocyanide is pptd. in the wood; or the wood is impregnated with a solution of a sol. ferrocyanide (other than of K or Na) which is subsequently oxidised by the air, Cl_2 , KMnO_4 , etc.; or it is impregnated successively with a sol. cyanide and a Fe^{++} salt and the ppt. formed in the fibres is oxidised. L. A. COLES.

Drying kilns [for lumber etc.]. Drying of road-making aggregates. Heat or sound insulation.—See I. Asphalt.—See II. Impregnating wood. Silicified fibres.—See VI. Heat-insulating substances.—See VIII. Exterminating vermin.—See XXIII.

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

Temperature measurement and regulation in metallurgical furnaces. F. KOFLER and G. SCHEFELS (Stahl u. Eisen, 1931, 51, 1529—1535).—The necessity for careful temp. control in steelworks' furnaces of all kinds to obtain uniform products in the various processes is emphasised and the operation of automatic temp.-control devices based on an optical pyrometer (the Ardometer) is described with reference to sp. examples. A. R. POWELL.

Blast-furnace data and their correlation. A. WAGNER and A. HOLSCHUH (Arch. Eisenhüttenw., 1931—2, 5, 279—290).—The work of Evans, Reeve, and Vernon (B., 1931, 635) is discussed. A. R. POWELL.

Dressing of iron ores. N. N. CHINKIN (Inst. Mekh. Obrabotki Polez. Iskop., 1931, 37 pp.).—Brown ores of the Central Industrial Region (Govt. of Tula) were investigated. CHEMICAL ABSTRACTS.

Roasting iron ores for reduction. I. N. MASLENTZKI (Inst. Mekh. Obrabotki Polez. Iskop., 1931, 47 pp.).—A review of processes of partial reduction of hæmatite and brown Fe ores for the purpose of magnetic concn. CHEMICAL ABSTRACTS.

Manganese reduction in manufacture of steel. E. MAURER and W. BISCHOF (Z. physikal. Chem., 1931, 157, 285—309).—The distribution of Mn between the metal and slag in the basic open-hearth process for the manufacture of steel has been calc. from data for the equilibrium $\text{FeO} + \text{Mn} \rightleftharpoons \text{MnO} + \text{Fe}$. For a given amount of Mn in the charge, the greater is the amount of Ca in the slag, the greater will be the proportion of Mn in the metal. The application of the theoretical deductions to actual furnace practice is described. R. CUTHILL.

Influence of atmosphere and temperature on the behaviour of steel in forging furnaces. D. W. MURPHY and W. E. JOMINY (Dept. Eng. Res., Univ. Michigan, 1931, Bull. No. 21, 150 pp.).—Scaling loss is increased by increasing the rate of flow of the gases from < 4 to 6 ft. per min., but no further increase occurs with a velocity of > 10 —20 ft. per min.; the loss varies with the partial pressure of the oxidising gas. Temp. is an important factor and the loss becomes marked at the m.p. of the oxides, about 1360° . Steels containing $> 0.5\%$ C become slightly more resistant. In an atm. of H_2O , CO_2 , H_2 , and CO the scale at 1100 — 1360° is composed mostly of FeO , and in an oxidising atm. Fe_3O_4 predominates. Equilibria conditions from 1093 to 1427° have been determined for the systems Fe—O—H and Fe—O—C from the condition that the composition of a gas phase in equilibrium with Fe and FeO must lie between the compositions of two gas phases one of which produces scaling and the other inhibits oxidation. The m.p. of FeO was determined as $1360^\circ \pm 10^\circ$. S gases increase scaling losses, H_2S to a lesser degree than SO_2 , and generally oxysulphide inclusions are formed along the grain boundaries. In practice steel should be exposed to high temp. for as short a period as possible and every precaution should be taken not to exceed the burning temp. of steel. C. A. KING.

Formation of cementite at the grain boundaries of mild steel and its effect on the mechanical properties. H. KORNFIELD and G. BRIEGER (Arch. Eisenhüttenw., 1931—2, 5, 315—322).—The conditions for the separation of cementite along the grain boundaries of mild open-hearth steel containing 0.11% C, 0.09% Si, 0.41% Mn, 0.01% P, and 0.03% S have been investigated by annealing sheets for 0.5—3 hr. at 740 — 820° after various degrees of reduction by rolling. The results show that annealing between the Ac1 and Ac3 points under the crit. recrystallisation conditions is most favourable for producing string-like segregations of cementite along the grain boundaries. Steel with a fine-grained structure and this type of cementite segregations has a lower impact strength than similar steel with a coarser grain and granular cementite segregations. Coarse-grained steel with string-like cementite is more sensitive to notched impact than that with granular cementite, but cannot be considered as brittle, although it exhibits "blue-brittleness" to a marked degree. A. R. POWELL.

Extraction of slag and oxide inclusions in iron or steel. Electrolytic method. F. W. SCOTT (Ind. Eng. Chem. [Anal.], 1932, 4, 121—125).—The specimen of Fe or steel is attached to a Pt wire and made the

anode of a cell containing MgI_2 solution and a Cu-screen cathode. The Fe dissolves and the inclusions are collected in a filter paper which forms a partition round the anode. The MgI_2 prevents the formation of $Fe(OH)_2$ and $Fe(OH)_3$. Directions are given for analysing the residue. In the "killed" steels, where the main inclusion is Mn silicate, the recovery is almost 100%; in the "rimmed" steels it is slightly less. E. S. HEDGES.

Electrolytic extraction of manganese oxide, manganese sulphide, iron sulphide, and silica inclusions from plain carbon steels. G. R. FITTERER (Amer. Inst. Min. Met. Eng. Tech. Pub., 1931, No. 440, 15 pp.).—The steel (50 g.), suspended in collodion bags in $FeSO_4$ solution, is electrolysed (3–4 volts). The electrolytic method is applicable only to straight steels.

CHEMICAL ABSTRACTS.

Proof of the independence of hydrogen content and hardness of electrolytic iron. GUICHARD, CLAUSMANN, BILLON, and LANTHONY (Compt. rend., 1931, 193, 1084–1085).—The discrepancy between the results of the authors and of Guillet and co-workers (cf. B., 1932, 65) is attributed to a misunderstanding of the authors' method (cf. B., 1931, 592) which implied that repeated annealing for 24 hr. each time was necessary for complete elimination of H, so that some would remain under the circumstances described. In the authors' experiment complete elimination was effected by treatment first for 24 hr. at 164° and then for 24 hr. at 240°. It is therefore maintained that complete dehydrogenation can be effected at a temp. at which the hardness is not diminished and that the hardness of electrolytic Fe is not due to H. C. A. SILBERRAD.

Megascopic method of detection of sulphides in steel. K. KATZOWNA (Przemysl Chem., 1931, 15, 383–390).—Ag is removed from collodion photosensitive paper by $Na_2S_2O_3$, the washed Ag-free paper is immersed first in $Pb(NO_3)_2$ and then in H_2SO_4 solutions, again washed, and the polished steel surface is applied to the paper for 5 min., when the presence of sulphides is indicated by a black stain. This method is applicable in the presence of phosphides. R. TRUSZKOWSKI.

Determination of sulphur in steel. A. GUÉDRAS (Aciers spéc., Mét., Alliages, 1931, 6, 75–80; Chem. Zentr., 1931, ii, 1323).—The steel is burned in O_2 at 1200° (electric furnace) and the gases after oxidation are passed into NaOH solution, the excess being titrated with H_2SO_4 . A. A. ELDRIDGE.

Application of chlorine to the analysis of pyrites. S. OSTROWSKI (Rocz. Chem., 1932, 12, 67–70).— Cl_2 is passed over heated pyrites or chalcopyrite for 1 hr., and the issuing gases are absorbed by NaOH, in which SO_4^{--} is then determined. The accuracy of this method is of the same order as that of Lunge, which takes twice as long to perform. R. TRUSZKOWSKI.

Diffusion of mercury into tin foil. W. GERLACH [with E. SCHWERTZER] (Sitzungsber. Bayr. Akad. Wiss., 1930, 223–224; Chem. Zentr., 1931, ii, 826).—The spectroscopic detection of Hg is facilitated by electro-deposition on a small electrode; the form and distribution of Hg in Sn were studied. Hg does not diffuse equally readily in all directions into rolled Sn foil;

diffusion ellipses were obtained owing to the effect of rolling on orientation. Similar ellipses were obtained with rolled Pb. A. A. ELDRIDGE.

Briquetting of chromite concentrates. M. A. GORDIENKO (J. Chem. Ind. Russ., 1931, 8, No. 18, 32–33).—Suitable binders are Sorel cement (80 g. MgO and 60 g. $MgCl_2$ per kg. of concentrate), or Ust-Sisersk mag-nesite (3·5%). R. TRUSZKOWSKI.

Cadmium in high-lead bearing metals. K. L. ACKERMANN (Metall-Wirt., 1931, 10, 593–597; Chem. Zentr., 1931, ii, 1626–1627).—The effect of changes in Cd content on the composition, cryst. condition, and mechanical properties of the alloys is discussed.

A. A. ELDRIDGE.

Volumetric determination of lead in babbitt metal. G. V. LERNERMAN (J. Chem. Ind., Russ., 1930, 7, 387–388).—The alloy (1 g.) is decomposed with 6N- HNO_3 (25 c.c.); H_2O (100 c.c.) is added and after 20 min., when oxides of Sn and Sb have settled, the solution is diluted to 250 c.c. and 100 c.c. are employed for the determination of Pb, which is pptd. in presence of a slight excess of AcOH as $PbCrO_4$. After washing with dil. AcOH this is dissolved in 15–20 c.c. of conc. KOH solution, which is then boiled with 20 c.c. of 5% $KMnO_4$. The excess of $KMnO_4$ is reduced with EtOH, the solution diluted to 250 c.c., and 200 c.c. of the filtrate are used for the iodometric determination of CrO_4^{--} in presence of HCl. CHEMICAL ABSTRACTS.

Silver in lead bullion. Determination by volumetric method. W. R. WAGSTAFF (Ind. Eng. Chem. [Anal.], 1932, 4, 51).—The sample is dissolved in HNO_3 with addition of tartaric acid to keep Sb in solution. AgCNS is pptd. by adding NH_4CNS , the ppt. is dissolved in aq. NH_3 , HCl is added, the AgCl is filtered off, and the filtrate titrated with $KMnO_4$. E. S. HEDGES.

Relation between rolling texture, deep-drawing properties, and hardness of silver, copper, and aluminium and of a silver-copper alloy. H. HOLZMANN (Siebert Festschrift, 1931, 121–148).—The Erichsen vals. for Ag and Cu fall to a min. with an 85–90% reduction in thickness by rolling, then rise sharply on further rolling; for Al the val. falls to a flat min. at 55–75% reduction, then rises slowly, whilst for the 83·5:16·5 Ag-Cu alloy the val. falls slowly to about 50% reduction, then very rapidly, the val. for a 90% reduction being only 0·4 mm. Cross-rolling produces only a slight reduction in Erichsen vals. in all cases and a smaller increase in hardness than continuous rolling in one direction. The Erichsen val. of annealed Ag, Cu, and Al is independent of the degree of rolling when the metal has been cross-rolled, but when rolling has been continuous in one direction it falls with increasing reduction in thickness above 75% for Cu, 95% for Al, and 80% for the Ag-Cu alloy. Unidirectional rolling produces a max. increase of hardness of 200% for Ag, 100% for Cu and the Ag-Cu alloy, and < 50% for Al.

A. R. POWELL.

Platinum as a world historical factor. W. OSTWALD and E. BRAUER (Siebert Festschrift, 1931, 240–256).—An historical account is given of the development of the Pt catalyst in the oxidation of NH_3 in the synthetic prep. of HNO_3 . A. R. POWELL.

Platinum and its alloys in dentistry. K. FALCK (Siebert Festschrift, 1931, 31—50).—The uses of Pt and its alloys as pins for artificial teeth, as foil for crowns, and as supports for dentures are described in detail.

A. R. POWELL.

Thermocouples and the reproducibility of their data. Criteria for their usefulness in measuring high temperatures. W. GOEDECKE (Siebert Festschrift, 1931, 72—99).—Small quantities of Fe, Ir, and Pd in Pt thermocouple wires reduce the e.m.f. of Pt/Pt-10% Rh thermocouples, the effect of Fe being greatest and that of Pd least; similar impurities in the Pt-Rh wire have a much smaller effect. A Pt-8% Re wire has an e.m.f. of 32.5 mv. and a Pt-4.5% Re-5% Rh wire an e.m.f. of 30 mv. against Pt at 1300°; the former alloy tends to lose Re by oxidation after prolonged use at high temp. and the structure becomes coarsely cryst., whereas the latter alloy is remarkably stable. Rh/Rh-8% Re thermocouples can be used up to 1900° (e.m.f. 7.2 mv.). Small quantities of Fe and Mn together in Cu-Ni alloy wires used in thermocouples with Fe wires reduce the e.m.f. of the couple only slightly, whereas Fe alone produces a considerable reduction; higher e.m.f. vals. are obtained for alloys melted in CO than for those melted in vac. For Ni constantan couples the alloy wire should contain 45% Ni, and for Ni/Ni-7% Cr couples addition of 5% Fe to the alloy wire increases the e.m.f. by 2 mv. The e.m.f. curve of Pt/Pt-10% Rh up to 900° is closely followed by that of a 12.3% Ni-87.7% Cu/20% Ni-80% Cu couple.

A. R. POWELL.

Age-hardening of alloys of the system gold-platinum with small additions of a third constituent. W. GOEDECKE (Siebert Festschrift, 1931, 100—107).—Alloys of Au with > 20% Pt can be hardened by quenching from 1000° and ageing at 550°. The effect is much intensified by addition of small quantities of Fe and can be produced in alloys containing only 2% Pt. Max. age-hardening is obtained with the alloy containing 6% Pt and 0.2% Fe; further addition of Fe accelerates the ageing process without appreciably increasing the max. hardness obtainable (130 Brinell units). Curves of hardness/time of ageing above 500° for the alloys with 20% Pt and 0.2—1% Fe show two well-marked max.; the mechanism of this ageing process appears to differ from that of other ageing processes as up to 20% Pt and 18% Fe are sol. in Au.

A. R. POWELL.

Spectrographic analysis of platinum metals and alloys. W. GERLACH and K. RUTHARDT (Siebert Festschrift, 1931, 51—71).—The most sensitive lines for the detection of Cu, Fe, Ir, Ni, Os, Pd, Rh, and Ru in Pt, of Cu, Fe, Ni, Os, Pd, Pt, Rh, and Ru in Ir, and of Cu, Fe, Ir, Ni, Os, Pd, Pt, and Ru in Rh are tabulated. The same lines are not available in all cases, as sometimes the lines of an impurity simply intensify a line due to the metal under test. Homologous pairs of lines for the determination of Ir and Rh in Pt are tabulated and their use in determining Rh and Ir in Pt alloys is described.

A. R. POWELL.

See also A., Feb., 112, **Magnetic Au-Fe alloys.** 116, **Al-Mn, Zn-Mn, and alloys of Ag with Bi, Sb,**

and As. 117, **Systems Cr-C and Fe-Cr-C.** 125, **Desulphurisation of steel.** **Equilibria between metals and slags in melts.** 128, **Passivity of Fe.** **Rate of dissolution of base metals.** **Topochemistry of corrosion and passivity.** **Be.** **Electrodeposition of metals from anhyd. NH₃.** 136, **Determinations of Cu, Al, Mn.** 137, **Determinations of Co and Mn, Fe, V (traces), and Bi.**

PATENTS.

Ore-roasting furnaces. E. DOHET (B.P. 363,100, 20.11.30. Belg., 3.10.30).—In furnaces with moving hearths, inclined scrapers are mounted on the end of a stem the other end of which forms a pivot which is hooked in two notches in the arch of the moving hearth above. When the hearths are stationary the stems are hooked in lateral projections from the rabble arms. The scrapers are removed easily and follow expansional movements of the arches.

C. A. KING.

Open-flame furnaces for melting metals [aluminium]. (SIR) W. G. ARMSTRONG, WHITWORTH & Co. (ENGINEERS), LTD., and I. L. HALL (B.P. 362,100, 20.10.30).—A horizontal, rotating, cylindrical furnace for melting Al and its alloys comprises an Fe casing cast in two halves and lined with refractory material, inside which and spaced therefrom is a cylinder cast in one piece from hæmatite Fe. The cylinder has a charging outlet in the periphery and a boss-like, axial inlet orifice at each end for entry of the flame and exit of the waste gases. Air for combustion of the fuel is preheated by drawing it through the annular space between the refractory and the Fe linings.

A. R. POWELL.

Utilising the heat of melting furnaces. VEREIN. STAHLWERKE A.-G. (B.P. 362,213, 17.1.31. Ger., 31.1.30).—A series of thermocouples is built into the walls of the flue so that the hot junctions come into contact with the stream of hot gases and the cold junctions are cooled in running H₂O outside the flue walls. The current set up in the wires is used to energise magnets arranged in the walls of the furnace below the bath of molten metal, which is kept in const. motion by the action of the magnetic fields thus produced.

A. R. POWELL.

Foundry cores and/or moulds. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of R. EARL, JUN. (B.P. 362,090, 15.10.30. U.S., 17.1.30).—The moulding sand is bonded with a solution of a resin formed by the condensation of a polyhydric alcohol and a polybasic org. acid or its anhydride, e.g., glycerol and phthalic anhydride. A drying oil or the acids obtained by hydrolysis of a vegetable oil, e.g., castor or linseed oil, may also be added. The core or mould is then baked.

A. R. POWELL.

Treating materials containing iron (A) in combination with sulphur, (B) and other values. SULPHIDE CORP., Assees. of (A) R. G. BROWN, (B) E. W. WESCOTT (B.P. 361,903 and 362,343, [A] 22.5.30, [B] 30.5.30. U.S., [A] 23.5.29, [B] 1.6.29).—(A) In a cyclic process for the treatment of pyritic ores with gases containing Cl₂, the preheated ore is treated with a current of hot N₂ containing Cl₂ and FeCl₃ from a succeeding step, whereby the Fe and other metals in the ore are converted into

$M'Cl_2$ and a gas rich in S vapour is obtained. The issuing gas serves to preheat a quantity of air to 350° , at which temp. it is passed over the $FeCl_2$ obtained in the first stage to convert it into Fe_2O_3 , $FeCl_3$, and Cl_2 . The $FeCl_3$ and Cl_2 are carried by the hot stream of residual N_2 into a fresh charge of pyritic ore and the Fe_2O_3 residue is leached for the recovery of Cu, Zn, Pb, etc. as chlorides. (B) The chlorination treatment is carried out at such a temp. ($> 950^\circ$) that all or nearly all of the $FeCl_2$, $CuCl_2$, $NiCl_2$, $CoCl_2$, and $ZnCl_2$ are volatilised, leaving a relatively clean gangue. In this way pure Fe_2O_3 is obtained in the subsequent operations.

A. R. POWELL.

Manufacture of powder from iron or iron alloys. HARTSTOFF-METALL A.-G. (HAMETAG) (B.P. 362,564, 26.9.30. Ger., 28.9.29. Addn. to B.P. 340,762; B., 1931, 300).—Wrought-Fe wire containing 0.05–0.08% C is cut into small pieces which are heated at 850° in vac. for 2 hr., then in N_2 , H_2 , or NH_3 for 1 hr. at the same temp. After cooling, the treated wire is readily pulverised in any of the usual types of mill. A. R. POWELL.

Manufacture of [non-rusting] cast-iron articles. B. VERVOORT (B.P. 362,375, 25.8.30. Ger., 19.5.30).—The alloy comprises Fe with 15–40% Cr, 0.7–2.6% C, and 0.4–3% Si; the castings are made non-rusting by a polishing treatment. [Stat. ref.] A. R. POWELL.

Manufacture of steel abrasive material. GLOBE STEEL ABRASIVE Co. (B.P. 361,383, 19.5.30. U.S., 9.1.30).—A broad stream of cold H_2O is projected under high pressure against a narrow stream of molten steel, whereby the steel is converted into shot of various sizes which are further chilled by being driven by the H_2O stream into a large vol. of cold H_2O . A. R. POWELL.

Treatment of steel and other ferrous alloys. SOC. D'EXPLOIT. DES PROC. MAHOUX, Assees. of A. P. and G. MAHOUX (B.P. 363,376, 6.8.30. Fr., 18.11.29).—The steel is treated below the Ac3 point with NH_3 while being subjected to high-frequency vibrations set up mechanically or electrically to produce a very hard outer nitrogenised layer. A. R. POWELL.

Production of relatively minute metal foils known as bronze colours. HARTSTOFF-METALL A.-G. (HAMETAG) (B.P. 363,604, 13.5.31. Ger., 16.5.30).—An increase in the yield of bronze colour is obtained by pulverising the bronze in a cylindrical mill the load of steel balls in which reaches, when the mill is at rest, a height $>$ two fifths but $<$ five sixths of the radius of the drum. A. R. POWELL.

