

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

NOV. 23 and 30, 1934.\*

### I.—GENERAL; PLANT; MACHINERY.

**Heat transmission in rotary kilns.** X. W. GILBERT (Cement, 1934, 7, 295—308; cf. B., 1934, 735).—The effects of variation in kiln diam. ( $D$ ) and charge % ( $C$ ) on the rate of heat transmission ( $H$ ) per unit vol. and per unit surface, in the drying zone, are examined.  $H$  is highest when  $C$  and  $D$  are large at the hot end of the kiln and small at the cold end.

T. W. P.

**Some factors affecting furnace heating practice.**

II. R. J. SARJANT (Trans. Ceram. Soc., 1934, 33, 331—351; cf. B., 1933, 367).—Available methods for estimating the heat transmission by radiation and convection in flowing gases, and for computing draught losses (e.g., in mines, in smooth and rough brickwork ducts, and in steel, galvanised, and smooth Pb pipes), are summarised and illustrated by extensive data. A chart is included showing the radiation from, and the sensible heat (for varying proportions of excess air) in, the (non-luminous) products of combustion of British coals.

A. L. R.

**Heat and power balance in chemical plants.**

G. STUDLEY (Chem. Met. Eng., 1934, 41, 464—467).—The use of steam for power (via turbo-alternators) and of exhaust steam for heating purposes, involving steam bleeding, reheating, and make-up  $H_2O$  evaporation (for boiler pressures  $> 650$  lb. per sq. in.), is discussed and illustrated by heat-balance diagrams.

D. K. M.

**Nomograms for gas and heating industries.**

W. SCHREIBER (Gas- u. Wasserfach, 1934, 77, 716—718).—A nomogram is described for reducing the vols. of gases saturated or partly saturated with  $H_2O$  to standard conditions ( $0^\circ$  and 760 mm. dry or  $15^\circ$  and 760 mm. wet). A calculator for the same operation is also described. The nomogram also gives the factor for calorific val. A further nomogram gives the correction term when the  $d$  of a wet gas is determined by diffusion.

E. H. M. B.

**Cooling mixtures (emulsions).** J. A. RUBINSKI

and G. A. MEDVEDEVA (Zavod. Lab., 1934, 3, 657—660).—Emulsions of oil in 0.4% aq.  $Na_2CO_3$  are recommended for cooling steel.

R. T.

**Crushing and grinding.** W. F. CAREY (Inst. Chem. Eng., Advance copy, Oct., 1934, 5 pp.; cf. B., 1934, 303).—The theoretical work required for crushing a solid = the product of log. mean size reduction, wt. of material crushed, and a const. the val. for which is given for a no. of materials. When coal is crushed in a single operation to  $< \frac{1}{2}$  its original size the particles aggregate to a flake which requires enormous stresses for further reductions in size. The low efficiency (1%)

of practical crushing is due only in a small degree to the production of oversize; ball mills and ring rolls tend to form aggregates, whilst impact machines suffer excessive windage losses, or else fail to fracture a large proportion of the particles which are hit. The development of the multi-stage, high-speed roll crusher as a means of reducing power consumption is indicated.

D. K. M.

**Crushing and grinding appliances—connexion between type and purpose.** B. W. HOLMAN (Inst. Chem. Eng., Advance copy, Oct., 1934, 10 pp.).—A review of grinding appliances and their uses, with illustrations.

D. K. M.

**Rational methods of selecting screw conveyors.**

R. F. BERGMANN (Chem. Met. Eng., 1934, 41, 470—472).—For each of 4 classes of a no. of different materials a definite loading fraction of the conveyor is recommended. From graphs, the capacity of conveyors of different size for each class of material and the max. no. of r.p.m. recommended may be obtained. A formula is given for calculating the power consumption. For conveyors inclined at  $> 20^\circ$  factors allowing for slip are given; the power required is that used by a similar horizontal conveyor + that for lifting the material.

D. K. M.

**Welding pipe and pressure-vessel joints in chemical industries.** C. O. SANDSTROM (Chem. Met. Eng., 1934, 41, 476—481).—The use of welded joints on pipe-lines and boilers is indicated, and methods of making different kinds of welds for various purposes are discussed, with illustrations.

D. K. M.

**Colorimetric determination of silica in boiler water.** M. C. SCHWARTZ (Ind. Eng. Chem. [Anal.], 1934, 6, 364—367).—The conditions for the max. intensity of the yellow colour of the silicomolybdate complex have been determined. After removing  $PO_4^{3-}$  by pptn. as  $Ca_3(PO_4)_2$  in a  $NaOH-Na_3BO_3$  buffer solution, the colour developed on adding  $NH_4$  molybdate and  $HCl$  is matched against a  $K_2CrO_4$  standard.