Production of sheets and bands of crude or refined zinc having improved mechanical properties and increased resistance to corrosion. BERNDORFER METALLWARENFABR. A. KRUPP A.-G. (B.P. 363,962, 12.6.31. Austr., 12.6.30).—Zn is cast in vertical cylindrical moulds, and the top portion containing the pipe and slag inclusions and the bottom portion containing most of the Pb are removed. The rest of the ingot is heated at above 100° , shaped in a rod-press, and then rolled. A. R. POWELL.

Coating refractory metals with rhenium. GEN. ELECTRIC Co., LTD., Assees. of PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 364,502, 4.6.31. Ger., 8.7.30).—Vapour of a volatile Re compound is con-

ducted by means of a current of an inert and a reducing gas, e.g., N_2 and H_2 , over a W wire heated at between 2000° and the m.p. of Re. If a single-crystal W wire is used a ductile single-crystal wire of Re is produced.

A. R. POWELL.

Coating [metallic] wires, bars, bands, threads, etc. GEN. ELECTRIC Co., LTD., Assees. of PATENT TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 363,236, 27.2.31. Ger., 15.4.30).—The material to be coated is passed continuously through a closed chamber in which it is heated electrically to incandescence above a bath containing a volatile compound of the coating material; the atm. in the chamber consists preferably of a reducing gas or of a mixture of a reducing gas and an inert gas. E.g., wires of W, Mo, Ta, Nb, Re, or Pt may be coated with ZrC by the use of a solution of $ZrCl_4$ in PhMe, with Ti_3N_2 using $TiCl_4$ in N_2 and H_2 , with Cr_2O_3 using CrO_2Cl_2 in H_2 , with Hf phosphide using $HfCl_4$ and PCl_3 in H_2 , or with U sulphide using compressed UCl_4 in H_2S . A. R. POWELL.

Production of alloys of beryllium with heavy base metals. H. BARON. FROM SIEMENS & HALSKE A.-G. (B.P. 363,837, 23.12.30).—Alloys of Fe, Ni, or Cu with Be are obtained by fusion of the metal with a slag containing BeO , a reducing agent, e.g., Al, C, or CaC_2 , and an alkaline-earth oxide, preferably BaO. The alloys may be refined by again fusing them with BaO and the BeO slag. A. R. POWELL.

Production of forgings of magnesium or its alloys. I. G. FARBENIND. A.-G. (B.P. 362,834 and 362,837, [A] 3.7.31, [B] 21.7.31. Ger., [A, B] 16.8.30).—(A) An alloy of Mg with 8% Al is cast into a cylindrical ingot which is forged at 280 – 400° until its height is reduced by 30%, then turned at right angles and forged while rotating about its previous vertical axis until the reduction in original height is only 15%. The whole operation is repeated several times until the original height is reduced by 70% and the ingot is then finished by swaging. The yield point after this treatment is 16–18 kg./sq. mm. both in the transverse and longitudinal direction, and the compression strength 14–16 kg./sq. mm. (B) The ingot is plastically deformed to produce a definite orientation of the mechanical properties, and blanks corresponding roughly with the finished shape of the forgings are cut from the ingot in such a way that the direction of the desired max. resistance to a particular kind of stress, e.g., compression, in the finished forging coincides with the direction in which this resistance attains a preferred val. in the rough-forged ingot. A. R. POWELL.

Electrodes for use in electric arc-welding and the fusion deposition of metals. QUASI-ARC Co., LTD., and A. P. STROHMENGER (B.P. 363,815, 12.12.30).—The electrodes are covered with a paste of starch or cellulose containing 20% of $K_2Cr_2O_7$ or $Na_2Cr_2O_7$ and then with blue asbestos string. The gases evolved during use as welding rods serve to protect the welds from oxidation. A. R. POWELL.

Sintering furnace. Conc. of ore slimes.—See I. **Purifying $ZnCl_2$.** S from water-gases.—See VII. **Resistance material. Articles of Pb or its alloys.**—See XI. **Metal-coated articles.**—See XIII.

XI.—ELECTROTECHNICS.

Pitch coke [for electrodes].—See II. Titration of food dyes.—See IV. Electrolytic H_2 and O_2 .—See VII. Moisture control of ceramic pastes.—See VIII. Inclusions in Fe or steel. Electrolytic Fe. Thermocouples.—See X. Titration of oil acidity.—See XII. Properties of stretched rubber.—See XIV. Electrolyser for soils. Conductivity of suspensions.—See XVI.

See also A., Feb., 110, Emission from oxide-coated filaments. Conductivity of compressed metallic powders. 112, Magnetic Au-Fe alloys. 126, Acid standard cells of the unsaturated type. Photoelectric cells. 129, Electrolysis of Be compounds. Electrodeposition of metals from anhyd. NH_3 . 130, Production of NO in the electric arc. 133, Ferromagnetic Fe_2O_3 . 137, High-temp. laboratory furnace. Improved quartz Hg-vapour lamp. Double potentiometer for measuring temp. 138, Const.-pressure device for autoclaves.

PATENTS.

Luminous electric-discharge tubes. GEN. ELECTRIC CO., LTD., Assees. of PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 364,484, 12.5.31. Ger., 18.7.30).—The carbonate, *e.g.*, $MgCO_3$, from which CO_2 is evolved during operation of the tube, is mixed with Pr oxide, MnO_2 , or $KMnO_4$ so that the O_2 evolved when the tube becomes hot will prevent blackening of the envelope. J. S. G. THOMAS.

[Indirectly heated] thermionic cathodes of vacuum-electric tube devices. E. Y. ROBINSON, F. BARTON, and ASSOCIATED ELECTRICAL INDUSTRIES, LTD. (B.P. 364,136, 18.8.30).— $Al_2(NO_3)_6$, mixed if desired with compounds of Mg, Zr, or Si, is applied as a coating to a W filament and the whole treated with aq. NH_3 and then converted into Al_2O_3 by heating. The processes of coating and conversion into oxide may be repeated several times. [Stat. ref.]

J. S. G. THOMAS.

Photoelectric apparatus. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of K. T. BAINBRIDGE (B.P. 364,006, 26.8.30. U.S., 28.8.29).—The current-carrying capacity of the discharge device arranged in an envelope devoid of gas is increased by secondary emission phenomena, *e.g.*, by multiple reflection of light between photosensitive cathodes. J. S. G. THOMAS.

[Restoring deteriorated] photoelectric cells [with thin-film cathodes]. GEN. ELECTRIC CO., LTD., and N. R. CAMPBELL (B.P. 363,877, 4.2.31).—The cells are heated at a temp. about that to which they were heated during manufacture. J. S. G. THOMAS.

Depolarisers for primary electric cells or batteries. EVER READY CO. (GREAT BRITAIN), LTD., and R. H. SPETTIGUE (B.P. 364,476, 30.4.31).—A mixture composed of SiO_2 gel (0.05 pt.), MnO_2 (4 pts.), and graphite (1 pt.) is used. J. S. G. THOMAS.

Material of high electrical resistance and method of using same. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 363,615, 12.6.30. Ger., 20.7.29).—High-resistance material suitable for use in glow-discharge appa-

ratus and electrical dust precipitators is composed of finely-divided metal distributed throughout non-conducting material to form a fine metal-sponge network. Thus, *e.g.*, a mixture of Ag and glass dusts is sintered under pressure and fused. Alternatively a porous ceramic mass impregnated with aq. $Cu(NO_3)_2$ is heated in H_2 . [Stat. ref.] J. S. G. THOMAS.

[Improving the contact points of] non-metallic [electrical] resistors. CARBORUNDUM CO., LTD., Assees. of A. H. HEYROTH and G. C. COX (B.P. 365,081, 15.7.30. U.S., 29.7.29).—The ends of a SiC resistor rod are dipped into a bath of Si, Ni, or Al, or an alloy of these, and heated gently at first to allow the liquid to impregnate the resistor and then more strongly to volatilise excess Si etc. Alternatively, a quantity of the impregnating material (Si) previously applied to the ends is fused by striking an electric arc between the resistor and another electrode. L. A. COLES.

Manufacture of electric cables and articles made of lead or lead alloys. PIRELLI-GEN. CABLE WORKS, LTD., and G. MARTINEZ (B.P. 364,289, 3.12.30).—A reinforcing layer, *e.g.*, of Cu, steel, or brass, covered with an alloy which readily amalgamates with Pb or Pb alloys, is arranged between the surfaces of materials made of Pb or its alloys to be welded or soldered together. J. S. G. THOMAS.

Electrically separating suspended particles from gases. LODGE-COTTRELL, LTD. FROM METALGES, A.-G. (B.P. 364,491, 21.5.31).—Gas entering the hollow space enclosed by a perforated collecting electrode passes through the perforations in a radial direction, *i.e.*, in the direction of the lines of force radiating from the axially-arranged discharge electrode. J. S. G. THOMAS.

Incandescence electric [projection] lamps. WESTINGHOUSE LAMP CO., Assees. of C. F. LORENZ (B.P. 365,740, 13.12.30. U.S., 20.12.29).

Electric rare-gas illuminating lamps. A. LEDERER (B.P. 364,088, 25.7.30. Austr., 30.7.29).

[Obliquely-tipped] carbons for electric lamps. E. and O. CONRADT (C. CONRADT) (B.P. 365,816, 3.2.31).

Insulation of electric cells, more particularly cells of electric batteries of the high-tension type. E. HIBOU (B.P. 364,240, 6.11.30).

Sp.-gr. indicator for liquids.—See I. Treatment of animal fibres. Paper for condensers.—See V. Purifying electrolytic O_2 and H_2 . Removing acids from H_2O_2 solutions.—See VII. Glass insulators.—See VIII. [Metal-]melting furnaces. Treatment of steel etc. Coating of metals. Electrodes for welding etc.—See X. Detergent etc.—See XII. Metal-coated articles.—See XIII.

XII.—FATS; OILS; WAXES.

Potentiometric titration of acidity in oils. R. R. RALSTON, C. H. FELLOWS, and K. S. WYATT (Ind. Eng. Chem. [Anal.], 1932, 4, 109—110).—The acidity of oils may be determined by potentiometric titration of the oil (dissolved in isoamyl alcohol saturated with LiCl) with 0.025—0.05N-KOH (in isoamyl alcohol), using a Pt-C

electrode couple. A procedure is described, which enables 0.5-g. samples to be used. The existence of more than one inflexion in the titration curves is believed to indicate the presence of acids having widely different dissociation consts.

E. S. HEDGES.

Twitchell reagent. XII. Relation between the kind of oil and the emulsifying power of the reagent. K. NISHIZAWA and T. INOUE (Kolloid-Z., 1932, 58, 225—229).—The general behaviour of different emulsifiers is similar, but the emulsifying power varies also with the kind of oil. The presence of 0.1N-H₂SO₄ generally increases the emulsifying power. Although the general physical and chemical properties of the oil appear to be important, no general relation has been found between the emulsification of a given oil and its sap., I, or acid val., its viscosity, or m.p.

E. S. HEDGES.

Twitchell reagent. VI. Fat-splitting power of a new commercial reagent, Divulson-D, and darkening of the fatty acids obtained therewith. VII. Darkening of the fatty acids in the Twitchell process. K. NISHIZAWA and K. HUZIMOTO (Tech. Rep. Tôhoku, 1931, 10, 107—124, 125—145).—Full German translations of pts. VI (B., 1931, 399; cf. B., 1932, 29) and VII (B., 1931, 499) of the Japanese papers are given.

E. LEWKOWITSCH.

Fatty acids of chicken fat and other edible fats. J. GROSSFELD (Z. Unters. Lebensm., 1931, 62, 553—566).—Chicken fat has (mean) sap. val. 195.2, I val. (Hanus) 69.9—78.2, thiocyanogen val. (cf. B., 1929, 401, 441) 62.7, n_D^{20} 1.4615. These are compared with similar data for goose, beef, mutton, and horse fats and for lard and hardened arachis oil, the (mean) % of solid fatty acids (Pb-salt method) being 27.83, 40.73—53.05, 54.22, 29.30, 38.10, and 69.9, respectively, and for chicken fat $21.36 \pm 1.01\%$. *iso*Oleic acid occurs only in beef and mutton fats (0.91—5.35%) and in hardened arachis oil (25.2%), and no saturated fatty acid above stearic was detected in lard, chicken, goose, beef, or mutton fat. Calculation of palmitic acid from the sap. val. gives a higher figure than that from the mol. wt. of the solid fatty acids, probably on account of the presence of medium fatty acids lower in mol. wt. than palmitic acid. A new method of determination of lower volatile fatty acids is described in which a solution of the saponified fat is pptd. with 6% MgSO₄ and the filtrate distilled with H₃PO₄, the distillate being titrated with 0.1N-NaOH to phenolphthalein. Chicken fat yielded butyric 0.03, octoic 0.05, and nonoic acid 0.06%, and other fats < 0.5% of the total acids. Kaufmann's method (*loc. cit.*) indicates the presence of oleic as well as linoleic acid in all cases. The fatty acids of chicken fat comprise stearic 7.4—8.5, palmitic 17.5—18.4, oleic 52.5, linoleic 17.0; lower volatile acids 0.1% (unsaponifiable matter 0.28, glycerol residue 4.4%). HNO₂ or ultra-violet light bleaches the colouring matter of chicken fat, the intensity of which may be measured by comparison against K₂Cr₂O₇; the SbCl₃ test (Carr and Price) gave a light-blue colour.

J. GRANT.

Cacao butter. J. VISSER (Chem. Weekblad, 1932, 29, 40—42).—A note on its definitions, sources, varieties, and adulterants.

H. F. GILLBE.

Rancidity [of fats]. H. O. TRIEBOLD (Cereal Chem., 1931, 8, 518—532).—Theories as to the mode of development of oxidative rancidity are discussed. By means of the spectrophotometer determinations were made of the % of light transmitted by the coloured solutions produced by the Kreis reaction on a prime steam lard after absorption of 0, 10, 50, 100, 200, 300, and 400 c.c. of O₂, respectively, per 100 g. of fat. As the greatest absorption occurred at wave-lengths of 530—570 m μ , the mean of five readings (at 530, 540, 550, 560, and 570 m μ) was taken as indicating a definite O₂ absorption by the fat. The logarithm of the reciprocal of the % of light transmitted is related almost linearly to the amount of O₂ absorbed. Wheat oil inhibits in a measure the oxidative deterioration of the shortening in baked goods.

W. J. BOYD.

Sol and gel forms of silica in relation to the analysis of soaps containing sodium silicate. K. SCHERINGA (Chem. Weekblad, 1932, 29, 37—38).—Fatty acid may be determined in soap containing Na₂SiO₃ by dissolving the soap in a large vol. of H₂O and acidifying rapidly; if much alkali be present the soap solution should be added gradually to an excess of 4N-acid. For determination of SiO₂ the soap is completely carbonised, H₂O added, and the solution over-neutralised with 4N-acid, neutralised, and finally rendered just acid to Me-orange; the whole of the SiO₂ may then be collected on a filter.

H. F. GILLBE.

Analysis of potash-fish-oil soaps used for emulsification [in insecticides]. A. W. CRESSMAN, H. H. BLISS, and A. J. HAAS, JUN. (J. Econ. Entom., 1931, 24, 1252—1255).—Modified methods of recording analyses are suggested to accentuate characteristics important in emulsification. The efficiency of emulsification depends on the molar concn. of soap at the oil-water interface and results expressed in mols. per g. of soap are preferred to those in wt.-%. Free alkali is determined since it depresses the hydrolysis of the soap and increases the molar concn. of the solution. Soaps of unsaturated fatty acids are usually more effective emulsifiers than those of saturated acids. Analytical data should include mean mol. wt. of acids, free alkali, and I val., in addition to customary data.

A. G. POLLARD.

Potentiometric determination of acid value of linseed oil and its free fatty acids. Comparison of results obtained in different solvents by indicator and potentiometer methods. B. P. CALDWELL and J. MATTIELLO (Ind. Eng. Chem. [Anal.], 1932, 4, 52—56).—The acid vals. of raw and heat-bodied linseed oil have been determined by potentiometric titration, and results are given for the potentiometric titration of stearic, oleic, and linolenic acids and the mixed free fatty acids of linseed oil in EtOH, BuOH, 95% amyl alcohol, and C₆H₆-EtOH (1:1) as solvents. The last-named mixed solvent is preferred. The quinhydrone electrode is preferred to the H electrode for this work. Dissociation consts. were not obtained, but the acids are arranged in the following order of decreasing strength: stearic > oleic > mixed linseed fatty acids > linolenic.

E. S. HEDGES.

Refractometric examination of edible oils. Index of Tunisian olive oils. R. MARCILLE (Ann. Falsif.,

1931, 24, 465—471).—The Abbé refractometer has been used to determine the n_D . The vals. at 20° are: olive (Tunis) 1.4689—1.4704, olive (Spanish) 1.4691, sesame 1.4732, arachis 1.4700—1.4711, cotton-seed 1.4705—1.4715, maize 1.4733—1.4744, sunflower 1.4736—1.4751, soya 1.4747—1.4765, paraffin 1.4648, camphorated olive (adulterated with paraffin) 1.4670, olive 1.4700, olive with 10 and 20% of camphor 1.4696 and 1.4697, respectively. With the exception of cereal oils n_D increases with the age of the oil and with acidity. When the acidity exceeds 2%, a correction of 0.0001 is required per 1% of acidity. Olive oils from Tunis fall into two distinct categories according to the locality of growth; those from the north have average val. 1.4698, and those from the central regions 1.4693.

H. J. DOWDEN.

Oil from Malayan *Aleurites montana* and the properties of Hong Kong oil. T. H. BARRY (Analyst, 1932, 57, 85—93).—Oil expressed or extracted from *A. montana* from the Malay States had abnormally low refractive indices (n_D^{20} 1.5135 and 1.5128, respectively). The oils gave abnormal results by the Browne polymerisation test (15½ and 18½ min.), though the extracted oil isomerised very readily. It is suggested that all genuine oils from *A. montana* require longer to polymerise than those from *A. Fordii*, and that abnormal figures obtained from several samples of Hong Kong oil are due to the presence of the former oil.

T. McLACHLAN.

Emulsive capacity of sulphonated oils. Miscibility of sulphonated oils and neutral oils. R. HART (Ind. Eng. Chem. [Anal.], 1932, 4, 119—120).—The emulsive capacity is defined as the max. amount of olive oil which, when mixed with 100 g. of the sulphonated oil, gives an emulsion of definite stability. An experimental procedure for determining emulsive capacity is outlined. Sulphonated oils are more miscible with mineral than with fatty oils, and both fatty and mineral oils are less miscible with sulphonated castor oil than with sulphonated olive oil. Sulphonated oils are better emulsifiers for mineral than for fatty oils; acid sulphonated castor oil is inferior to acid sulphonated olive oil. Complete neutralisation of a sulphonated oil decreases its miscibility with neutral oils. Neutral sulphonated oil, with the proper addition of EtOH, is a better emulsifier for mineral oils, but poorer for fatty oils, than is the acid oil.

E. S. HEDGES.

Bleaching earths.—See I. **Coalification of waxes.**—See II. **Butter and butter fat. Butter analysis. Palm-kernel and coconut cake feeds. Soya-bean meal. Pilchard oil for calf feeding.**—See XIX.

See also A., Feb., 141, **Pyrogenic decomp. of highly unsaturated hydrocarbons.** 184, **Fatty acids from oil of cantharis.** 200, **Vitamin-A from fish oils.** 203, **Carotene in palm oil.** **Wax constituents of (203) the apple cuticle, (204) forage grasses.** **Seed of *Euphorbia marginata*.**

PATENTS.

Production of buoyant soaps. K. JUNGMAAN (B.P. 362,754, 3.2.31. Czechoslov., 6.2.30).—Metal powders (Al, Zn), hydrides, iodised Mg, etc. are incor-

porated in the soap jelly, in order to generate H₂ during manufacture, which is retained in the soap on setting.

E. LEWKOWITSCH.

Treatment of fat- and oil-bearing seeds [soya beans]. G. P. TUSSAUD (B.P. 364,309, 16.12.30).—The seeds, especially soya beans or cereal grains, are soaked in H₂O and treated with SO₂ and steam at 110—140° until the seeds are dried out to less than the original natural H₂O content; the flavour and dietetic and keeping properties are improved.

E. LEWKOWITSCH.

Production of thickened oil with addition of wood oil. J. SOMMER and S. J. RALPH (B.P. 362,545, 17.9.30).—Plant is illustrated whereby tung oil is heated to a temp. near that of polymerisation (but below the gelatinisation point) and introduced and dispersed (e.g., through a perforated coil or nozzles) into hot polymerised (bodied) linseed oil during agitation.

E. LEWKOWITSCH.

[Continuous] process and apparatus for thickening wood oil. R. WIRTH, O. R. WOLF, and A. KÖHLER (B.P. 364,516, 27.7.31).—The oil is passed continuously through a heating element and discharged at 260—280° into a collecting vessel, fitted with a stirrer, from which it is removed at the same rate, the time during which the oil remains in the collector to thicken being controlled by adjustment of the level of the liquid; after discharge the oil is rapidly chilled.

E. LEWKOWITSCH.

Detergent, shampoo, or sheep dip. E. V. HAYES-GRATZE (B.P. 363,653, 12.9.30).—A neutralised sulphonated (vegetable) oil is ionised by electrolysis or by the action of a high-tension and/or high-frequency discharge, until the p_H of a 1:10 aq. solution is 7.5—8, and then diluted with H₂O, with or without addition of 1—3% of H₂O₂.

E. LEWKOWITSCH.

Cleansing compound. W. PEEL (B.P. 363,794, 25.11.30).—A compound for removing oil stains from textiles is prepared by emulsifying C₆H₆ hydrocarbons, and/or their chlorinated derivatives, and/or light petroleum with an approx. equal wt. of Castile soap in 40—50% aq. solution at about 90°, and adding a small amount of a weak acid (H₃BO₃, AcOH) after cooling.

E. LEWKOWITSCH.

Manufacture of high-quality wax-like substances. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 362,632, 15.11.30).—The higher fatty acids obtained by the oxidation of montan wax (cf. B.P. 305,552; B., 1929, 273), preferably in the form of Et or Me esters, are reduced to alcohols ("montanol") which are esterified with higher fatty acids (e.g., up to C₂₅); excess acid if present may be converted into soaps.

E. LEWKOWITSCH.

Wetting etc. agents.—See III. **Artificial silk from viscose. Threads from cellulose derivatives. Treatment of animal fibres. Cleansing rags etc.**—See V. **Polish. Resinous products.**—See XIV.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Use of slate powder in the paint and allied industries. H. RABATE (Peint., Pig., Ver., 1932, 9, 1—10).—An account is given of the sources of slate powder and of its uses in the manufacture of paints,

wood fillers, and jointing mastics. Analyses and formulæ are included. S. MARKS.

Antiseptic value of phenols and chlorinated phenols in paints. P. PORTIER and A. KLING (Peint., Fig., Ver., 1932, 9, 14—16).—The bacteria *Bacillus coli* and *Staphylococcus* are destroyed by surfaces coated with paints containing chlorinated phenols (actual compounds and quantity not stated). Continued washing of such a surface with water does not reduce its antiseptic activity. S. MARKS.

Paint-testing and weather observation. W. VAN WÜLLEN-SCHOLTEN (Farben-Ztg., 1931, 37, 126—127).—The importance of local weather conditions, particularly in the early stages of the life of a paint film, in determining durability is stressed. S. S. WOOLF.

Metal stains on white-painted dwellings. H. A. GARDNER (Amer. Paint & Varnish Manufrs.' Assoc., Nov., 1931, Circ. No. 402, 392—395).—The degree of staining of a large number of white pigments and extenders when 10% FeCl₃ solution is dropped on them is tabulated; basic materials showed most discoloration. The bearing of these results on the development of brown stains on white paint in contact with unpainted metal is stressed. S. S. WOOLF.

Evaluation of condition, particularly degree of rusting, of exposed paints. R. KEMPF (Farben-Ztg., 1931, 37, 161—163).—The various attributes to be taken into account in examining exposure panels are detailed and classified under optical, mechanical, and structural properties and presence of foreign substances. The question of assessing the total degree of failure when various grades of failure are visible on the same film is stressed. In the case of recording rusting, 6 exponential stages are defined (*i.e.*, 0, 1, 3, 10, 30, and 100% of rust) and these are illustrated by graphs and photographs. The method of proportionally assessing a plate by calculation from the extent of the various rust stages is described. S. S. WOOLF.

Prevention of hazard in paint application. STILLER (Farben-Ztg., 1931, 37, 124—126, 164—165, 199—200).—A general account is given of the risks that have arisen during the gradual replacement of brush application by spraying, flowing, and dipping, and the introduction of cellulose lacquers etc. in place of the older oil paints. Pb poisoning, fire and explosion risks, toxicity of solvents, etc. are discussed at length. Improvement in spray apparatus and in the removal of spray mist etc. are directions in which progress is required and developments on these lines are described. S. S. WOOLF.

Pigment and vehicle. H. GERET (Farben-Ztg., 1932, 37, 586—587).—An abstract is given of a comprehensive series of experiments on white and coloured pigments and extenders, including settling determinations, sp. gr., particle size and shape, MeOH-vapour adsorption, and heat of wetting by different liquids. The character of a pigment is defined by physico-chemical surface properties (oxophilic or carbophilic, heat of wetting, etc.), surface extent (particle size etc.), and surface intensity (roughness, active spots, degree of adsorption). S. S. WOOLF.

Black printing inks. T. HAMBURGER (Chem.-Ztg., 1931, 55, 962—963).—Information on both staining power and composition of black inks is necessary for the evaluation of such inks. The former is determined by mixing the ink with a known amount of white pigment and oleic acid, together, if desired, with a trace of a wetting agent, the resultant shade being compared with standards. The analysis into black pigment, dye, and vehicle by successive extraction with light petrol-um and COME₂ is described in detail, and typical results are discussed. S. S. WOOLF.

Plasticisers for cellulose acetate. H. WOLFF and I. RABINOWICZ (Farben-Ztg., 1932, 37, 585—586).—The loss of wt. at 80° of a known plasticiser for cellulose acetate and a new material, "Hydropalat CY," alone and when incorporated into cellulose acetate films was observed. It is shown that conclusions as to the behaviour of a plasticiser in a lacquer film should not be drawn from observations on the plasticiser alone. The variation of tensile strength and elasticity of such films with age was also determined. S. S. WOOLF.

Rapid exposure tests on finishes. H. A. GARDNER (Ind. Eng. Chem. [Anal.], 1932, 4, 94—97).—Outdoor and accelerated laboratory exposure tests for paints, varnishes, etc. are described. In the salt water-air test the low-cook phenolic and modified phenolic resin-long oil varnishes generally give satisfactory results as compared with the other coatings. Rapid failure was shown by S-treated varnishes, Pb tungstate liquids, boiled linseed oil, gloss oil, and a supposedly H₂O-resistant rubber varnish. E. S. HEDGES.

Practical tests of varnishes in the tropics. ARNOLD (Chem.-Ztg., 1932, 56, 55).—Tests of varnishes of known composition on woods in use in Brazil over a period of 12 months are reported. All showed stickiness over considerable periods, though this may be due to the high tannic acid content of the wood used, or to resin inclusions. Varnishes containing synthetic resins do not give this unfavourable result. Co compounds are not suitable as driers: a Pb-Mn drier is better; for white enamels lithopone or Ti-white are better than Zn-white. White enamels generally become yellow in time and an ivory-coloured white is preferred. Bitumen should be used for insulating paint; the low softening point of coal-tar pitch preps. renders them unsuitable. C. IRWIN.

Shellac. O. M. OLSEN (Ind. Eng. Chem. [Anal.], 1932, 4, 47—48).—Methods are outlined for determining the content of EtOH-insol. matter, wax, and ash, and sp. gr. of shellac. Results are given. E. S. HEDGES.

Nature and constitution of shellac. IV. Saponification number. W. F. WHITMORE and H. WEINBERGER [with W. H. GARDNER] (Ind. Eng. Chem. [Anal.], 1932, 4, 48—51; cf. B., 1929, 1046).—Refluxing shellac with 0.5N-KOH in EtOH for 30 min. does not give complete saponification; refluxing should be continued for at least 2 hr. Aq. HCl may be used for back-titrating with thymol-blue as external indicator. Saponification takes place in 3 stages, indicating that shellac contains 2 types of ester linkings. Potentiometric titration curves of the saponified shellac solutions show that all the constituent OH-acids are weakly dissociated in

EtOH, since their K salts can be titrated completely with HCl. Sap. vals. of different varieties of shellac are given; when these vals. are computed on a basis of true resin content, fairly uniform ester vals. are obtained.

E. S. HEDGES.

Resins of phenolphthalein series. M. D. ZELINSKY and B. W. MAKOROV (Ind. Eng. Chem., 1932, 24, 63—64).—A variety of di-esters and -ethers of phenolphthalein are prepared; with the exception of the di-(*m*-methylbenzyl) ether, all exist in cryst. forms which are converted by fusion or by evaporation of their conc. solutions into colourless resins that crystallise with a greater or less degree of ease on keeping. The dibenzyl ether is isolated in a cryst. form, m.p. 153°, with difficulty, but the di-(*m*-methylbenzyl) ether forms a completely stable, hard, colourless resin that shows no tendency to crystallise and is exceptionally resistant to acids and alkalis. Both are monomeric in C_6H_6 and are intermediate in composition between the lactonic and OH-acid forms.

H. A. PIGGOTT.

Coalification of resins.—See II.

See also A., Feb., 139, Determining particle size by photo-recorder. 140, Ultramarines. 150, Study of $CH_2O-CO(NH_2)_2$ condensation.

PATENTS.

Manufacture of composite calcium sulphate-titanium oxide pigments. TITANIUM PIGMENT CO., INC., Assees. of W. F. WASHBURN and L. AAGAARD (B.P. 364,112, 23.9.30. U.S., 24.9.29).—The $CaSO_4$ used is pptd. by adding a Ca compound, e.g., $Ca(OH)_2$, to conc. (e.g., 75%) H_2SO_4 .

L. A. COLES.

Varnishing of printed surfaces. MASA GES.M.B.H. ZUR HERSTELLUNG KÜNSTL. OBERFLÄCHEN (B.P. 364,520, 19.8.31. Ger., 28.8.30).—A fine-mist coat of nitro-cellulose varnish is applied under pressure (e.g., 4 atm.) over ordinary oleograph inks, when a full protective coat of the varnish may be at once applied without "lifting" or otherwise attacking the inks.

S. S. WOOLF.

Preparation of polishing composition. J. A. J. COLLARD (B.P. 363,036, 15.10.30).—A mixture of fats or oils, e.g., linseed oil (3—10 pts.), synthetic or natural resins, e.g., pine resin, copal (2—8 pts.), and synthetic or natural waxes, e.g., carnauba, lignite, montan (2—8 pts.), is heated to 110—160° and a suspension of a fine grit, e.g., kieselguhr, "Tripoli" (25—40 pts.), in white spirit or turpentine (40—60 pts.) is added.

S. S. WOOLF.

Resinous compositions. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of H. J. CAMERON and E. H. JACKSON (B.P. 364,304, 12.12.30. U.S., 13.12.29).—A phenol, e.g., cresol (1 mol.), is heated rapidly to about 100° with an aldehyde, e.g., paraformaldehyde (2 mols.), in the presence of an ethanolamine, e.g., triethanolamine, a dispersing agent, e.g., KBr, and $CO(NH_2)_2$ and guanidine carbonate, and the mixture is rapidly chilled to room temp. The resinous condensation product, permanently liquid at room temp., may be "cured" by heat.

S. S. WOOLF.

Moulded [resinous] mixtures and articles. H. WADE. From BAKELITE CORP. (B.P. 363,694, 18.6.30).—

Retarders, e.g., natural resins, xylydines, toluidines, benzidine, $NHPh_2$, are added to $PhOH$ (3 mols.)—hexamethylenetetramine (1 mol.) mixtures at an intermediate stage of condensation and the reaction is continued. Fillers, e.g., mica, may be incorporated with the potentially reactive resin.

S. S. WOOLF.

Coating of moulded articles with metal. J. GLOSTER, LTD., and L. J. GLOSTER (B.P. 363,432, 27.9. and 7.10.30).—Metal is electrodeposited on articles moulded from a mixture comprising a $PhOH-CH_2O$ type of synthetic resin and a finely-divided electrically conductive material, e.g., C.

S. S. WOOLF.

Manufacture of synthetic resins. IMPERIAL CHEM. INDUSTRIES, LTD. From E. I. DU PONT DE NEMOURS & Co. (B.P. 364,400, 19.2.31).—A polymerised vinyl chloroacetate is caused to react with an alkali compound of an org. acid, phenol, or alcohol, e.g., Na salts of drying-oil acids, KOPh, Na glyceroxide, Na Bu phthalate, in presence of a non-reactive org. solvent, preferably an acetic ester, in which the polymeride is sol., e.g., β -ethoxyethyl acetate, cyclohexyl acetate. Pale resins free from reactive Cl are obtained.

S. S. WOOLF.

Manufacture of adhesives, coating and binding agents. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 363,348, 7.8.30).—Butadiene is polymerised by heating under increased pressure, in the absence of H_2O and hydrazine derivatives and in the presence of O_2 (pure or admixed) and a practically anhyd. org. solvent capable of dissolving the polymerisation product, e.g., tetrahydrostyrene, C_6H_6 , PhMe.

S. S. WOOLF.

Artificial resinous substances. IMPERIAL CHEM. INDUSTRIES, LTD., W. BAIRD, and R. HILL (B.P. 362,972, 10.9.30).—The acidity of polybasic acid-polyhydric alcohol condensation products is reduced by heating them with alkyl or aralkyl halides, e.g., PrI , CH_2PhCl .

S. S. WOOLF.

Manufacture of artificial materials from polyhydric alcohols and polybasic acids or their anhydrides. SCHERING-KAHLBAUM A.-G. (B.P. 363,029, 9.10.30. Ger., 22.10.29).—The condensation is effected in the presence of high-boiling indifferent liquids (fatty oils excluded), e.g., tetra- or deca-hydronaphthalene, cymene, which are later distilled off together with H_2O of reaction; softening, filling, and colouring materials may be incorporated if desired.

S. S. WOOLF.

Water-soluble or -dispersible synthetic resins and solutions or dispersions thereof. W. W. TRIGGS. From ELLIS-FOSTER CO. (B.P. 363,997, 13.6.30).— H_2O -insol. or -indispersible polyhydric alcohol-polybasic acid resins, modified if desired with monobasic acids or other agents, e.g., phthalic anhydride-cottonseed-glycerol resins, are treated with org. or inorg. bases, e.g., aq. NH_3 , KOH, MgO , Na_2S , hexamethylenetetramine, NH_2Ph , in the presence of H_2O , sufficient base being used to neutralise excess acidity without saponifying or deresinifying the resin. Such dispersions are suitable for the treatment of paper. [Stat. ref.]

S. S. WOOLF.

Manufacture of resinous condensation products. ARMSTRONG CORK CO., Assees. of E. J. PIEPER (B.P. 363,345, 12.6.30. U.S., 17.6.29).—Oxidised ("blown") fatty acids obtained from drying or semi-drying oils are

added to polyhydric alcohol-polybasic acid mixtures before or during condensation. S. S. WOOLF.

Manufacture of [inlaid] linoleum. ARMSTRONG CORK Co., Asses. of J. A. PHELAN (B.P. 366,375, 4.5.31. U.S., 9.6.30).

Chlorinated Ph₂.—See III. Azo pigments.—See IV. Cleansing rags etc.—See V. BaSO₄ granules.—See VII. Foundry cores.—See X. Coating of leather.—See XV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

X-Ray study of rubber structure. M. F. ACKEN, W. E. SINGER, and W. P. DAVEY (Ind. Eng. Chem., 1932, 24, 54—57).—When rubber is stretched a time interval elapses before the rubber shows evidence of cryst. character as revealed by X-ray diffraction. Of several possible explanations the most probable assumes the presence of mol. chains (either spiral or tangled) the ends of which are aligned in the direction of stretch, but of which the middle portions become straightened only by displacement of unfavourably orientated mols. or portions of mols. D. F. TWISS.

Electrical properties of stretched rubber. A. GEMANT (Z. Physik, 1931, 73, 526—537).—The electrical technical efficiency of rubber can be considerably increased by stretching. A. B. D. CASSIE.

PATENTS.

Production of rubber-latex concentrates. REVERTEX, LTD. FROM METALLGES. A.-G. (B.P. 363,872, 29.1.31).—The optimum proportion of non-volatile alkali, preferably KOH, to ensure stability of conc. latex obtained by evaporation in the presence of protective colloids and free alkali, and to prevent deleterious alteration of the latex or its constituents, is such that after diluting about 60-fold, titration in the presence of an indicator with an inversion point near p_H 7, e.g., bromothymol-blue, reveals a free alkali content of 80—180 milli-equivs., preferably 115—125 milli-equivs., per kg. of concentrate. A rapid method for ascertaining that the correct proportion of alkali is present is to moisten a piece of filter-paper with 0.1% aq. alizarin and to apply, preferably from the other side of the paper, a sample of the concentrate; the alizarin colour should change to orange or reddish-brown. D. F. TWISS.

Formers or moulds for production of articles of or containing organic materials [such as rubber]. DUNLOP RUBBER Co., LTD., D. F. TWISS, and E. A. MURPHY (B.P. 363,660, 22.9.30).—The formers (etc.) are coated with an aq. dispersion or solution of rubber, and movement of the applied layer relative to its supporting surface is induced so as to cause localised irregularities, such as a wrinkled or grained marking, which are then rendered permanent by concurrent or subsequent solidification. The coating may further be vulcanised to a soft or hard condition. The former thus coated can be used directly for the production of articles, or it may serve as a master mould for the production of replicas, e.g., in clay or metal. D. F. TWISS.

Vulcanisation of rubber. RUBBER SERVICE LABS. Co., Asses. of R. L. SIBLEY (B.P. 363,644, 20.9.30. U.S.,

2.10.29).—An accelerator of the type 2:4-dinitrophenyl-2-benzthiazyl sulphide is used together with a Schiff's base (or an aliphatic aldehyde derivative thereof) preferably derived from an aliphatic aldehyde below C₅, e.g., produced from a reaction mixture containing but-aldehyde (3 mols.) and NH₂Ph (1 mol.). D. F. TWISS.

Cleansing rags etc.—See V.

XV.—LEATHER; GLUE.

X-Ray studies of animal skin. I. Structure of calfskin as indicated by molybdenum radiation. K. G. CHESLEY, H. V. ANDERSON, and E. R. THEIS (J. Amer. Leather Chem. Assoc., 1932, 27, 12—22).—X-Ray photographs have been prepared showing the diffraction patterns of fresh, dried, salt-cured, soaked, limed, and bated calfskins, respectively. The rings "C" and "I" corresponded to reticular distances of 2.8 Å. and 12—13 Å. The halo "L" varied in dried samples from 4.3 to 4.7 Å., but was const. in moist samples at 3.3 Å. The salt crystals were under strain in the salt-cured skin. No fundamental change in the collagen was detected during the processes of curing, soaking, liming, and bating. The collagen in calf- and goat-skin was structurally identical. The photographs are not explained by the structures hitherto proposed for gelatin and collagen.

D. WOODROFFE.

Use of amines in liming steer hides. E. K. MOORE, J. H. HIGHBERGER, and F. O'FLAHERTY (J. Amer. Leather Chem. Assoc., 1932, 27, 2—11. Cf. B., 1927, 971; 1928, 681).—Soaked raw-hide pieces were treated with a saturated solution of Ca(OH)₂ made 0.08, 0.16, 0.32M with NH₂Me, NHMe₂ or NMe₃. No harmful effect, abnormal swelling effect, or hair damage was produced by the amines. The hair was loosened more quickly by Ca(OH)₂ and NH₂Me or NHMe₂ than by Ca(OH)₂ alone, but the hair-loosening was unaffected by NMe₃. The hair was slightly impaired by high concns. of NHMe₂, which were greater than those necessary for good acceleration of the hair-loosening.

D. WOODROFFE.

Function of acido-groups in chrome[tanned] leather. K. H. GUSTAVSON (J. Amer. Leather Chem. Assoc., 1931, 26, 635—667).—The order of decreasing amounts of protein-bound SO₄" removed from Cr-tanned leather by various treatments was: diffusion neutralisation method, Palitzsch buffer solution p_H 8.0, 4% solution of pyridine. The removal of SO₄" from Cr-tanned leather by pyridine was only slightly affected by the presence of Na₂SO₄. Neither the total SO₄" content of the original leather, the vol. of pyridine solution used, nor the number of extractions affected the acidity of the Cr complex in the leather (cf. Merrill, B., 1928, 496). 25% of the Cr-bound SO₄" in a neutralised Cr leather was replaced by Cl' by treatment with 2M-NaCl. "Blue" and fat-liquored Cr-tanned leathers, which had been once treated with a pyridine solution, were unaltered by a second treatment with the base, but 30% of the SO₄" in the fat-liquored leather was removed by degreasing and subsequent treatment with pyridine. The acido-groups in the Cr complex in Cr-tanned leather are removed by treatment with vegetable tannins. Acido-groups cannot be

replaced by pyridine since the latter requires a secondary valency, whereas the acido-groups are attached to the Cr by a primary valency. Pyridine may be held by zwitterionic SO_4 groups, or by acido-groups in general. The amount of such complexly held pyridine is governed by the nature of the sulphato-groups and ultimately by the nature of the tanning agent employed and the subsequent history of the tanned leather. It is usually obtained with dry tanning agents or solutions which have undergone prolonged boiling. D. WOODROFFE.

Effect of temperature on determination of water-soluble matter in leather. H. B. MERRILL and C. BENRUD (Ind. Eng. Chem. [Anal.], 1932, 4, 66—67).—The rate of extraction of H_2O -sol. matter from leather at 20° is considerably greater than at 10° , but rise of temp. above 20° has little further effect.