E. S. H.

**Filtration with kieselguhr.** R. SALVÉ (Ann. Zymol., 1934, 340; Woch. Brau., 1934, 51, 307—309).—Buffer solutions (I) were influenced by kieselguhr (II) if the  $p_H$  of (I) and (II) differed. (II) with small loss on ignition was usually neutral or slightly alkaline; with large loss it was acid. Rapidity of filtration was dependent on the size of particles, as was also the decolorising effect. Different samples of (II) affected the  $d$  of sugar solutions in an irregular manner, whilst addition of (II) to beer caused adsorption of uncoagulable  $N \propto$  the amount of (II) used, adsorption being increased

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

by heating. Using a Seitz- or a Meta-filter, the clarification was dependent on the microscopical state of (II). Examination of (II) should include microscopical investigation, particle size (sieve), and filtering power, and from the results its suitability for various types of filtration can be gauged. I. A. P.

**Changes in the viscosity of liquids with temperature, pressure, and composition.** C. S. CRAGOE (Proc. World Petroleum Congr., 1933, 2, 529—541).—The abs. viscosity ( $\eta$ ) and the kinematic viscosity ( $\eta\nu$ , where  $\nu$  is sp. vol.) of a liquid can be expressed, to a good approximation, by:  $\eta = Ae^{B/L}$  or  $\eta\nu = Ae^{B/L'}$ , where  $A = 5 \times 10^{-4}$ ,  $B = 1000 \log_e 20$ , and  $L, L'$  are functions which are linearly related to the temp., and the reciprocals of which are linearly related to the pressure; also for mixtures of two liquids a linear mixture rule is applicable to  $L$  and  $L'$ . Tables are given for converting  $\eta$  into  $L$ , or  $\eta\nu$  into  $L'$ , and for converting Saybolt sec., Redwood sec., or Engler degrees into  $L'$ , and the application of the formulæ to practical problems is briefly explained. A. B. M.

**Höppler viscosimeter.** G. STEINHOFF and E. WIGEL (Z. Spiritusind., 1934, 57, 253).—The instrument is described, and its use for measuring the  $\eta$  of starch solutions discussed. Determinations are simple, rapid, and accurate. I. A. P.

**Design of liquid-gas contact equipment. I. Coefficients of mass transfer through the gas film.** R. FALLAH (J.S.C.I., 1934, 53, 262—266 *t*).—Equations for correlating mass-transfer ( $M$ ) data in gases (in wetted wall towers) and for solid-packed towers, introducing a wall-effect factor ( $F$ ), are proposed. A curve is constructed giving  $F$  for different conditions, from experimental results of Furnas, based on the relationship between  $M$  and pressure drop.

**Flame furnace.**—See IX. **Dielectric constants.**—XI. **Tonometer.**—See XIII. **Super-centrifuge.** **Grinding of feeding-stuffs.**—See XIX.

#### PATENTS.

**Heat-transfer apparatus for semi-solid products.** A. JOHNSON, Assr. to LAND O'LAKES CREAMERIES, INC. (U.S.P. 1,949,374, 27.2.34. Appl., 17.5.33).—The apparatus comprises an inner axial space ( $A$ ), an intermediate, rather narrow, annular space ( $B$ ), and an outer annular space ( $C$ ). The material is passed through  $B$ , being conveyed by a helical blade on the rotatable casing of  $A$ , and heating-cooling medium is passed through  $A$  and  $C$ . The apparatus is suitable for the treatment of cream. B. M. V.

**[Heat]-insulating material.** M. D. SQUIERS (U.S.P. 1,949,087, 27.2.34. Appl., 20.6.32).—A felted sheet is formed from 1500 lb. of  $H_2O$  (soft, or softened by addition of  $Na_3PO_4$ ), 100 lb. of mineral wool, and asphalt emulsion equiv. to 7 lb. of asphalt. B. M. V.

**Vapour generators intended particularly for mercury.** BRIT. THOMSON-HOUSTON Co., LTD., Assees. of A. J. NERAD (B.P. 416,769, 29.12.33. U.S., 29.12.32).—A heating unit ( $U$ ) is arranged below the drum ( $D$ ) containing liquid to be evaporated, and conduits are

provided for connecting  $U$  to  $D$  at a point above the cold-liquid level at the commencement of vaporisation. J. S. G. T.

**Crushing machinery.** G. W. BORTON, Assr. to PENNSYLVANIA CRUSHER Co. (U.S.P. 1,948,504, 27.2.34. Appl., 3.2.31).—In a hammer crusher the screen grid is in the form of a complete cylindrical shell, is rotated, and is provided with lifting members on the interior. The material is fed through a large trunnion at the end remote from the shaft of the hammers. B. M. V.