E. S. HEDGES.

Tanning-substance balance in the extraction of pine bark. E. BĚLAVSKY and G. WANEK (Gerber, 1931, 57, 11—13; Chem. Zentr., 1931, ii, 366).—In extraction for 10 days the amount of tanning substance extracted plus that remaining in the bark is greater than that originally determined by the international official method. The following analytical method is proposed: the finely-ground bark is steeped for 12 hr., then heated at 80° , and after 25 min. 100 c.c. are withdrawn. Another 100 c.c. are withdrawn at 90° , and the remainder at 98 – 100° . The extraction is limited to 4 hr. and 1 litre. For the analysis of the residue the material is finely divided and extracted without previous drying. The original and modified vals. were: fresh bark 15,306, 16,319; extract 14,323, 14,232; residue 2358, 2152 kg. Hence the chief loss is in the residue; “undetermined losses” were not observed.

A. A. ELDRIDGE.

See also A., Feb., 122, **Adsorption of HCl by cowhide.**

PATENTS.

Coating of leather. IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 365,338, 30.1.31. U.S., 31.1.30).—A composition comprising a cellulose derivative (nitrate or acetate), a wax of animal, vegetable, insect, mineral, or synthetic origin, a plasticiser, a pigment, a solvent for the cellulose derivative, and a diluent (e.g., EtOH), is worked into the leather. L. A. COLES.

Production of glue from bones. A.-G. F. CHEM. PRODUKTE, VORM. H. SCHEIDEMANDEL (B.P. 363,888, 13.2.31. Ger., 13.2.30).—The bones (degreased, if desired) are ground, treated with sufficient acid to remove 20–50% of the $\text{Ca}_3(\text{PO}_4)_2$, and extracted several times with boiling H_2O . A residue of glue in the bones is extracted by pressure. D. WOODROFFE.

Adhesive mountants. A. COHEN (B.P. 363,710, 19.9.30).—A tissue is coated on both sides with substances having adhesive properties developed, respectively, by heat (e.g., shellac or sandarac solution) and moisture (glue, casein); or these substances may be applied in separate tissues. D. WOODROFFE.

Wetting etc. agents.—See III. **Cleaning animal hair. Impregnating leather. Fibrous substances impermeable to fluids.**—See VI.

XVI.—AGRICULTURE.

“Single value” soil properties. **Significance of certain soil constants.** VI. **Changes produced in a soil by exposure to high temperatures.** VII. **Moisture equivalent and some related quantities.** J. R. H. CUTTS (J. Agric. Sci., 1932, 22, 200—202, 203—211; cf. B., 1930, 875).—VI. The “loss on ignition” of soils was not altered by temp. $> 600^\circ$. The loss of wt. of heated soils is ascribed to (a) removal of free and interstitial H_2O up to 100° , (b) destruction of org. colloids from 100° to 250° , and (c) destruction of inorg. colloids above 250° .

VII. Relationships between the moisture equiv. “sticky point,” loss on ignition, and the mechanical analysis of soil are examined. Except in alkaline and saline soils, determinations of the moisture equiv. do not amplify information obtained from the other data. From the sp. gr., moisture and xylene eqivs. of a soil its imbibitional H_2O content may be calc.

A. G. POLLARD.

Laws of soil colloidal behaviour. VII. **Proteins and proteinated complexes.** S. MATTSO (Soil Sci., 1932, 33, 41—72; cf. B., 1932, 73).—The general principles governing the electrokinetic and chemical properties of sesquioxide complexes previously examined are shown to apply equally to similar protein complexes.

A. G. POLLARD.

Relationships between the fine material of soils and their physical characteristics. G. J. BOUYOUCOS (Soil Sci., 1932, 33, 27—39).—Close correlation is shown to exist between the “crumbling point,” flow point, and upper elastic limit of soils and the proportion of “total colloids,” clay, and fine clay as determined by the hydrometer method. The use of this method for the indirect determinations of physical characteristics of soils is discussed.

A. G. POLLARD.

Influence of hydrogen-ion concentration on the decomposition of soil organic matter by hydrogen peroxide. J. S. HOSKING (J. Agric. Sci., 1932, 22, 92—100).—A portion of the org. matter of the soil is oxidised by H_2O_2 irrespective of the soil reaction, but the decomp. of the remainder is a definite function of the $[\text{H}^+]$, being low in alkaline soils and *vice versa*. A mathematical relationship is established. The proportion of the org. matter oxidised is also a function of the clay content of the soil. Results of mechanical analyses are more accurate for soils which are acidified prior to H_2O_2 treatment, although the actual degree of dispersion is not appreciably affected. A. G. POLLARD.

Soil nitrogen. B. DIRKS (Landw. Jahrb., 1931, 74, 535—551).—The N status of soils is not in all cases indicated by its C:N ratio. The latter is affected by soil reaction, tending to widen in acid conditions. In alkaline soils the relationship between N status and C:N ratio is closer. The N-free org. matter is decomposed more slowly and the N matter more rapidly in acid than in alkaline soils. The humus produced from N matter is similar, whether produced under neutral or acid conditions. During decomp. N losses occur in acid (but not in alkaline) soils to an extent which depends on $[\text{H}^+]$. N fertilisers are fully utilised only in neutral and alkaline soils. A. G. POLLARD.

Soil "sickness." A. I. ACHROMEIKO (Landw. Jahrb., 1931, 74, 713—727).—In sand and soil cultures, a second sowing of oats and wheat in containers in which the roots of the first crop remained resulted in considerably decreased yields even where moderate applications of fertiliser were made. Large additions of fertiliser to the second crop produced normal yields. The decomp. of the roots of the first crop reduces the proportion of sol. N and P present. This is ascribed in part to biological absorption. No evidence was obtained of the presence of toxic org. material. A. G. POLLARD.

Nitrogen relationships in muck soils. B. D. WILSON and G. R. TOWNSEND (Cornell Univ. Agric. Exp. Sta. Mem., 1931, No. 137, 14 pp.).—Relationships between the NO_3^- content and acidity in these soils depended on the sampling depth adopted. Seasonal fluctuations in reaction depended on the extent of drying out, rather than on the activity of nitrification. Biological decomp. of org. matter did not prevent the accumulation of considerable amounts of NO_3^- . Incorporation of timothy or clover hay depressed nitrification, the effect being more marked in cultivated than in virgin areas, and more extensive and persistent in the case of timothy than with clover. A. G. POLLARD.

Carbon and nitrogen cycles in soil. IV. Natural and artificial humic acids. M. M. S. DU TOIT and H. J. PAGE (J. Agric. Sci., 1932, 22, 115—125; cf. B., 1930, 875).—Comparison is made of humic acids obtained from soil and peat, from wheat straw by the "Adco" process, and artificial humus preps. from cellulose, glucose, sucrose, glycine, furfuraldehyde, quinol, and lignin. The fractions sol. in NaOH, reprecipitated by HCl and insol. in EtOH behaved as colloidal acids in electrometric titration in all cases except those from sucrose and furfuraldehyde. Preliminary observations of humus formation from furfuraldehyde and from the interaction of glycine and glucose are recorded. A. G. POLLARD.

The soil versus the solution method as a means of studying bacterial activity in soil. J. E. GREAVES and H. C. PULLEY (J. Agric. Res., 1931, 43, 905—917).—The ammonifying, nitrifying, and N-fixing organisms withstand 2% of Na_2SO_4 , Na_2CO_3 , or NaCl for long periods. W. G. EGGLETON.

Influence of the form of combination of iron and manganese on the catalytic power of normal and sour soils. E. PFEIL (Z. Pflanz. Düng., 1932, 23A, 129—139).—The catalytic power of normal soils is greater than that of sour soils. In the latter there is a greater tendency for the dissolution of Mn, which is catalytically active in the form of oxide, hydroxide, and carbonate, but inactive in sol. compounds. Moreover, the total Mn content of acid soils may be diminished by leaching. Soil reaction influences catalytic power only through its effect on Fe and Mn compounds. The decreased activity of ignited soils is attributed to the conversion of hydroxides into the less active oxides. A. G. POLLARD.

Analysis of factors contributing to the determination of saturation capacity in some tropical soil types. P. E. TURNER (J. Agric. Sci., 1932, 22, 72—91).—The saturation capacity of soils is closely related

to their contents of clay and org. matter, but not to that of fine silt. The correlation in tropical soils is significantly greater in types having smaller proportions of these constituents. The increase in saturation capacity resulting from an increase of 1% of org. matter averages 1.508 ± 0.375 mg.-equiv.%, for clay 0.245 ± 0.035 , and for silt 0.05 approx. The equiv. wts. of org. matter, clay, and silt may be calc. by means of retrogression coeffs. The saturation capacity of a soil may be represented as $\alpha + \beta x_1 + \gamma x_2$, where x_1 and x_2 are the clay and org. matter contents, respectively, and α , β , and γ the consts. characteristic of the soil type. Among inorg. constituents the capacity for base exchange declines rapidly when the particle size becomes > 0.02 mm. A relationship is established between org. matter and clay contents. Where clay:org. matter is > 20 the org. matter may exist entirely as surface films on inorg. particles. A. G. POLLARD.

Rapid methods of examining soils. I. Measurements of rolling weights. R. K. SCHOFIELD and G. W. S. BLAIR (J. Agric. Sci., 1932, 22, 135—144).—The heaviness of a soil is measured by determining the wt. required just to cause the elongation of a small cylinder of moist soil when rolled between a wooden and a lightly-ground glass plate. The soil sample is sieved through a 100-mesh sieve and must contain an appropriate H_2O content to prevent adhesion to the plates during rolling. The crit. rolling wt. is a fundamental soil characteristic and is closely related to the drawbar pull in ploughing tests, and to the clay content in soils of any one type. Wide divergences in the rolling wt.-clay relationship appear in soils of different types. A. G. POLLARD.

Seedling method and new chemical methods for examination of soils. H. NEUBAUER (Ernähr. Pflanze, 1931, 27, 505—511).—The significance of possible changes in the availability of K and P in soils during crop growth is examined in relation to the seedling method. In a number of soils examined the initial amount of available P practically equalled the residual amount after cropping + that removed by the crop. In the case of K an increase in the net amount frequently appeared during cropping, presumably as the result of root-solvent action. Differences in results of the seedling method and the newer chemical-extraction methods for determining available P are attributed to inherent differences in the nature of the extractive process and also to the fact that chemical methods remove relatively large amounts of bases (notably Ca, Mg) which affect the solubility equilibrium of PO_4^{3-} in the soil. A. G. POLLARD.

Dispersion procedure used in the hydrometer method for making mechanical analyses of soils. G. J. BOUYOCOS (Soil Sci., 1932, 33, 21—26).—Dispersion of soils in H_2O by a mechanical stirrer working at $> 10,000$ r.p.m. for 10 min. (hydrometer method) was, in general, the same as that by 16 hrs. shaking (A.O.A.C. method). Pretreatment with H_2O_2 did not affect the results of mechanical analyses except on soils containing much undecomposed org. matter. Stirring for > 5 —10 min. tended to break down sandy particles. The rapidity with which hydrometric analyses may be carried out cannot be attributed to incomplete dispersion. A. G. POLLARD.

Rapid determination of the nitrogen requirement of soils by chemical analysis. A. NĚMEC and A. KOPPOVÁ (Z. Pflanz. Düng., 1932, 23A, 140—148).—Soils containing < 75 mg. N per kg. as $\text{NO}_3^- + \text{NH}_4^+$ (0.5N-NaCl extract) normally require N fertilisers. Analyses must be made with freshly air-dried samples.
A. G. POLLARD.

Determination of the magnesia requirement of soils by means of *Aspergillus niger*. H. NIKLAS, H. POSCHENRIEDER, and A. FREY (Ernähr. Pflanze, 1931, 27, 465—470).—A relationship exists between the yield of mycelium and the Mg content of the nutrient, but in soils of different types the growth rate is primarily controlled by the amount of CaCO_3 present. The wt. of mycelium produced is more closely related to the exchangeable than to the total Mg content of soils.
A. G. POLLARD.

Measurement of electrical conductivity of aqueous soil suspensions and its use in soil fertility studies. A. SEN (J. Agric. Sci., 1932, 22, 212—234).—Seasonal changes in the conductivity of soil suspensions in poor, unmanured soils are small. In manured soils reduced conductivity is associated with plant growth only when the latter is particularly rapid. In the course of a few months added fertilisers are washed down to soil depths below those of customary sampling. Storage of a soil suspension (1:5) at 25° for 7 days results in an increased sp. conductivity ("7-days' increase") which is related to cultural treatment. The 7-days' increase declines slightly after the application of inorg. fertilisers, but normally rises considerably following treatment with easily decomposable org. manures. In very rich soils (e.g., those of high NO_3^- content) the 7-days' increase may be negative. The val. is not appreciably affected by fallowing, but ploughing produces changes as a result of bringing subsoil to the surface. The 7-days' increase is a measure of microbiological activity tending to produce sol. nutrients, and when thus used as an index of fertility, soil samples should be taken before, or at least some time after, manuring, from uncropped soil and examined in the fresh condition. In permanent grassland, changes are definitely seasonal and comparisons must be made in corresponding seasons.
A. G. POLLARD.

Determination of the nutrient content of soils by means of their electrical conductivity. A. MARQUARDT (Mitt. Lab. Preuss. Geol. Landanst., 1931, 14, 32; Proc. Internat. Soc. Soil Sci., 1931, 6, 157—158).—Nutrient contents of soils cannot be determined by conductivity measurements, either with d.c. or a.c.
A. G. POLLARD.

Three-compartment electro-dialysis apparatus [for soil examination]. A. LÖDDESÖL (J. Amer. Soc. Agron., 1932, 24, 74—81).—Apparatus is described for the examination of exchangeable bases in soils.
A. G. POLLARD.

Is the soil type homogeneous with respect to its fertiliser needs? T. L. LYON (J. Amer. Soc. Agron., 1932, 24, 58—71).—Fertiliser trials over a number of years show that tests in a single locality are insufficient to indicate the fertiliser requirements of an entire soil type.
A. G. POLLARD.

Action of artificial fertilisers, especially of ammonium sulphate, on soil organisms. W.

SCHWARTZ and W. MÜLLER (Arch. Mikrobiol., 1931, 2, 620—638).—Heterotrophic soil organisms are very resistant to high concns. of $(\text{NH}_4)_2\text{SO}_4$. The activity of nitrite bacteria is reduced by the presence of > 1% of $(\text{NH}_4)_2\text{SO}_4$ in media. *Azotobacter* produces max. growth with 1% $(\text{NH}_4)_2\text{SO}_4$, but is still very active in 5% solutions. In soils, 2.5—5% of $(\text{NH}_4)_2\text{SO}_4$ does not affect the rate of nitrification appreciably. No differences in the effects of various forms of N fertilisers on the growth of soil organisms were observed, except in the case of $(\text{NH}_4)_2\text{SO}_4$, which is especially favourable to nitrifying bacteria.
A. G. POLLARD.

Nitrates and ammonium salts as sources of nitrogen for higher plants at constant p_{H} . III. K. PRIRSCHLE (Planta [Z. wiss. Biol.], 1931, 14, 583—676; cf. B., 1931, 1022).—With strongly acid and with alkaline nutrients in sand and water cultures, all plants examined produced better growth with N supplied as NO_3^- than as NH_4^+ . Under conditions more comparable with those of a normal soil, both sources of N are tolerated. NH_4^+ is preferentially utilised within a narrow range of p_{H} , although even in this range NO_3^- produces moderately good growth. The optimum $[\text{H}^+]$ for plant growth is not an abs. val., but is to a large extent dependent on nutritional conditions. For many plants the optimum is within p_{H} 5.5—6.5, and in this range differences in the utilisation of NO_3^- and NH_4^+ are minimal. The double optimum shown by the growth curves of many plants is not a characteristic property, but depends on nutritional factors which are affected only indirectly by $[\text{H}^+]$. No uniform relationship exists between the intake of N, P, and K by plants and the $[\text{H}^+]$ or growth rate. In general, the % of N and P in plants reaches a max. at p_{H} 7 and the K content at p_{H} 5.0. The intake of P is increased when N is supplied as NH_4^+ and the K intake is greater when NO_3^- is used, differences in the case of P being greater in acid zones and those of K in alkaline regions. The total-intake ratio P:K:N varies considerably with different plants, with p_{H} , and with the nature of the N source. A mixture of NO_3^- , NH_4^+ , and urea produces better growth than NO_3^- alone.
A. G. POLLARD.

Reciprocal effects of nitrogen, phosphorus, and potassium as related to the absorption of these elements by plants. W. THOMAS (Soil Sci., 1932, 33, 1—20).—Ion antagonism and the effects of $[\text{H}^+]$ on the selective intake of ions by plant roots are discussed. An extensive bibliography is appended.
A. G. POLLARD.

Determination of phosphoric acid in fertilisers. L. GISIGER (Landw. Jahrb., 1931, 73, 221—246).—Differences between results of the molybdate method and the various citrate-magnesia mixture pptn. methods are attributed to the co-pptn. of $\text{Ca}_3(\text{PO}_4)_2$ in the latter case. By increasing the proportion of NH_4^+ citrate solution used to 100 c.c. for 50 c.c. (= 1 g.) of phosphate solution, results which agree with those of the molybdate method are obtained. The latter is not appreciably affected by the amount of Ca present.
A. G. POLLARD.

Microbiology of farmyard-manure decomposition in soil. III. Decomposition of the cells of micro-organisms. H. L. JENSEN (J. Agric. Sci.,

1932, 22, 1—25; cf. B., 1931, 556).—Decomp. of microbial substance in sand and soil cultures indicates that only a portion (19—61%) of its N is easily nitrifiable. The remainder consists of alkali-insol. and very resistant substances comparable with the "ulmin" or "humin" of soils, together with, in some cases, material resembling α -humus. Of the org. N of manure 18—25% is of the α -humus type. During the decomp. of manure in soil, up to 25% of its total N may be utilised by micro-organisms to produce cell substances. Where the C:N ratio is wide, available N from the soil is utilised for this purpose. The cycle of operations tends to produce in soil an accumulation of resistant nitrogenous material which is not identical with fungal chitin.

A. G. POLLARD.

Active charcoal as an accessory factor in the manuring of plants. II. S. HOLYŃSKI (Przemysl Chem., 1932, 16, 4—8).—The better growth of plants in sand containing activated C in addition to the usual inorg. salts is due to binding of H_2O by C, with consequent increase in concn. of salts and reduction of heat loss by evaporation.

R. TRUSZKOWSKI.

Ensilage of minced potatoes. G. RUSCHMANN and G. GRÄF (Landw. Jahrb., 1931, 74, 633—664).—Conditions necessary for the production of a good silage from minced potatoes are examined. The tendency to frothing is minimised by maintaining conditions favourable to lactic fermentation. Addition of molasses accelerates lactic acid formation, particularly in the case of sugar-free potatoes. Addition of 2—3% of peat serves to absorb drainage liquor.

A. G. POLLARD.

Nutritive value of pasture. VIII. Influence of intensive fertilising on the yield and composition of good permanent pasture (seasons 1 and 2). H. E. WOODMAN and E. J. UNDERWOOD (J. Agric. Sci., 1932, 22, 26—72; cf. B., 1931, 646).—Under a system of monthly cuttings the dry-matter yields from good pasture receiving $CaCO_3$, superphosphate, K_2SO_4 , and farmyard manure averaged 90% of those similarly manured but with the addition of $(NH_4)_2SO_4$. The effect of $(NH_4)_2SO_4$ is apparent chiefly in the early season when growth is very rapid. A small increase in the crude protein content of the grass occurs in the first season of the $(NH_4)_2SO_4$ treatment. The fibre content of the herbage is affected more by weather conditions than by manuring. Dressings of $(NH_4)_2SO_4$ reduced the Ca content of the grasses, more particularly in the second season, did not materially affect the P and Na contents, and increased the K content only during the last month of the season. The increased N content of the crop receiving N, as compared with basal fertiliser only, averaged in the 2-year period 26% of that added.

A. G. POLLARD.

Production of crude protein, fat, and husk in oats. A. MIX (Landw. Jahrb., 1931, 73, 795—840; Chem. Zentr., 1931, ii, 926).—By increasing the N and total fertilisation the husk content is diminished and the protein content increased; complete fertilisation slightly increases the fat content. H_3PO_4 increases the protein and husk. K increases the fat, Ca the protein; K + N increases the protein and husk but diminishes the fat, whilst K + P diminishes the yield but increases

the protein and fat. Heavy soils yield grain rich in protein. The protein content of individual varieties is a transmitted characteristic. The fat content is influenced greatly by the weather, but little by the habitat.

A. A. ELDRIDGE.

Influence of the nature and quantity of potash dressings on the growth of varieties of beetroots, and formation of the valuable constituents of the roots. M. LEIBRANDT (Landw. Jahrb., 1931, 74, 587—632).—The use of K manures improves the growth of beetroots, particularly in the early stages, and induces the formation of enlarged vascular systems, lowered lignification, and an increased ratio of storage:conducting tissues. Dry-matter production and the development of the vascular system are closely related, the effect of K being to increase the entry of H_2O into the roots and lower the dry-matter content. In general, K increases the root:leaf ratio in large-leaved, and decreases that of small-leaved, varieties. The quality of the roots (based on dry matter and sugar contents) was improved in certain cases by applications of K Mg sulphate and of 40% potash salts, but more generally was diminished. Any beneficial effects in this respect are attributed to the Na or Mg salts present rather than to the actual K fertilisers.

A. G. POLLARD.

Factors affecting the composition of dry lime-sulphur solutions. D. E. BULLIS (J. Econ. Entom., 1931, 24, 1247—1252).—Solutions prepared by adding the requisite amount of H_2O and shaking at intervals during 20 hr. had a higher polysulphide-S content than those obtained by stirring with successive small quantities of water and decanting. With increasing periods of contact with H_2O (to 96 hr.), dissolution of the S sludge proceeds slowly yielding solutions of increasing polysulphide-S and "thiosulphate" content. The latter, determined normally by I titration, may consist, partly or wholly, of $CaSO_3$ produced by decomp. of CaS_2O_3 . Similar, but more pronounced, changes in composition result from raising the temp. of dissolution. At 95° practically all the sludge dissolves. With decreasing proportions of H_2O the sludge increases in amount and contains substances other than S.

A. G. POLLARD.

Use of sulphur in the control of red leaf blight. G. L. KOTTUR and S. S. MARALIHALLI (Agric. and Live Stock, India, 1931, 1, 638—641).—Successful control of the red leaf mite was obtained by S dusting. Yields were markedly increased and no leaf injury occurred in American varieties. In Asiatic species (which are not, however, attacked by leaf blight) S dusting at flowering caused a serious fall of flowers and leaves.

A. G. POLLARD.

Insecticidal control of the European maize borer. C. H. BATCHELDER and D. D. QUESTEL (J. Econ. Entom., 1931, 24, 1152—1167).—Control by spraying during the early stages of infestation is examined. A suspension of Pb arsenate in a 1% paraffin oil emulsion gave best results. The success of this prep. is attributed to its low surface tension and consequent ease of penetration into interfoliar spaces. No other contact insecticides examined (pyrethrum, nicotine, etc.) were effective.

A. G. POLLARD.

Field tests in Texas of insecticides for control of the cotton bollworm. R. W. MORELAND and F. F.

BIBBY (J. Econ. Entom., 1931, 24, 1173—1181).—Results of 3 seasons' trials show cotton yield increases following dusting with Ca arsenate. Fluosilicates gave negative results. A. G. POLLARD.

Four years' experiments with chemically treated codling moth bands. L. F. STEINER and G. E. MARSHALL (J. Econ. Entom., 1931, 24, 1146—1151).—Successful control was obtained by the use of corrugated paper bands impregnated with a solution of β -naphthol (1 lb.) in red engine oil ($1\frac{1}{2}$ pints), or α -naphthylamine (1 lb.) in engine oil ($1\frac{1}{2}$ pints) containing paraffin wax ($\frac{1}{2}$ lb.). Both materials prevent the emergence of larvae and have no injurious effects on the trees.

A. G. POLLARD.

Copper carbonate, lead arsenate, and other compounds [insecticides] against the Mediterranean fruit fly in Florida. R. L. MILLER and O. C. MCBRIDE (J. Econ. Entom., 1931, 24, 1119—1131).—The toxicity of Cu carbonate used in sugar solutions was almost as great as that of Pb arsenate. Other Cu compounds were highly toxic. Low temp. and concn., and also alkalinity of the mixtures, reduced their efficiency. Cu carbonate does not injure citrus trees, whereas Pb arsenate causes some leaf fall and reduces the citric acid content of maturing fruit. A. G. POLLARD.

Spraying and dusting cucumbers for control of downy mildew. G. F. WEBER (Florida Agric. Exp. Sta. Bull., 1931, No. 230, 58 pp.).—Bordeaux mixture (2-4-50) applied as a spray or dust, Cu carbonate, and Cu stearate proved satisfactory. The efficiency of Bordeaux mixture was not increased by the use of Ca caseinate spreader. Various forms of S were detrimental to the development of the plants. A. G. POLLARD.

Adhesive strength of arsenical insecticides. I. A. PARFENJEV (J. Econ. Entom., 1931, 24, 1261—1263).—The adhesive power of dusts is measured by dusting known wts. on to polished metal sheets which are placed in an inclined frame and tapped by an automatic hammer. Dust removed is collected and weighed. Leaves may be attached to the plates and examined similarly. Concordant results with commercial preps. are recorded. A. G. POLLARD.

Use of wetting or degumming agents in the removal of spray residues from apples. H. C. MCLEAN and A. L. WEBER (J. Econ. Entom., 1931, 24, 1255—1261).—Addition of textile wetting or degumming agents to HCl solutions used for cleaning sprayed apples accelerates the process and gives satisfactory results even where Pb arsenate-oil combinations have been used. A. G. POLLARD.

Green manuring of rubber. R. K. S. MURRAY (Trop. Agric., 1931, 77, 257—276).

Green manuring of tea, coffee, and cacao. T. H. HOLLAND (Trop. Agric., 1931, 77, 197—218).

Citrate-insol. PO_4''' in phosphates. Solubilisation of $Ca_3(PO_4)_2$.—See VII. Fish-oil soaps as insecticides.—See XII.

See also A., Feb., 141, Soils from Argolis. Reactions of horizons in tropical soils. 196, Effect of soil bacteria on growth of root-nodule bacteria. 196—7, Diphtheria toxin.

PATENTS.

Cyananides. Fertilisers.—See VII. **Cellular blocks etc.**—See IX. **Sheep dip.**—See XII.

XVII.—SUGARS; STARCHES; GUMS.

Teatini process [for raw sugar juice] in the light of present knowledge, communications received, and factory results in 1930—31. D. TEATINI (Bull. Assoc. Chim. Sucr., 1931, 48, 423—432, 433—490; cf. B., 1931, 736, 1114, 1152).—After a synopsis of 33 known processes for purifying raw sugar juices, the action of SO_2 in such processes is discussed. The use of SO_2 in the Teatini process differs from raw-juice sulphitation in that its aim is not to produce $CaSO_3$ or change the p_H val., but to supplement and stabilise the flocculation produced by the CaO . This is most effectively accomplished by liquid SO_2 introduced into the juice as rapidly as possible, so that a high "instantaneous concentration" is attained, resulting in greater electrolytic and chemical activity than can be attained with gaseous SO_2 . The author replies to criticisms of the process by Claassen, Schander, and Spengler. The juices obtained are commonly more coloured than those from the usual process, but they darken less during evaporation and yield syrups lighter than those obtained after the ordinary process. To the claim that equally good purification of juices can be obtained by fractional liming, without SO_2 , the author replies that this is true only insofar as the initial liming is such as to attain the isoelectric point (B., 1931, 1114), and the resulting flocculation is stabilised either by removal of the flocculate or by some treatment equiv. to the use of SO_2 . Evidence of the satisfactory working of the process in practice includes a report on a detailed control of the process at Hougaerde factory by Smolenski, and data for prolonged runs in two factories in France and one in England. J. H. LANE.

Reduction in consumption of lime for purification of diffusion juice from beets. G. CAPELLE (Bull. Assoc. Chim. Sucr., 1931, 48, 508—512).—Recent work has shown that, as a rule, more CaO than necessary is employed. For the usual procedure 1.6%, calc. on beets, is sufficient in most cases. The author and F. Baerts (Sucrerie Belge, 1925—7) found that the total CaO consumption could be reduced to 0.6—1%, calc. on beets, by a preliminary defecation with 0.25% of CaO and separation of the flocculate before further liming and carbonatation. The juices thus obtained with 1% of total CaO were equal, in respect of purity quotient and contents of ash and Ca, to those obtained by the usual process, but they were rather more coloured than the latter. Very similar results have since been obtained by Spengler and Böttger (B., 1931, 736, 777). The Teatini process differs from this procedure only in not removing the first flocculate and in using SO_2 . To attain the greatest possible saving of CaO by such methods it may be found advisable to re-employ carbonatation ppts. as an aid to clarification, and perhaps to adopt carbonatation under pressure (B., 1931, 1114), which improves the texture of the ppt. J. H. LANE.

Action of different kieselguhr preparations (Hyflocel, Supercel, and purified German kieselguhr) on [beet] sugar juices. O. SPENGLER and S.

BÖTTGER (*Z. Ver. deut. Zucker-Ind.*, 1931, 81, 791—812).—Raw beet-sugar solutions of 15°, 50°, and 65° Brix were filtered at 90° and 70° through thin layers of each of the three filter aids investigated, on a Buchner funnel under const. vac. In all cases equal rates of filtration were obtained under similar conditions with the American Hyfocel and the German prepared kieselguhr 80 S. Supercel, owing to its finer structure, gave much lower rates of filtration, but rather more brilliant filtrates. None of the three had any detectable decolorising action on the solutions. The following analytical data were obtained for Supercel, Hyfocel, and 80 S, respectively: insol. in 1:1 HCl 98.38, 99.45, 98.80%; ($\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$) sol. in 1:1 HCl 0.15, 0.06, 0.17%; SiO_2 sol. in 1:1 HCl 0.02, 0.01, 0.01%; org. matter 0.57, 0.29, 0.18%. These filter aids are used to much greater advantage if a small quantity only is employed as a pre-formed layer (0.2 mm. thick) on the filter and a further quantity is suspended in the liquid, than if the same total quantity is employed as a pre-formed layer. J. H. LANE.

Beet marc. E. MÜLLER and J. PUCHERNA (*Z. Ver. deut. Zucker-Ind.*, 1931, 81, 841—843).—A method of investigating beet marc used by Kopecký (*B.*, 1932, 125) is almost identical with one described by the authors (*B.*, 1930, 75). J. H. LANE.

Determination of the affining values of raw [beet] sugars. K. ŠANDERA (*Z. Ver. deut. Zucker-Ind.*, 1931, 81, 844—848). O. SPENGLER (*Ibid.*, 849—850). K. ŠANDERA (*Ibid.*, 851).—I. A reply to Spengler and Grunow (*B.*, 1932, 41). The author's conductometric method (*B.*, 1928, 344; 1931, 900) takes less time and gives more information to the refiner than the Berlin method.

II. The Berlin method (*B.*, 1927, 730; 1928, 30; 1932, 75) is intended to show only whether a raw sugar will yield, by affination alone, a sugar suitable for direct consumption, and it is therefore concerned only with the colour and appearance of the affined product.

III. The superiority of the Berlin method for this purpose is admitted, but the suitability of a raw sugar for refining is better shown by the author's method.

J. H. LANE.

Detection of commercial invert sugar [in honey] by the lævulosin method. C. I. KRUISHEER (*Chem. Weekblad*, 1932, 29, 38—40).—A solution (200 c.c.) of the sample (25 g.) is boiled, yeast is added, and the whole digested for 3 days at 22—30°. After filtration the solution is neutralised (neutral-red) and conc. to 100 c.c., and 96% EtOH (200 c.c.) and saturated aq. $\text{Ba}(\text{OH})_2$ are added; the alkaline solution is filtered after 24 hr. and excess $\text{Ba}(\text{OH})_2$ pptd. by CO_2 . EtOH is removed by repeated evaporation to 25 c.c. and after diluting the solution to 100 c.c. two 50-c.c. samples are inverted, one for 10 min. and the other for 3 hr., with 5 c.c. of 9.5*N*-HCl at 68—70°. The difference between the fructose contents of the two solutions is due to lævulosin. Honey-cake may be dealt with similarly, after extracting with warm H_2O and centrifuging. Commercial glucose does not interfere, whereas its presence renders Fiehe's method inapplicable. Lævulosin is not formed in pure honey unless it is heated for about 24 hr.

H. F. GILLBE.

Iodometric determination of invert sugar in sugar-factory products. R. OFNER (*Z. Zuckerind. Czechoslov.*, 1932, 56, 249—255).—An alternative form of a recent method (*B.*, 1929, 832), in which the Cu_2O is determined directly instead of by difference, is described. After the reaction mixture has been boiled and cooled, 15 c.c. of *N*-HCl are added carefully down the side of the flask, and an excess of I solution is immediately run in from a burette, the liquid being shaken round after the first few c.c. have entered. The flask is then at once stoppered and shaken round at intervals, and after exactly 2 min. 5 c.c. of 0.5% starch solution are added and excess I is titrated. If 0.03*N*-I and $\text{Na}_2\text{S}_2\text{O}_3$ solutions are used, 1 c.c. of I reduced by the $\text{Cu}_2\text{O} \equiv 1$ mg. of invert sugar. The vol. of I is corrected by 0.1 c.c. for every g. of sucrose in the reaction mixture. To determine invert sugar in raw beet sugars, 153.6 c.c. of the clarified solution used for polarisation are de-lead with Na_2HPO_4 , made up to 200 c.c., shaken with 1 g. of Norit or similar C to adsorb reducing non-sugars, set aside for 15 min., and filtered; 50 c.c. of the filtrate are used for the determination, or an aliquot part with a complementary amount of H_2O . For beet molasses, a solution containing 26 g. with 2.5 g. of $\text{Pb}(\text{OAc})_2$ is made up to 200 c.c. and filtered, and 153.6 c.c. are de-lead with phosphate, shaken with 4 g. of Norit, set aside for 15 min., and filtered; 50 c.c. of the filtrate or diluted filtrate are used, but the reaction mixture is boiled for 7 min. instead of 5 min. With low-grade products a blank determination, without heating, is advised for the highest accuracy.

J. H. LANE.

Oxford process and sugar-factory technique. A. REJTHÁREK (*Z. Zuckerind. Czechoslov.*, 1931, 56, 197—202).—The methods of working employed at the Eynsham factory are described. The drying of the cossettes was a technical success but not economical, conditions of storage were unsatisfactory, and extraction of the sugar was very unsatisfactory and costly (cf. *B.*, 1931, 410). 4000 tons of the dried cossettes from Eynsham were taken over by the Wisington factory and worked for white sugar. They were of very irregular quality, and in part badly deteriorated. It was found best to produce a thin juice and apply the usual purification treatment. Even under these conditions the working of these dried cossettes was much more troublesome than that of fresh beets, but very good sugar was obtained. The Oxford process could be greatly improved, but only under exceptional conditions could it compete with the old process.

J. H. LANE.

Impurities in white sugar. IV. Determination of nitrogen. J. A. AMBLER and S. BYALL (*Ind. Eng. Chem. [Anal.]*, 1932, 4, 34—36; cf. *B.*, 1931, 1115).—Total N is determined by Kjeldahl digestion of the sugar followed by measurement of the NH_3 in the distillate by Nessler's method. Protein N is pptd. as tannate and then determined by the above method. $\alpha\text{-NH}_2$ -acids are determined by the ninhydrin method. NO_3' is reduced by Devarda's alloy to NH_3 , which is distilled and determined by Nessler's reagent. NO_2' is determined by Ilosvay's modification of Griess' colorimetric test.

E. S. HEDGES.

Commercial production of lævulose. II. Conversion of Jerusalem artichoke juices. J. W. EICHINGER, JUN., and J. H. MCGLUMPHY [with J. H. BUCHANAN and R. M. HIXON] (Ind. Eng. Chem., 1932, 24, 41—44; cf. B., 1932, 76).—On account of the buffering action of raw artichoke juice, its p_H is a function both of normality with regard to added acid and concn. of the juice; the velocity of conversion into fructose is a function of p_H and the nature of the added acid. The decomp. of fructose in 1 hr. at p_H 1.12 (H_2SO_4) and temp. up to 80° is negligible. Conditions are worked out for the complete conversion of any juice with 4—40% total solid content in 1 hr.

H. A. PIGGOTT.

Potato glucose. Its modern commercial production. A. E. WILLIAMS (Chem. Trade J., 1932, 90, 151—152, 181).

Pectic substances in retted flax.—See V. **Olier continuous diffusion. Measuring p_H values of diffusion juice. Determining sugar in red wines.**—See XVIII. **Fodders containing sugar. Heat of hydration of certain starches.**—See XIX.

See also A., Feb., 203, **New sugar in dextrorotatory honeys.** 206, **Determining reducing sugar in syrups etc.**

PATENTS.

Manufacture of sugar or similar products.

W. W. GROVES. From D. TEATINI (B.P. 362,911, 9.9.30).—In the centrifuging of massecuites, after removal of the mother-liquor, the sugar is cured by treatment with SO_2 dissolved in H_2O or mixed with a gas or vapour, in such amount that the p_H of the run-off, or mixture of mother-liquor and run-off, is not lowered too far for subsequent operations. Treatment with dissolved CaO or other alkali may precede that with SO_2 where otherwise sufficient SO_2 could not be used without lowering unduly the p_H of the run-off. Sulphites, bisulphites, carbonates, or CO_2 may be used instead of SO_2 , and the treatment may be applied before or during the removal of the mother-liquor.

J. H. LANE.

Manufacture of white sugar directly from cane-sugar juice. Y. INADA (B.P. 363,470, 12.11.30).—

Cane juice is treated with a solution of Ca saccharate, then carbonated or sulphited as usual, evaporated, and decolorised with C. A suitable saccharate solution containing about 10% CaO may be prepared from mixtures of carbonised org. matter and CaO obtained in the production of decolorising C, by slaking, stirring with 50% sugar solution, and filtering. J. H. LANE.

Treatment of gum hydrocarbons. F. F. VON

WILMOWSKY (U.S.P. 1,808,191, 2.6.31. Appl., 8.2.30).—*Sapotacea* gums freed from resins are segregated into physically distinct grades by fractional pptn. from petroleum solution or by fractional extraction with petroleum. The grades may be blended. The gums are cured by heating with active S at 60° or with flowers of S at up to 145°, preferably 130°, under pressure in a stream of inert gas, e.g., CO_2 or H_2 , in a jacketed vessel closed at the bottom by a cover movable in a vertical direction by scissor-like levers. D. K. MOORE.

XVIII.—FERMENTATION INDUSTRIES.

Olier continuous diffusion in distilleries. J.

PERARD (Bull. Assoc. Chim. Sucr., 1931, 48, 499—508).—A report on a month's technical control of an Olier diffuser (B., 1923, 673, 1088) producing beet juice for fermentation from rather inferior beets. The diffuser required only one operator, it had a capacity of 100 tons of cosettes per 24 hr., and yielded pulp exhausted to 0.09—0.15% of sugar with a draw-off of 130 litres/100 kg., compared with about 150 litres in most distillery diffusion plants. The liquor supplied was a 1:1 mixture of vinasses and H_2O at 70—80°. Heating of the diffuser, by injection of steam, consumed 70 kg. of steam per ton of beets with hand control, or 33 kg. with automatic control. The mechanical working of the apparatus was entirely satisfactory. The pulp is discharged from the top of the diffuser and there are no waste waters. The diffuser could probably be adapted to sugar-factory work, but for this the requirements in respect of purity of juice are very different from those in distilleries, and further investigations would be necessary.

J. H. LANE.

Continuous measurement of p_H values of diffusion juice by a photocolometric method. G. GRIMAUD (Bull. Assoc. Chim. Sucr., 1931, 48, 543—544).—A preliminary note on a proposed apparatus for regulating the acidification of juice from the Olier continuous diffuser in distilleries. (Cf. *supra*). J. H. LANE.

Importance of hydrogen-ion concentration in potato distilleries. J. DEHNICKE (Z. Spiritusind., 1932, 55, 2—3).—

In the yeast mashes prepared either by the lactic acid or H_2SO_4 method, the p_H of the ripe yeast varied little from that of the fresh yeast in spite of an increase in the acid content of 0.1—0.2°. With lactic acid yeast the average p_H vals. from 45 distilleries were 4.0 for the fresh and 3.9 for the ripe yeast. The corresponding vals. for H_2SO_4 yeast were 3.75 and 3.48, the lower vals. being due to the smaller amount of buffers present. Occasionally the vals. approximated to 3.1, but these were too low, and, owing to the presence of free H_2SO_4 , had an injurious effect on the yeast growth. The average p_H of the main saccharified mash was 5.36. This val. was decreased by 0.2 by the addition of the yeast and after a fermentation of 48 hr. fell to 4.56, approx. 0.5 of the decrease taking place in the first 24 hr. The mean p_H (4.4) of the final fermented wort varied little from that of the above mash, whilst that of the slop showed little difference at 4.37. Abnormal vals. shown by a few samples were due to seasonal variations in the potatoes and other raw materials.

C. RANKEN.

Consumption of heat in preparing absolute

alcohol. O. PAMPE (Z. Spiritusind., 1932, 55, 14—15).—In the production of abs. EtOH directly from the mash, the dehydration column is heated by the hot slop flowing from the mash column. The temp. of the slop falls from 102° to 92°, and sufficient heat is left to preheat the mash. The heat transferred to the dehydrating column is not lost and, apart from small losses, reappears in the H_2O from the condensers at 70° and is used to heat H_2O etc. The process is very economical of steam, and for a mash containing 10% of EtOH it

requires only 324.5 kg. of steam per hectolitre of abs. EtOH, of which 37.0 kg. are used to raise the mash from 70° to 90°, and 287.5 kg. for the distillation and dehydration of the heated mash. By the normal process, 400 and 430 kg. of steam per hectolitre of abs. EtOH were required for 94 wt.-% of raw spirits, and 460 kg. for 90 wt.-%.

C. RANKEN.

Methyl alcohol in alcoholic liquors. II. Removal of the methyl alcohol. III. Origin of the methyl alcohol. N. A. ESPINOSA (Anal. Assoc. Quím. Argentina, 1930, 18, 274—304; 1931, 19, 39—79. Cf. B., 1931, 608).—II. A rectifying apparatus and method of operation are described whereby from 55% fermentation spirit containing 9% MeOH a 70% yield of EtOH (96%) free from COMe₂, MeOH, and furfuraldehyde, and containing only traces of higher alcohols, may be obtained. Details are given of a rectifying plant operating on the Barbet principle, which from 4—5% EtOH containing MeOH 1.5%, aldehydes 0.7%, and various other impurities yields 88.9% of neutral potable EtOH (96—96.5%) and 10—12% of a less pure EtOH (90—92%), the total yield in 24 hr. being 2000 litres. Cazenave's modification of the Barbet principle is adversely criticised.

III. Previous work and hypotheses are reviewed. Analytical data are given for over 200 specimens of spirits and other alcoholic liquors, and are discussed in their relation to the problem of maintaining a low MeOH content during manufacture.

H. F. GILLBE.

Detection of fruit wine in grape wine according to Werder's sorbitol method. J. SCHINDLER and I. KOZAK (Z. Unters. Lebensm., 1931, 62, 647—652).—Numerous tests have been made, using a slightly modified form of the Werder-Jahr methods (B., 1929, 619; 1930, 787) sensitive for 10% mixtures. It is unnecessary to identify the sorbitol hexa-acetate from the m.p. since the crystals (which are pictured) are easily recognised under the microscope. Aggregates of large crystals are formed by seeding, and fine feathery or star-shaped groups by cooling, the individual crystals being monoclinic needles the end angles of which (110) are always very sharply defined and easily recognisable.

J. GRANT.

New sorbitol process for detection of fruit wine in grape wine. F. M. LITERSCHIED (Z. Unters. Lebensm., 1931, 62, 653—657).—The wine is first treated according to Werder's method (B., 1929, 619), and then evaporated and pptd. with 0.2 c.c. of *o*-chlorobenzaldehyde in the presence of 2 vols. of fuming HCl. The resulting sorbitol derivative when washed with MeOH has m.p. 175—210° (generally about 200°), whilst the corresponding mannitol compound (m.p. > 260°) separates less readily and may be obtained from the filtrate after removal of the former. The usual acetylation method may then be used as confirmation. Addition of < 2.5 vol.-% of decolorised apple wine was detectable.

J. GRANT.

Residual sugar in red wines. Its importance and determination. J. DUBAQUIÉ and G. DEBORDES (Ann. Falsif., 1931, 24, 477—484).—Wines often contain traces (2—10 g./litre) of fermentable sugar. Red wines need decolorising before titrimetric determination, and

the use of charcoal involves errors (e.g., 2—3 g. of C black with 50 c.c. of wine gave 2 g. of sugar instead of 3.2 g.). Determination of the sugar is best carried out by a modified Fehling's solution, the Cu₂O being separated with precautions against oxidation, and subsequently determined by the iodide method. The presence of > 2 g. of residual sugar in 1 litre of wine may lead to slow fermentation or bacterial decomp. during storage in barrels or bottles, with the production of high acidity.

H. J. DOWDEN.

Determination of lactic acid in wines. A. MICHEL (Ann. Falsif., 1931, 24, 471—474).—Bonifazi's method (B., 1926, 603), involving the isolation of the lactic and volatile acids as Ba salts in the presence of 75% EtOH, is subject to certain errors. Excess BaCl₂ is partly reduced during ashing and this causes an apparent increase in the alkalinity at the final stage. With wines rich in K salts this error is not so great, the KCl being more stable. Exact neutralisation is essential as any excess Ba(OH)₂ is sol. in EtOH. The modified method is as follows. To the sample (20 c.c.), neutralised with Ba(OH)₂ in slight excess, are added 2 c.c. of BaCl₂ (10%) and about 80 c.c. of 95% EtOH, and, after re-neutralising, the mixture is diluted to 110 c.c. After 3 hr. the liquid is filtered, the excess Ba is pptd. by bubbling CO₂, and the BaCO₃ removed by filtering. After evaporating most of the EtOH from 80 c.c. of the filtrate, 2 c.c. of *N*-K₂SO₄ are added and evaporation is continued to dryness. The ash is calcined and its alkalinity titrated with 0.2*N*-HCl and 0.2*N*-NaOH. Volatile acids are determined separately.

H. J. DOWDEN.

Determination of lactic acid in wines. J. H. FABRE and E. BRÉMOND (Ann. Falsif., 1931, 24, 474—477).—Möslinger's method as modified by Bonifazi and by Ferré (cf. B., 1926, 603; 1928, 383) gives satisfactory results. Samples with known additions of lactic acid gave vals. correct to 0.2 g./litre, and determination of volatile acids was not affected. A synthetic liquid corresponding to wine and containing 2.5 g. of lactic acid per litre gave 2.57—2.52 g./litre. Further additions of lactic acid gave parallel results. The errors are found to be < 0.32 g./litre.

H. J. DOWDEN.

Detection of benzoic, salicylic, and cinnamic acids and of the esters of *p*-hydroxybenzoic acid in wine. R. FISCHER and F. STAUDER (Z. Unters. Lebensm., 1931, 62, 658—666).—The conc. EtOH-free wine is extracted with a mixture of Et₂O and light petroleum. Several micro-sublimation methods are described for the removal of the above preservatives from the residue after evaporation of the solvents, and photomicrographs of the sublimates are pictured. Saccharin sublimes at 150—160° and the remainder at 60—100°; nipagin has different cryst. forms at 110° and 126°. The crystals may also be identified by their behaviour under the polarising microscope and by micro-reactions. BzOH gives prisms with 3% Cu(OAc)₂; the esters of *p*-hydroxybenzoic acid (nipagin, nipsol, and the Et ester) all give a red colour with Millon's reagent; cinnamic acid is detected by the formation with Ba(OH)₂ of prismatic crystals with pyramidal ends, and by oxidation in Na₂CO₃-KMnO₄ to PhCHO; saccharin is identified by taste and reaction with HgCl₂.

Fractional-sublimation methods are described for mixtures, 0.001% of any one substance being detectable in the wine. J. GRANT.

Determination of the acidity of wines. H. ASTRUC and P. AUBOUY (Ann. Chim. Analyt., 1932, [ii], 14, 1—5).—For ordinary and white wines slightly lower total acidity vals. are obtained using litmus paper than using phenolphthalein, the differences being somewhat greater in the case of certain hybrid wines.

W. J. BOYD.

Step-titration of wine vinegar. Determination of non-volatile acids. P. HIRSCH and O. DELP (Z. Unters. Lebensm., 1931, 62, 589—593).—The vinegar is steam-distilled with HCl to remove AcOH, and NaOH added to the residue (50 c.c.) till the p_H val. is 8.3 (phenolphthalein). The mixture is then titrated to p_H 2.0 (thymol-blue) with 0.25N-HCl in a titration colorimeter (t c.c.), the correction (s c.c.) for the presence of strong acid being found by adding the same amount of NaOH to 50 c.c. of H_2O , followed by 0.25N-HCl, to give p_H 8.3, and then titrating to p_H 2.0. Then the sum of the non-volatile acids (100% of the succinic, malic, and lactic acids and 97% of the tartaric acid) = ($t-s$). Any original colour should be obviated by a compensating solution, since active charcoal adsorbs the acids. Results (c.c. of 0.25N-HCl 100 c.c.) are: pure wine vinegar 22.0—27.2, "double" (50% mash) 16.4, ordinary (20% mash) 4.5—5.9, spirit vinegar and commercial essence vinegar 0—0.2, commercial wine vinegar 0.7—5.6 (cf. B., 1930, 482). J. GRANT.

Determination of fusel oil and aldehydes in alcoholic liquids. E. RUPPIN (Z. Unters. Lebensm., 1931, 62, 215—217).—Colours obtained in Zellner's colorimetric method for fusel oil and in the fuschin aldehyde determination should not be matched in a colorimeter, since different degrees of colour are not comparable; errors in the former case amount to 20%. Precautions for the distillation of alcoholic liquids are given; a 100—125-c.c. flask is used for fusel oil, with an outlet tube 7—8 mm. diam. inclined at 60°. For aldehydes the flask should be as small as possible and the apparatus filled with CO_2 . J. GRANT.

[Glass] apparatus for modern taste analysis of spirituous liquors. C. LUCKOW (Z. Unters. Lebensm., 1931, 62, 585—589).—The sample is distilled in the "birectificator" (cf. B., 1931, 859), an apparatus in which the vapour is twice rectified, the non-volatile portions being returned to the flask by means of a U-tube type of condenser. Eight 25-c.c. fractions (b.p. 78—100°) are collected from 240 c.c. of sample, the highly alcoholic fractions being diluted and tasted. A thin 50-c.c. pear-shaped type glass for the taste test is recommended. J. GRANT.

Diastase of honey.—See XIX.

PATENT.

Abs. EtOH and Ca(OH)₂.—See III.

XIX.—FOODS.

Comparative evaluation of yellow and white oats. T. BANIN (Landw. Versuchs-stat., 1931, 113, 121—199).—The wt. per 1000 grains was, in general,

higher for white oats than for yellow. In sieve tests, white oats had the greater proportion of large grains, and also correspondingly higher husk and crude-fibre contents. Differences in the protein and N-free extractive contents of various strains were considerable, and no clear differentiation between white and yellow varieties was possible. The tendency to high oil production was most marked among yellow oats. Differences in ash content were too small to be characteristic.

A. G. POLLARD.

Methods for testing cake flour. J. W. MONTZHEIMER (Cereal Chem., 1931, 8, 510—517).—The American Association of Cereal Chemists cake formula (see Blish, B., 1928, 463) is satisfactory for routine tests, but gives high tests on weak flours. A layer-cake formula is given which was found excellent, although the use of egg-yolks diminishes its utility for colour evaluation. A flour may respond better to one formula than to another. Short, patent, finely-ground flours produce the best cakes. The cake-making properties of flour are improved by the use of "Beta Chlora" as a bleaching agent. Viscosity tests are of little value for cake flours, but a granulation test would be useful. W. J. BOYD.

Heat of hydration of wheat flour and certain starches, including wheat, rice, and potato. C. A. WINKLER and W. F. GEDDES (Cereal Chem., 1931, 8, 455—475).—A method of determining the sp. heat of wheat flour and starches is described, the vals. found being, respectively, 0.397 g.-cal. and 0.44 g.-cal. per g. of dry matter. The heat of hydration of wheat flour varied from 18.3 g.-cal. per g. of dry matter for flour of 1.7% moisture content to 0.5 g.-cal./g. (for 16.3% moisture). The relation between moisture content and heat of hydration is not linear. Wide variations in protein content did not affect the heat of hydration, but variation in protein quality had a slight effect and heat-treatment lowered it slightly. Variation in the state of aggregation of the flour had no effect. Wheat starch from different flours had almost the same heat of hydration, this being slightly higher than that of wheat flour of the same moisture content. The heats of hydration of rice, wheat, and potato starches varied widely and increased in the order given. The large and small granules of potato starch had no significant difference in heat of hydration. The evolution of heat when H_2O is mixed with flour or starch is due to simple adsorption. The H_2O permeates the starch granule and is adsorbed on the surface of the micelles. W. J. BOYD.

Determining the ash content of the flour in self-rising and phosphated flours. C. B. GUSTAFSON (Cereal Chem., 1931, 8, 475—481).—The flour (20—25 g.) is suspended in CCl_4 , centrifuged, skimmed from the surface of the liquid, and air-dried overnight. Moisture and ash are determined as usual. With self-rising soft wheat flours the ash content found did not differ by >0.023% from that of the plain flour from which it had been prepared. Low results were obtained for self-rising hard wheat flours, but by reducing the speed of the centrifuge to 1100 r.p.m. and setting aside the mixture for 30 min. before skimming it, good results were obtained unless the ash content was >1%. Phosphated soft wheat flours gave low results even when a

speed of 800 r.p.m. and a longer period of keeping were adopted. Phosphated hard wheat flours gave better results which, however, were still low. By applying the corrections given for different grades of phosphated flour results within $\pm 0.02\%$ of the ash of the plain flour are obtained. W. J. BOYD.

Comparison of the utility of flour-protein extractions in distilled water and in inorganic salt solutions. R. H. HARRIS (Cereal Chem., 1931, 8, 496—509).—Pure H_2O was found to be as useful as inorg. salt solutions for determining protein extractability as an indication of baking strength. Loaf vol. appears to be significantly related to content of H_2O -insol. protein, but not to content of sol. protein. W. J. BOYD.

Comparison of composites and averages [of the component flours] with respect to baking quality. I. Pure samples of one variety. R. K. LARMOUR and S. F. BROCKINGTON (Canad. J. Res., 1931, 5, 491—500).—Either the composite or average loaf vol. may be used to predict the other with reasonable accuracy. P. G. MARSHALL.

Chemical analyses of some important baking ingredients. A. W. MEYER (Cereal Chem., 1931, 8, 482—495).—Methods of analysis of various baking ingredients (including yeast foods and flour improvers), and for detection and determination of various oxidising agents, are given. W. J. BOYD.

Buckwheat milling and its by-products. M. R. COE (U.S. Dept. Agric. Circ., 1931, No. 190, 11 pp.).—The distribution and milling processes of buckwheat are described, with full analytical data for the grain, flour, and by-products. E. B. HUGHES.

Colloid chemistry of gluten. IV. H. L. B. DE JONG and W. J. KLAAR (Cereal Chem., 1931, 8, 439—454; cf. B., 1931, 177).—The influence of varying concn. of KI and K_2SO_4 on the viscosities of acid gliadin sols was studied at varying p_H vals. close to the isoelectric point. At all p_H vals. investigated, the discharging effect of the K_2SO_4 predominates at low concns. and the lyotropic influence at higher concns. The electrolyte concn. at which the lyotropic influence begins to predominate depends on the initial charge of the sol. The influence of uni- and multi-valent ions in $COMe_2$ gliadin sols was studied, together with the effect of varying p_H . In $COMe_2$ and EtOH acid gliadin sols containing equimol. quantities of different univalent electrolytes, a reversal of the lyotropic series occurs at high concns. of the solvent. It is suggested that two kinds of adsorption occur, viz., an ionic adsorption influencing the boundary potential, and a mol. adsorption causing the lyotropic effect. In special EtOH or $COMe_2$ media the last-mentioned effect is the same or zero for electrolytes of the same valency. The differences between sols containing equiv. quantities of electrolyte of different valency can be attributed to a different state of charge of the particles. W. J. BOYD.

Determination of gluten. MOHS-CZYZEWSKI (Z. ges. Getreide-Mühlenw., 1931, 18, 109—110; Chem. Zentr., 1931, ii, 340).—A discussion; Neumann's method is preferred. A. A. ELDRIDGE.

Determination and sp. gr. of gluten. E. HÖHNE (Z. ges. Getreide-Mühlenw., 1931, 18, 104—109; Chem. Zentr., 1931, ii, 340).—The sp. gr. of gluten (average 1.344) depends on the $[H]$ of the washing H_2O and on the temp. of the H_2O during weighing. For summer wheat it is higher than for winter wheat. Berliner and Rüter's method of determining gluten gives a higher val. than does drying for 15 hr. at 105° . A. A. ELDRIDGE.

Feeding value of maize and sunflower silages and the effect of both on the yield and fat content of cows' milk. K. RICHTER, K. E. FERBER, and H. KOPPISCH (Landw. Jahrb., 1931, 74, 521—533).—The % of crude protein and N-free extractives in sunflower silage is lower, and of pure protein, fat, and fibre higher, than in maize silage. Although digestibility and starch vals. were lower in the case of sunflower silage, this material was rather more fully utilised by the animals than maize silage. Fed in equal quantities sunflower silage produced smaller yields of milk of lower fat content than did maize. A. G. POLLARD.

Feeding trials with milch cows on palm-kernel and coconut cakes and mixtures of these. T. SCHMIDT and H. VOGEL (Bied. Zentr., 1932, B, 3, 520—539).—Palm-kernel cake, coconut cake, and mixtures of these increased the fat content of milk and the total fat yield. A. G. POLLARD.

Substitution of pilchard oil for butter fat in milk for calf feeding. T. A. LEACH and N. S. GOLDING (Sci. Agric., 1931, 12, 204—205).—Pilchard oil, used as a substitute for butter fat in milk rations for calves, caused the death of the calves in 2—3 weeks. In parallel experiments with rats no ill-effects were observed. A. G. POLLARD.

Percentage fat as a basis for estimating the composition of milk. O. J. KAHLBERG and LE R. VORIS (J. Agric. Res., 1931, 43, 749—755).—A statistical study of milk-yield data from 12 Holstein-Friesian cows throughout an entire lactation period has been made. Correlation coeffs. and regression equations covering the relationship of fat content to the protein, total solids, solids-not-fat, and energy contents are given.

W. G. EGGLETON.

Adulteration of ewes' milk by addition of cows' milk. R. MARTIN (Ann. Falsif., 1931, 24, 462—465).—Detection of adulteration is easy, but determination of the proportions is less so as the composition of the cows' milk is unknown. The problem is further complicated by skimming. A useful guide is given by determining the diminution in the sum of the butter-casein elements of the coagulum. Taking the butter contents of cows' and ewes' milk at 4.0 and 7.4%, and the casein contents as 3.3 and 5.46%, respectively, calculation of the degree of adulteration and/or skimming is simple, but not necessarily precise. H. J. DOWDEN.

Destruction of ropy- and bitter-milk organisms by hypochlorite. C. K. JOHNS (Sci. Agric., 1931, 12, 38—42; cf. B., 1930, 683).—Less-alkaline hypochlorite solutions (of Na or Ca) are more effective against these organisms than is a $NaClO-Na_3PO_4$ mixture. No difference was noted in the stability of these preps. during 6 months' storage. A. G. POLLARD.

Chemistry of butter and butter making. I. Comparison of four methods for the analysis of butter, with an explanation of a discrepancy found to exist in fat determinations. E. W. BIRD and D. F. BREAZEALE (Iowa Agric. Exp. Sta. Res. Bull., 1931, No. 144, 356—381).—The Mojonnier method yielded vals. averaging 0.22% lower than those of the A.O.A.C. method, and in 10% of the samples divergence in duplicate analyses was considerable. The latter is attributed to emulsification in the extraction apparatus and consequent incomplete removal of fat and to loss of the ethereal fat solution when removing stoppers from flasks. The deficit in Mojonnier vals. as compared with those of the A.O.A.C. method is equiv. to the fatty acids which are not extracted in the former, but are recorded as fat in the latter method. The above and the Kohmann methods yield high fat vals. for rancid butter. A rapid modification of the A.O.A.C. method is described. A. G. POLLARD.

Water content of butter. I. Determination of high water content. II. Water content of un-kneaded butter. III. Water content of kneaded butter. IV. Adhesion between butter fat and water or brine. V. Keeping quality. VI. Determination of air content. M. OTTE (Milchwirt. Forsch., 1931, 11, 537—589; Chem. Zentr., 1931, ii, 339).—Conditions requisite for the accurate determination of H₂O are described. The H₂O content of "raw" (washed) butter depends on the total surface of the butter particles (state of division and temp.). If the acid content of the cream is high enough to coagulate the albumin the H₂O content of the unwashed butter is high. Washing with salt solutions diminishes the H₂O content with increasing salt concn. Kneading increases the H₂O content up to a point, beyond which the increase is variable. The H₂O content diminishes with increasing fat content of the cream. The manner of pasteurisation, churning time, churning rate, and churn charge are without effect. Adhesion between butter fat and H₂O or brine depends on the temp. of the H₂O, fat (summer or winter), precooling, and amount of NaCl. The abs. H₂O or air content does not affect the keeping quality. Rahn's method for determining air in butter has been modified. A. A. ELDRIDGE.

Detection *in situ* of tin solder causing dark discoloration in cheese. W. L. DAVIES (Analyst, 1932, 57, 95—96).—A thin section of the cheese, showing, if possible, the exposed nucleus, is moistened with H₂O; a drop of 5% (NH₄)₆Mo₇O₂₄ in N-HCl is added, followed by a drop of 5% Na₂HPO₄. A blue colour indicates the presence of Sn (cf. B., 1928, 640), though the initial colour is due to Pb. T. McLACHLAN.

Turkish yoghurt. D. MAZHAR, HATIDJE, and K. HANIM (Chem.-Ztg., 1932, 56, 46—47).—Methods of prep. and analysis of yoghurt milk are described and analyses are given. W. J. BOYD.

Iodine in eggs. A. JASCHIK and J. KIESELBACH (Z. Unters. Lebensm., 1931, 62, 572—575).—I is determined by alkaline fusion and extraction with warm EtOH, the I liberated from the residue on addition of H₃PO₄ and NaNO₂ being titrated or matched in CCl₄ solution (cf. A., 1930, 1314). Ordinary eggs from Leghorn hens

contained 24.2×10^{-6} g. I/kg.; those from hens the daily diet for which contained 1.5 mg. I were lighter in colour and thinner in shell, and contained 2.981 mg./kg. or 168.5×10^{-6} g./egg (yolk 152.8, white 15.6).

J. GRANT.

Determination of the coloring matter of egg-yolk. A. TERÉNYI (Z. Unters. Lebensm., 1931, 62, 566—571).—The yolk is twice extracted with a 20-fold mixture of equal vols. of Et₂O, EtOH, and light petroleum, and the colour of the filtered extract matched in a compensated Walpole colorimeter within 30 min. against that of 1—0.1% K₂Cr₂O₇ (standardised against pure lutein). The sensitiveness is $\pm 0.4 \times 10^{-6}$ g./c.c. for 4—13 $\times 10^{-6}$ g. lutein/c.c. Six eggs yielded 0.0093—0.0186% or 1.5—2.6 mg. peryolk. Lutein decomposes to the extent of 50% in air at 40°, 11% *in vacuo* at 20°, and 20% on keeping for 24 hr. in the dark in the above solvent. J. GRANT.

Determination of chlorides in dairy products and biological material. W. L. DAVIES (Analyst, 1932, 57, 79—85).—A modification of the Volhard method is employed; the sample is treated with an excess of 0.05N-AgNO₃ and digested with sufficient HNO₃ to make the concn. 50%. AgCl is collected on a filter, if necessary, before titration with KCNS, and the results are accurate, in the case of milk, to 0.5 mg. per 100 c.c. The method is useful for routine purposes, calculation of the approx. lactose content of milk, and the evaluation of vols. of ppts. obtained while preparing a clear serum. All milks containing over 0.146% Cl are abnormal. T. McLACHLAN.

Effect of soya beans and soya-bean oil meal on the quality of pork. S. BULL, W. E. CARROLL, F. C. OLSON, G. E. HUNT, and J. H. LONGWELL (Illinois Agric. Exp. Sta. Bull., 1931, No. 366, 38—80).—Soya beans used in a grain ration for pigs produced soft pork. This occurred whether the beans were fed throughout the fattening period, or for shorter periods before or after a ration producing firm fat. The inclusion of extracted soya-bean meal in a grain ration produced pork of saleable quality, but still less firm than when the meal was omitted. A. G. POLLARD.

Indirect determination of the total solids of tomato preserve. F. ARNAUDO (Annali Chim. Appl., 1931, 21, 539—545).—The H₂O content is determined by the process of distillation with an org. liquid. C₆H₆ is used in this method. The collecting vessel is provided with a siphon so that emulsions of H₂O and C₆H₆ can be subjected to repeated distillation. Vals. obtained with PhMe and xylene are higher, but the residue undergoes alteration. The total solids obtained by difference are always higher than those given by direct exsiccation. O. F. LUBATTI.

Methods of distinguishing fresh and dry plum jams and their mixtures. P. RUDOLPH and H. BARSCH (Z. Unters. Lebensm., 1931, 62, 581—582).—(a) Since the enzymic reducing power is destroyed by drying, filtered 10% solutions of fresh pulps give no reaction, and dry pulps a blue colour on addition of starch after 1 min. at 55°, in the presence of 10% of 0.02N-I. (b) Fresh pulps give a red-brown colour and dried pulps no reaction with fuschin-H₂SO₃ and

0.01% of NaOH. The sensitiveness of these tests for mixtures of fresh and dried pulps is 20 and 10%, respectively, and either is preferred to that of Kappeller, Praege, and Rademeister (B., 1930, 790). J. GRANT.

Determination of water in jams. P. BERG and S. SCHMECHEL (Z. Unters. Lebensm., 1931, 62, 575—580).—A modification of Pritzker and Jungkunz's distillation method (A., 1929, 1029) gives results reproducible to within about 0.25% for 63% of sucrose containing 1% of tartaric acid, and for various jams; calculation of sucrose, and thence H₂O by difference, from d_{15}^{20} of a 10% filtered extract in hot H₂O gives results up to 1% lower. J. GRANT.

Examination of cinnamon. W. PEYER (Z. Unters. Lebensm., 1931, 62, 666—669).—Cinnamons from Ceylon and Java (*Cassia vera*) are similar in flavour and analysis, the latter being as a rule cheaper, higher in oil, and lower in (96%) EtOH extract (cf. B., 1929, 376). So-called Seychelles cinnamon may prove to be re-exported pure Ceylon cinnamon. The EtOH extract of the genuine product is often low (14.1%), ash 8.4% (0.4% insol. in HCl), oil 0.6%. Features of its microscopy (which are pictured) are thick-walled store-cells > 300 μ long, few cork cells, and medullary rays 2 cells thick with CaC₂O₄. J. GRANT.

Comparison of artificial, heated, and bee honey. P. RUDOLPH and H. BARSCH (Pharm. Zentr., 1932, 73, 3—4).—10 g. of honey are stirred for 3 min. with 5 c.c. of HCl (d 1.124) and 10 drops of phloroglucinol-HCl (0.625%) are added. With fresh bee honey no colour ensues, but with heated honey a reddish colour gradually develops which is easily distinguishable from that given by artificial honey (intense red). Colour reactions with pyridine acetate are also described.

P. G. MARSHALL.

Simplified test for the diastase content of honey, and detection of foreign honey by pollen analysis. J. PRESCHER and E. BOHM (Z. Unters. Lebensm., 1931, 62, 583—585).—Since in the authors' test (B., 1931, 858) the limiting starch val. for the appearance of a red colour is approx. $\frac{1}{3}$ that for the blue colour (the diastase val.), the test may be simplified by digesting 5 c.c. of 20% honey and 1—3 c.c. of 1% starch for 1 hr. at 41°, when 6 \times the no. of c.c. of starch giving a red colour on addition of 0.02N-I is the diastatic val. If this is 12, Gothe's test should be applied. Examples are also given illustrating the value of pollen analysis to determine the place of origin of chemically similar honeys. J. GRANT.

Applications of lactic acid in the [food] preserve industry. H. SERGER and K. CLARCK (Konserven-Ind., 1931, 18, 375—378; Chem. Zentr., 1931, ii, 925).—In taste, 100 pts. of citric, 113 pts. of lactic, 111 pts. of tartaric acid, and 88 pts. of AcOH are equiv. Lactic acid alone in pickles does not prevent the growth of moulds. To remove the CO₂ hardness of H₂O for the Cu-greening of vegetables, but not to avoid Fe-coloration of vegetables and fruits, lactic acid is preferred.

A. A. ELDRIDGE.

Brown discoloration of potatoes during steaming. G. RUSCHMANN (Z. Spiritusind., 1932, 55, 15).—

The brown discoloration of potatoes which often occurs during steaming and results in defective acidification is due to the combination at a high temp. of the sugars with NH₂-compounds and other substances containing N. Under normal conditions of heating, potatoes with a low sugar content develop no colour, but steaming at a pressure of 3 atm. causes them to become intensely brown owing to the formation of sugar by hydrolysis of the pectic substances. The coloration is also influenced by the duration of the steaming, 4 hr. at 100° being sufficient to produce browning. Longer periods at lower temp. give similar results, and potatoes which cool slowly in large containers colour easily. Discoloration may be avoided by the use of low-pressure steaming at 200° and by cooling rapidly. Although such potatoes show no colour, they ferment and acidify readily owing to their high content of sugar. C. RANKEN.

Changes in chemical composition of mangels and rutabagas [swedes] during storage. K. S. MORROW, R. B. DUSTMAN, and H. O. HENDERSON (J. Agric. Res., 1931, 43, 919—930).—Loss of H₂O by both roots during storage after harvest is continuous and uniform, until after 3 months the relative increase in % dry matter is of sufficient magnitude to be important in calculating the feeding val. At this time the most pronounced changes in the relative composition of the dry matter of swedes are loss of N-free extract and, to a less extent, of crude protein. The greatest change in mangels is loss of crude protein. With mangels, but not with swedes, this loss is inversely related to the dry-matter content.

W. G. EGGLETON.

Thermophilic anaerobes. F. S. PAINE (Zentr. Bakt. Par., 1931, 85, 112—129).—Anaerobic bacteria isolated from tinned vegetables are described. All strains fermented arabinose and fructose, and were sensitive to O₂. Gases produced had a high % of H₂ and low % of CO₂. A. G. POLLARD.

Rôle of the bacterial flora of pickle brine halophiles. L. M. HOROVITZ-VLASSOVA (Z. Unters. Lebensm., 1931, 62, 596—602).—The characteristics and reactions of 3 types of bacteria are described: (1) Denitrifying bacteria (10,000/c.c. in old pickle liquor, and 15 varieties isolated from Leningrad air) which usually require the presence of 1—10% of NaCl for growth and/or gas-production (e.g., *B. halobicus*). (2) Bacteria producing acetylmethylcarbinol which is detected (cf. the Voges-Proskauer test) from: (a) the eosin-red colour produced after 2 hr. on addition of NaOH and peptone to the distillate; (b) the reduction of AgNO₃ in aq. NH₃ and of cold Fehling's solution; (c) tests characteristic of the alcohol group; (d) m.p. of the osazone (243°). (3) Single, paired, or grouped lipolytic ("slime-producing"), Gram-negative cocci, 0.9 μ in diam. (e.g., *B. pyocyanus* and *fluorescens liquefaciens* and *Micrococcus lipolyticus*).

J. GRANT.

Distribution of manganese in food. W. H. PETERSON and J. T. SKINNER (J. Nutrition, 1931, 4, 419—426).—The Mn contents of 83 foods, ranging from 0.028 mg. per litre (milk) to 49.9 mg. per kg. (bran flakes), are recorded, and the foods are classified with

respect to their Mn content. Considerable variations for the same product were observed.

CHEMICAL ABSTRACTS.

Digestibility of bur clover as affected by exposure to sunlight and rain. H. R. GUILBERT and S. W. MEAD (*Hilgardia*, 1931, 6, No. 1, 1—12).—Reduced digestibility of bur clover after exposure is largely accounted for by the leaching of sol. constituents.

A. G. POLLARD.

Decomposition of straw without chemicals. F. HONCAMP and H. HILGERT (*Landw. Versuchs-stat.*, 1931, 113, 201—219).—Simple steaming of straw improves its digestibility although there is some loss of crude nutrients. The starch equiv. of the treated material is higher than that of the original and approximates to that of a poor meadow hay. Steamed straw contains no digestible protein, the small amount originally present being lost during treatment.

A. G. POLLARD.

Fodders containing sugar, as means for relieving the sugar market. F. HONCAMP (*Z. Ver. deut. Zucker-Ind.*, 1931, 81, 825—840).—Over-production of sugar in normal and good years may be avoided by foregoing max. recovery of sugar and employing after-product sugars, molasses, and dried cossettes from the Steffen process or from original beets, as fodder. The relative merits of these products for different classes of animals are discussed. Sugar and starch are approx. equiv. as sources of body heat and muscular energy. For ruminants, molasses and sweet cossettes are preferable to sugar itself.

J. H. LANE.

Food dyes.—See IV. **Determining SO₂ [in dried fruit].**—See VII. **Edible fats. Cacao butter. Rancidity of fats. Edible oils.**—See XII. **Cacao soils. Pasture.**—See XVI. **Invert sugar in honey.**—See XVII.

See also A., Feb., 200—1, **Vitamins (various). 203, New sugar in dextrorotatory honey. 206, Determining reducing sugars in milk etc.**

PATENTS.

Production of milk preparations. L. MARCHAUDON (B.P. 363,664, 22.9.30. Fr., 19.10.29).—A prep. similar to junket is made by adding rennin to cold milk, previously boiled if desired. After keeping for 15 min., the prep. is heated rapidly to 100° on a water-bath and allowed to cool.

E. B. HUGHES.

Preservation of eggs. L. LIPSCY (B.P. 364,128—9, 25.6.30).—(A) Healthy fresh eggs can be sterilised in the shell by short-wave (1—100 m.) electric radiation; heat and other rays of lesser wave-length should be eliminated. The shells are washed and dried, then irradiated, slightly de-aerated, sealed by a mixture of paraffin (m.p. 45—50°) or ceresin and stearine, and finally coated with shellac. (B) Eggs removed from their shells can be similarly sterilised by irradiation in open shallow vessels or in sealed metal containers. The exposure needed is 1—10 min.

E. B. HUGHES.

Drying of fish and slaughterhouse refuse. C. SCHLOTTERHOSE and H. BRANDT (*SCHLOTTERHOSE & Co.*) (B.P. 365,308, 9.1.31).—An apparatus is described

for drying substances which normally acquire a glue-like texture. While drying, the material is alternately heated and cooled, thereby reducing the viscosity.

E. B. HUGHES.

Manufacture of food for animals. P. S. VOIGT (B.P. 364,113, 24.9.30).—A method for manufacturing cattle feed and cakes in which materials containing vitamins-A and -C are incorporated in the form of the peel of citrus fruits (or its alcoholic extract) is claimed; carotene (as red unrefined palm oil) may also be added.

E. B. HUGHES.

Freezing of food products. FROSTED FOODS CO., INC., Assees. of C. BIRDSEYE (B.P. 366,373, 1.5.31. U.S., 25.2.31. Addn. to B.P. 345,618).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Behaviour of nitrous oxide at high pressure in relation to its application for the production of narcosis. J. C. J. WALLEBROEK (*Pharm. Weekblad*, 1931, 68, 1324—1331).—The temp. within a steel N₂O cylinder fell to -0.9° after permitting the gas, at an initial pressure of 73 atm., to escape at 10 litres per min. for 165 min.; after closing the valve the pressure rose from 39 to 58 atm., and on re-opening the valve the temp. fell to -19° (270 min.). With the usual velocity of about 20 litres per min. the temp. probably falls to -40°. It is recommended that in ordinary practice 2 cylinders should be used, so that the max. gas velocity does not exceed 10 litres per min. The influence of impurities (N₂) is described.

H. F. GILLBE.

Analysis of calcium glycerophosphate. J. OPIEŃSKA-BLAUTH (*Spraw. Prac Działu Chem. Panst. Zak. Hig.*, 1929, No. 1, 30 pp.; *Chem. Zentr.*, 1931, ii, 1326).—The method of D.A.B. VI is inapplicable in the presence of citric acid. The citric acid may be determined approx. by titration first with NaOH (phenolphthalein) and then the glycerophosphate by titration with HCl (Me-orange). For the accurate determination of citric acid in glycerophosphate Denigès' reagent is employed.

A. A. ELDRIDGE.

Injection of acetylcholine chloride solutions [aqueous and anhydrous]. L. LEMATTE, G. BOINOT, and E. KAHANE (*J. Pharm. Chim.*, 1932, [viii], 15, 49—60).—Hydrolysis of acetylcholine chloride with H₂O could not be retarded either by adjustment of the pH or by addition of any "negative catalyst." Solutions or suspensions in 45 anhyd. org. solvents have been examined. Glycerol and glycerol derivatives, especially CH₂Cl·CH₂·OAc and OH·CH₂·CH₂·OAc, and OH·CHMe·CO₂Et gave the most stable solutions which are probably the most suitable for injection.

E. H. SHARPLES.

Evaluation of pharmaceutical guaiacol preparations. M. DOMINIKIEWICZ (*Spraw. Prac Działu Chem. Panst. Zak. Hig.*, 1930, [B], No. 2, 16 pp.; *Chem. Zentr.*, 1931, ii, 284).—The assumption that in *Kali sulfoguaiacolicum* only the OH:OMe:SO₃H = 1:2:5 isomeride is of therapeutic val. is incompletely valid. Ordinary preps. contain 60—75% of the 4-SO₃H compound. The colorimetric determination of K by Rupp and Brixen's method is inapplicable; the KSO₃ group is preferably oxidised with HNO₃ and KNO₃ to

SO_4^{4-} which is pptd. with BaCl_2 , the excess of which is determined volumetrically with chromate.

A. A. ELDRIDGE.

Determination of morphine in medicinal preparations. Opium preparations. E. SCHULEK and F. SZEGHÖ (Pharm. Zentr., 1932, 73, 5—9, 17—22).—The base can be extracted from mixtures containing its salts with CHCl_3 (saturated with NH_3) containing 1% of EtOH. After removal of the solvent it is dissolved in excess of 0.02N- H_2SO_4 and the excess titrated against 0.02N-NaOH (Me-red). 1 c.c. of the acid \equiv 5.703 mg. of anhyd. morphine. With codeine-morphine solutions of known alkaloid content, the former is first determined as follows: 2 c.c. of the solution are made alkaline with 2 c.c. of 10% NaOH and extracted with CHCl_3 . The extract is washed with H_2O (alkaline with NaOH) and the CHCl_3 removed. The residue is warmed at 100° with 10 c.c. of 0.02N- H_2SO_4 and excess of acid titrated with 0.02N-NaOH (1 c.c. of acid \equiv 5.984 mg. of anhyd. codeine). The alkaline morphine solution is then acidified with H_2SO_4 and evaporated to 2 c.c., the base being then determined by the NH_3 or other method described. A detailed method for extraction and determination of morphine in opium powder or tincture is given. P. G. MARSHALL.

Influence of Bonicot injection on the nicotine content of cigar smoke. A. VAN DRUTEN (Z. Unters. Lebensm., 1931, 62, 633—647).—"Bonicot fluid" for denicotinisation is an EtOH- H_2O solution containing 2.1% of solids, and giving qual. reactions for NH_4^+ , Fe^{2+} , with small amounts of Fe^{3+} , Na^+ , SO_4^{4-} , tartrate, and citrate. Its effect is to reduce the nicotine content of cigar smoke by about 10%, and is therefore slightly less than that of distilled H_2O . J. GRANT.

Determination of nicotine in tobacco. Denicotinising of tobacco. Z. CSIPKE (Magyar Gyóg. Társ. Ert., 1931, 7, 291—318; Chem. Zentr., 1931, ii, 1646).—Analytical methods are discussed. Results for Hungarian products are recorded. The diminution in nicotine content is for "Nikotex" products 40—50%.

A. A. ELDRIDGE.

Detection of emodin and phenolphthalein in patent medicines. W. PEYER (Apoth.-Ztg., 1931, 46, 574; Chem. Zentr., 1931, ii, 1608).—Phenolphthalein is detected with KOH; it is removed with cold EtOH and the residue tested for emodin by boiling with alcoholic KOH (D.A.B.). Aloes are detected in the aq. extract with CuSO_4 and H_2O_2 (Hirschsohn) before removal of the phenolphthalein. A. A. ELDRIDGE.

Lycopodium; its collection and yield. J. MUSZYŃSKI (Pharm. Zentr., 1932, 73, 49—52).—The occurrence and characteristics of 5 species of lycopodium are described, especially those of *L. clavatum* grown in Poland. The fresh, ripe ears of this variety give 16—21% of pure lycopodium, but the commercial article seldom contains >10%, due to contamination with stems and unripe ears. E. H. SHARPLES.

Radix althææ, D.A.B. VI. H. WILL (Apoth.-Ztg., 1931, 46, 453—454; Chem. Zentr., 1931, ii, 1459).—For the detection of liming 2% of the chopped marsh-mallow root should be treated on a filter with 5 c.c. of 1% AcOH; the filtrate should give only a slight tur-

bidity with $(\text{NH}_4)_2\text{C}_2\text{O}_4$. On combustion, 1 g. of the root should leave \geq 0.07 g. of residue; treatment of a solution of this in 5 c.c. of dil. HCl followed by addition of NaOAc and $(\text{NH}_4)_2\text{C}_2\text{O}_4$ should give a ppt. which on ignition weighs \geq 0.015 g. Mucilage is determined in 6-hr. 20% solution with a Peyer viscosimeter.

A. A. ELDRIDGE.

Colutea arborescens. W. PEYER and H. HÜNERBEIN (Pharm. Zentr., 1932, 73, 1—2).—Analysis of the product "Alaxo" showed that it contained no extract of *Colutea arborescens*. The latter is very bitter and the leaves and seeds contain an emetic, whilst emodin is absent, although present in "Alaxo." The % extracted by various solvents is recorded. P. G. MARSHALL.

Umbelliferone in the micro-sublimates from Asafoetida. L. KOFLER and H. RATZ (Arch. Pharm., 1931, 269, 689—691).—The crystals in such micro-sublimates consist of umbelliferone, ferulic acid being contained in the amorphous mass and remaining behind when a second sublimation is carried out. R. S. CAHN.

Orange oil. A. H. BENNETT (Perf. Ess. Oil Rec., 1932, 23, 2).—The only satisfactory method for the evaluation of Sicilian orange oil ($d^{15.5}$ 0.8485—0.8496, and $\alpha^{15.5}$ +98.5—99.5°, aldehyde 1.30—1.60%, non-volatile residue 1.50—2.0%) is to examine the residue obtained (1.25—1.47%) by concn. at reduced pressure. For genuine oils this has d 0.8868—0.9038, α +28.0—31.6°, and contains 31.20—34.30% of aldehyde (calc. as decaldehyde) and 38—44% of free alcohols. Mandarin oil gives 2.0—2.5% of concentrate (α 4—6°) containing 15—20% of aldehyde, 25—30% of esters, and 14—18% of Me anthranilate. E. H. SHARPLES.

Lemongrass oil of Equatorial Africa and the Comoro Is. Y. R. NAVES (Parfums de France, 1931, 9, 60—72; Chem. Zentr., 1931, ii, 329—330).—Lemongrass (*Cymbopogon citratus*) oil from the French Congo had d^{15} 0.8885, 0.8891; α_D —0° 12', —0° 8'; citral 79, 81%. Oil from the Comoro Is. had d^{15} 0.883—0.895, α_D —1° 20' to 0° 30' (usually laevorotatory), citral 76—84%. Oil from Equatorial Africa had d^{15} 0.876—0.901, α_D 0°, citral 65—78%. On keeping the oil, the citral content diminishes and the d increases. The constituents of the oil are recorded. A. A. ELDRIDGE.

Examination of essential oils. L. W. WINKLER (Pharm. Zentr., 1931, 72, 705—709; 1932, 73, 52—55).—Density (d_4^{20}) determinations and b.p. (commencement of vigorous boiling under standard conditions, using 3—4 c.c. of oil) of many varieties of each of 30 different essential oils and suitable limits for these vals. are given together with crystallisation and solubility points of anise, star-anise, and fennel oils. E. H. SHARPLES.

Determining traces of CO in C_2H_4 . Et_2O explosion.—See III. Cinnamon.—See XIX.

See also A., Feb., 142, New hypnotics. 149. Amorphous and cryst. insulin. 157, Phenolic arylpropanolamines. 167, New antimalarials. 176, Microchemical determination of nicotine. 177, Alkaloids of tobacco and of columba root. 178, Lupin alkaloids. New *Aconitum* alkaloid. 179, *Strychnos* alkaloids and derivatives. 180, Aromatic arsinic acids containing NH_2 groups. 200—1, Vitamins (various). 202, Pectin of tobacco.

Non-nitrogenous constituents of tobacco smoke. 204, Caffeine and theobromine in tissues of guarana.

PATENTS.

Wound-treating materials. A. J. STEPHENS. From ASOCIACION DE PRODUCTORES DE YODO DE CHILE (B.P. 364,273, 25.11.30).—Silk is treated with an aq.-EtOH solution of I, with or without the addition of iodides of alkali or alkaline-earth metals, for use as a wound dressing. E. H. SHARPLES.

Manufacture of alkamine esters of aromatic acids [local anæsthetics]. C. MANNICH (B.P. 361,493, 25.9.30. Ger., 1.11.29).—Amino-alcohols, $\text{CH}_2(\text{OH})\text{-CRR}'\text{-CH}_2\text{-NR}_2$, where R = alkyl and R' = alkyl or H, are converted into their benzoic or nitrobenzoic esters, which produce surface as well as regional anæsthesia. β -Diethylamino- α -dimethylpropyl alcohol, b.p. 80–81°/10 mm., obtainable by reduction of the aldehyde, b.p. 175–177°, from Pr^sCHO , NHEt_2 , HCl , and CH_2O , gives a benzoate hydrochloride, m.p. 131–132°, *p*-nitrobenzoate hydrochloride, m.p. 160°, and *p*-aminobenzoate hydrochloride, m.p. 195–196°. Esters of the dimethylamino-alcohol, b.p. 166–168° [benzoate hydrochloride, m.p. 153°; *p*-nitrobenzoate hydrochloride, m.p. 180°; *p*-aminobenzoate, m.p. 79–80° (B.HCl, m.p. 93°)], and of δ -dimethylaminoisohexan- γ -ol [*p*-nitrobenzoate hydrochloride, m.p. 174°; *p*-aminobenzoate, m.p. 95° (B.HCl, m.p. 170–171°)] are also described. C. HOLLINS.

Manufacture of arsenic acids. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 360,970, 14.8.30).—3 : 3' : 4 : 4'-Tetrahydroxyarsenobenzene or a NO_2 -derivative thereof is oxidised, e.g., by H_2O_2 , to the 3 : 4-dihydroxybenzenearsinic acid. C. HOLLINS.

Manufacture of arsenobenzene compounds. W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 360,957, 12.8.30).—Sol. asymmetrical arseno-compounds are obtained by usual methods from components containing an acetic or (in the case of benzimidazole derivatives) a glycollic acid group, and one or more NH_2 -groups, primary NH_2 -groups being finally acetylated or caused to react with $\text{CH}_2\text{O-NaHSO}_3$ or glycide. Amongst the examples are : 4-acetamido-1-phenyl-2 : 3-dimethylpyrazolone-*p*-arsinic acid reduced with 2-carboxymethoxy-1-methylbenzimidazole-5-arsinic acid; 5-carboxymethoxytoluene-2-arsinic acid with 3-acetamido-4-hydroxybenzene-1-arsinic acid; *m*-hydroxybenzylidene-semicarbazide-*p*-arsinic acid with 3-acetamido-4-carboxymethoxybenzene-1-arsinic acid; 5 : 5'-dichloro-3 : 3'-diacetamido-4 : 4'-dihydroxyarsenobenzene agitated with aq. NaOH and $\text{CH}_2\text{Cl-CO}_2\text{Na}$ (1 mol.) in N_2 to convert one OH group into a carboxymethoxy-group (product, m.p. 208–209°). C. HOLLINS.

Production of nicotine-free tobacco. L. LIPPMANN (B.P. 364,464, 14.4.31. Ger., 3.5.30).—Tobacco is extracted with a solvent, e.g., H_2O , until free from nicotine. The extract is allowed to ferment, with continuous or periodic neutralisation of the bases formed with acids or acid-producing substances, until all the nicotine is decomposed. The exhausted leaves are then treated with the extract and dried. Fermentation may be assisted by inoculation with bacterial cultures. E. H. SHARPLES.

Org. compounds containing F. Separation of alcohols etc. Mol. compounds of salts of 2-phenylquinoline-4-carboxylic acid with pyrazolones.—See III. Sheep dip etc.—See XII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

See A., Feb., 131, Ag ion-gelatin equilibrium in emulsions. 139, Photo-recorder for determining particle size. 155, Diazo compounds sensitive to light.

PATENTS.

Uranium intensifiers for photography. E. L. FIEVEZ, and UNION CHIM. BELGE SOC. ANON. (B.P. 363,854, 10.1.31).—The U salt is melted with Na_2SO_4 , alum, etc., and the solidified product powdered. This product can be mixed with $\text{K}_3\text{Fe}(\text{CN})_6$ and the other usual ingredients without any chemical interaction. J. LEWKOWITSCH.

Renewing used kinematograph films. R. KUPFER (B.P. 363,989, 15.9.30).—Celluloid films become too damaged in use to be adaptable for recoating. An Fe film, coated on both sides with Al, is suitable. The gelatin layer is removable, without attacking the metal, by treatment with decaying bacteria culture. The carrier is then recoated with fresh emulsion. [Stat. ref.] J. LEWKOWITSCH.

Packing for photographic plates. I. G. FARBENIND. A.-G. (B.P. 366,387, 12.5.31. Ger., 12.5.30).

Adhesive mountants.—See XV.

XXII.—EXPLOSIVES; MATCHES.

Denitration of nitrocellulose in mixtures of nitric and sulphuric acids. DEMOUGIN and BONNET (Mém. Poudres, 1930–31, 24, 147–156).—In an investigation of the equilibrium between nitration and denitration, the N contents of nitrated linters and denitrated CP_1 and CP_1BFP , and of nitrated linters and denitrated CP_2 and CP_1 , were determined at intervals up to 90 days after immersion in mixed acid of 17.5 and 19.9% dilution, respectively. The nitration and denitration curves in the first series were symmetrical in relation to the condition of equilibrium at 12.73% N, there being in each case a slight reduction in the N, corresponding to disintegration of the fibre, after 50 days. In the second series the curves were at first symmetrical in respect of equilibrium at 12%, but the nitration curve showed a fall after approximately 28 days. Nitration of linters to CP_1 in a mixture containing 10.6% H_2O did not indicate appreciable disintegration before 2 months. It is concluded that in a given mixed acid at definite temp., nitration and denitration take place at first at equal rates, but equilibrium cannot be reached during nitration, as a secondary reaction (disintegration) becomes marked before nitration is attained. This disintegration is more rapid in dil. baths, which explains the high solubilities and low viscosities found by Berl and Klaye (B., 1907, 1255) to result from re-nitration of nitrocellulose of low N content. The trend of the reactions of nitration and denitration shows that etherification and saponification of the OH groups of the cellulose do not take place independently. W. J. WRIGHT.

Equilibrium between nitrocellulose and its nitration acid. DEMOUGIN and BONNET (Mém. Poudres, 1930—31, 24, 157—173).—Analyses were made of the waste acid, obtained by Selwig's process, and of the acid retained by the nitrocellulose after centrifugation, and comparative analyses were made of the waste acid after re-immersion of the nitrocellulose. The N content of the nitrocellulose, ratio of wt. of acid to wt. of nitrocellulose, and compositions of the acid baths are tabulated. The formation of cellulose pernitrate in the centrifuged nitrocellulose, as postulated by Carrière (B., 1926, 503), is improbable. When nitrocellulose is immersed in an acid bath with which it is in equilibrium it absorbs at the surface, and retains, an amount of the $\text{HNO}_3\text{--H}_2\text{SO}_4$ mixture dependent on the physical nature of its surface. The composition of the absorbed acid depends only on the composition of the bath in which the nitrocellulose was immersed, and is the same whether equilibrium was obtained by nitration of cotton or immersion of nitrocellulose. In both cases equilibrium is reached after about 1 hr. at 20°. Over the whole range of nitrating acids the % H_2SO_4 and HNO_3 in the adsorbed acid may be calc. from the equations: $s = 0.75S$ and $100 - n = 0.85(100 - N)$, s and n being the % of H_2SO_4 and HNO_3 in the adsorbed acid, and S and N that in the waste acid, respectively. Furthermore, there is a const. partition coeff. for the absorbed and waste acids for the H_2SO_4 and $\text{H}_2\text{SO}_4 + \text{H}_2\text{O}$ consns., since $s + h = 0.85(S + H)$, where h and H are the % of H_2O . W. J. WRIGHT.

Affinity of nitrocellulose for nitroglycol. DESMAROUX (Mém. Poudres, 1930—31, 24, 211—222).—An investigation was made by a similar method to that employed for nitrocellulose and nitroglycerin (B., 1931, 995), Et_2O being substituted for CHCl_3 . Results give the ratio of wt. of nitroglycol to total wt., and fraction of nitroglycerin mol. per mol. of mixture, in the Et_2O phase and nitrocellulose phase, respectively, and the activity of nitroglycol in both phases. There is apparently no combination, even a reversible one, between nitroglycol and nitrocellulose. Samples of SD prepared with nitroglycol and a mixture of CP (13.35% N) and CP (11.0% N) were difficult to roll and extrude; with nitrocellulose (11.7% N), rolling was satisfactory, but extrusion still difficult. It is evidently not possible to substitute nitroglycol for nitroglycerin, partly owing to the more feeble affinity for nitrocellulose of nitroglycol, and partly also to inferior lubricating characteristics. The losses sustained by mixtures containing nitroglycol and nitroglycerin, respectively, on heating to 50° for periods up to 163 days are tabulated. The curves for the two types showed the greatest divergences at the commencement of stoving, which is explained by the faster evaporation of the more volatile nitroglycol from the superficial layers of the material. W. J. WRIGHT.

Combustion of colloidal propellants. A. D. CROW and W. E. GRIMSHAW (Phil. Trans., 1932, A 230, 387—411).—An expression for the rate of burning of a colloidal propellant has been deduced, assuming that the primary cause of the breakdown of the grain surface is the vibrational energy of the incident gas mols. Agreement with experimental results is satisfactory.

C. W. GIBBY.

Variation of $f/p.d.t$ with charging density for B powders, containing either vaseline or centralite. H. MURAOUR and G. AUNIS (Compt. rend., 1931, 193, 1082—1084; cf. B., 1931, 515).—(Reciprocal of max. pressure)/(reciprocal of $f/p.d.t$) and (duration of combustion)/ $f/p.d.t$ curves for two B powders containing 40% sol. cotton with 6% of (a) vaseline, (b) centralite show that, as in the case of similarly constituted nitroglycerin powders, $f/p.d.t$ decreases for low densities of loading far more rapidly for (a) than for (b).

C. A. SILBERRAD.

Determination of the stability of smokeless powders. F. GROTTANELLI (Mem. R. Accad. d'Italia, 1931, 2, Chim. No. 6, 17 pp.).—A current of humid CO_2 -free air is passed at a rate of about 0.50 litre per hr. over 500 g. of the explosive, which is placed in a thermostat at 70—85°, and then through an absorption vessel; the rate of change of p_{H} of the solution in the vessel, i.e., the rate of production of free acid by the explosive, forms a measure of the stability of the powder. The method has the advantages that the explosive is tested in its usual form, i.e., without grinding, that the quantity required is of the same order as is used in conjunction with a projectile of average size, and that the temp. is as low as is consistent with a reasonable duration for the test. Typical results are recorded, together with results of determinations of the stabilising influence of petroleum jelly of various Br vals.

H. F. GILLBE.

Stability test of nitroglycerin-nitrocellulose smokeless powder. D. R. WIGGAM and E. S. GOODYEAR (Ind. Eng. Chem. [Anal.], 1932, 4, 73—76).—Five stability tests for double-base powders are described. Modifications of the Taliani test are suggested. Preference is given to this test, but a surveillance test is recommended for questionable powders. The Taliani test is least affected by the nitroglycerin content of the powders.

E. S. HEDGES.

Explosion-time test of double-base powders. D. R. WIGGAM and E. S. GOODYEAR (Ind. Eng. Chem. [Anal.], 1932, 4, 77—78).—The absence of stabiliser from a powder is not detected by the explosion-time test and the explosion time is not a function of the amount of nitroglycerin in the powder. Many salts lower the test without lowering the storage life; the effect appears to be limited to uni-univalent salts. Metallic oxides increase the explosion test without increasing the storage stability of the powders. Powders rendered unstable by long heating in a closed container gave satisfactory explosion tests. As the explosion test does not measure the true stability of double-base powders, it is valueless in indicating their chemical condition.

E. S. HEDGES.

See also A., Feb., 131, **Explosion of alkali chlorates due to shock. Oxides of the alkaline-earth metals.**

PATENTS.

Production of smokeless hunting and practice powder. E. BAZYLEWICZ-KNIAZYKOWSKI and K. PARTYKA (B.P. 364,223, 23.10.30).—Smokeless propellant powder, after gelatinisation, is extruded in the form of solid cords (0.3—1.5 mm. diam.), which are cut into 20-cm. lengths and superficially dried at 50° for 1—2 min., so that the surface hardens whereas the inner core

remains soft. The lengths are then immersed for 10–25 min. in an inert liquid, *e.g.*, H_2O , at 45–80°, the consequent expulsion of the solvent resulting in the formation of tubes having walls 0.03–1 mm. thick. If the original solvents included Et_2O , a reducing agent should be added to the liquid to neutralise peroxides produced. The tubular lengths are finally cut into grains, dried, and coated. W. J. WRIGHT.

Uninterrupted preparation of fulminate of mercury. J. MEISSNER (B.P. 363,661, 22.9.30. Ger., 25.9.29).—The apparatus comprises a tower, in the upper portion of which is a series of funnel-shaped reaction vessels, which can be opened or closed as required, whilst the lower portion contains funnels provided with baffles. $EtOH$ and a solution of Hg in HNO_3 , continuously introduced into the top of the tower, react in the uppermost vessel, whence, when it is full, they pass successively into the lower vessels, reaction being accelerated by the rising reaction vapours. The mixture of fulminate and mother-liquor flows down, and is cooled by, the baffled funnels and passes through a pipe at the base. Another pipe at the bottom of the tower serves to introduce cooling air, and one at the top conducts the vapours to a condenser. W. J. WRIGHT.

Shot-gun cartridges. IMPERIAL CHEM. INDUSTRIES, LTD., H. W. BROWNSDON, W. R. NIMMO, and G. P. POLLITT (B.P. 354,718 and Addn. B.P. 364,282, [A] 12.2.30, [B] 29.11.30).

XXIII.—SANITATION; WATER PURIFICATION.

Efficiency of certain germicides in the preservation of biologic products. W. G. MALCOLM (J. Bact., 1931, 22, 403–425).—Comparative tests of the efficiency of a number of phenolic compounds and of proprietary org. mercurials are recorded. A. G. POLLARD.

Water-softening plant. J. C. COTTERILL (Gas World, 1932, 32, 78–81).—The advantages of the soda-lime and zeolite process over that using soda-lime alone are discussed and costs are given. D. K. MOORE.

Membrane filters for filtration of water, especially drinking water. W. HOFFMANN (Angew. Chem., 1932, 45, 143–146).—Examples are given of the reduction of the content of impurities of samples of moor- H_2O effected by filtration through membrane filters of various grades. Although a sand filter removes a considerable amount of impurity, even a rapid membrane filter will subsequently remove a further quantity. Detailed analyses are given of a brown moor- H_2O in its crude state, after aëration, after filtration through sand and through membrane filters, and after a combination of these treatments. Sand filtration gave a clear colourless H_2O , but the Fe content was 3 mg. per litre and the specimen on boiling became brown. Aëration and filtration through a 10- or 20-sec. membrane filter gave a clear H_2O which became only faintly yellowish on boiling and contained only 0.1 mg. of Fe per litre; the H_2O was free from colloidal matter and practically sterile. H. F. GILLBE.

Titration of [total] alkalis in water. J. TILLMANS and E. NEU (Z. Unters. Lebensm., 1931, 62,

593–596).—The H_2O is evaporated with H_2SO_4 , the excess acid being removed by gentle ignition of the residue, a boiling- H_2O extract of which is then pptd. with $Ba(OH)_2$, filtered when cold, and the red colour produced on addition of phenolphthalein destroyed by passing in CO_2 , and finally restored by evaporation. If exactly 20 c.c. of 0.1N- Na_2CO_3 are then added at the b.p. and the solution is filtered cold, the sum of the equivs. of the Na and K salts may be obtained (*e.g.*, in terms of NaCl) by titration with 0.1N-HCl to methyl-orange. The accuracy is ± 0.3 mg. for 8–88 mg. of NaCl (or the equiv. of KCl). J. GRANT.

[Determination of] dissolved oxygen in presence of organic matter, hypochlorites, and sulphite wastes. E. J. THERIAULT and P. D. McNAMEE (Ind. Eng. Chem. [Anal.], 1932, 4, 59–64).—A technique has been developed for determining dissolved O_2 by Winkler's method in presence of large amounts of org. matter. Various sources of interference are discussed and attention is directed to the error caused by the decomp. of $Na_2S_4O_6$ in highly alkaline solutions. A modification of Winkler's procedure for determining dissolved O_2 in sulphite wastes is proposed. E. S. HEDGES.

Choice of international index of measurement for examination of waters. A. SULFRIAN (Ann. Chim. Analyt., 1931, [ii], 13, 353–356).—It is suggested that hardness of water should be expressed in "millinorms," *i.e.*, milliequiv. of foreign matter per litre. A chart is given for converting British, French, and German hardness units into "millinorms." J. W. SMITH.

Boiler water.—See I. NaCl manufacture.—See VII. Applications of lactic acid.—See XIX.

See also A., Feb., 135, Testing distilled H_2O . Conductometric titration. 198, Bactericidal H_2O by filtration and sterilisation.

PATENTS.

Collecting and governing the storage of gas derived from sewage treatment. M. W. MILLS, J. BOLTON, J. F. BOLTON, and R. AMES (B.P. 364,285, 2.12.30).—The gas evolved during sludge digestion is collected in a submerged dome in the tank (A) and conveyed through a governing chamber (B) to a gas-holder (C) in such a way that the pressure of the gas and the weighting of C automatically regulate the liquid within B. Seals are arranged on the respective gas connexions and on the water connexion between B and A, so that the level of the sludge in the latter can be varied without varying the pressure in B and C, and the gas produced can be collected without serious loss. C. JEPSON.

Means for exterminating vermin. W. and C. F. L. LANGE (B.P. 364,503, 11.6.31).—A 5% solution of $K_2Cr_2O_7$, alum, and potash in the proportions of 2 : 1 : 1 is claimed as suitable for a spray insecticide. C. JEPSON.

Respiratory apparatus [supplying filtered air] for protection against poisonous and injurious substances. A. MUNTWYLER (B.P. 365,762, 7.1.31. Switz., 20.1.30).

Furnaces [for garbage etc.].—See I.