**Rock-crushing machine.** W. H. SALLWASSER (U.S.P. 1,946,763, 13.2.34. Appl., 3.6.31).—A crushing sleeve or roll ( $R$ ) is mounted (with anti-friction bearings) on an eccentric shaft ( $S$ ) and operates between a pair of adjustable breaker plates ( $P$ ).  $R$  is restrained from acquiring the rotary speed of  $S$  when the machine is empty by means of a brake. Damage by uncrushable pieces is prevented by frangible supports for  $P$ . B. M. V.

**Crushing apparatus.** (A) N. D. LEVIN, (B) W. J. ARMSTRONG, Assrs. to (A, B) JEFFREY MANUFG. Co. (U.S.P. 1,947,783—4, 20.2.34. Appl., [A] 14.5.29, [B] 25.2.30. Renewed [A] 30.7.32).—Forms of hammers are described, adapted to disappear within the circumference of the driving discs when uncrushable pieces are struck. B. M. V.

**Crushing and grinding machine.** P. S. KNITTEL (U.S.P. 1,946,701, 13.2.34. Appl., 29.6.29).—In a machine of the type described in U.S.P. 1,698,268 (B., 1929, 306), at the end of the arc of the grate section, a gate is provided through which uncrushable material may be removed from time to time, and the rotor embodies only two mullers or hammers set at  $180^\circ$  to each other. B. M. V.

**Intercone mill. [Gyratory cone crusher.]** H. H. RUMPEL, Assr. to SMITH ENG. WORKS (U.S.P. 1,945,791, 6.2.34. Appl., 17.9.28).—The claims relate mainly to an eccentric sleeve shaft. B. M. V.

**Grinding mill.** E. L. V. PELT, Assr. to AMER. BALL MILL Co. (U.S.P. 1,947,505, 20.2.34. Appl., 23.3.31).—A renewable lining for a ball mill is described. B. M. V.

**Discharge for ball mills.** E. L. V. PELT, Assr. to AMER. BALL MILL Co. (U.S.P. 1,946,511, 13.2.34. Appl., 5.10.31).—A peripheral screen ( $S$ ), separated from the grinding compartment, is provided. After passing through  $S$  the pulp is lifted to the trunnion for discharge. B. M. V.

**Pulverisation of minerals and similar materials.** N. H. ANDREWS and W. J. WILLOUGHBY, Assrs. to AMER. PULVERIZING CORP. (U.S.P. 1,948,609, 27.2.34. Appl., 18.1.32).—In a pulveriser operating by impact of particles borne at high velocity in gaseous jets directed to a focus, the solid material ( $M$ ) is not fed with the gas, but the jets of gas (steam) are injected to a focus situated well below the upper surface of a loose mass of  $M$ . B. M. V.

**Mills for grinding and the like.** D. Y. BRYANT, and RITCHIE-ATLAS ENG. Co., LTD. (B.P. 416,869, 23.3.33).—An airtight outer casing ( $C$ ) is fitted to the mill ( $M$ ) and connected to a suction fan. Two pockets ( $P$ ), fitted with screens ( $S$ ), are fixed inside  $M$ , and the

finer, as formed, fall into *P*, whence they are drawn into *C* through *S*. A. WE.

**Separating [screening] apparatus.** A. J. WEINIG (U.S.P. 1,948,606, 27.2.34. Appl., 12.1.31).—A trommel suitable for attachment to a ball mill or the like is of larger diam. than that of the inlet trunnion, and is provided with screens of progressive mesh; inside there is a helical blade to cause travel of the material irrespective of slope. B. M. V.

**Apparatus for separation of materials of different specific gravity.** T. M. CHANCE (by F. M. CHANCE), and PROVIDENT TRUST CO. OF PHILADELPHIA (EXORS.) (U.S.P. 1,949,242, 27.2.34. Appl., 11.2.30. Renewed 16.5.33).—In a sand-pulp gravity-separation system the agitation and separation are continuous but the discharge of heavy refuse is intermittent through a lock chamber (*L*). After a discharge, *L* is filled with H<sub>2</sub>O that has been used for washing the sand from the clean coal (*e.g.*), and separated from the sand by settling. B. M. V.

**Apparatus for recovering material [heavy liquid] used in gravity separation.** F. I. DU PONT (U.S.P. 1,949,117, 27.2.34. Appl., 27.6.30).—The coal or the like which has been cleaned in a heavy solution, *e.g.*, of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, is washed in a staircase of vessels similar to a train of Zn boxes in the cyanide process, but each vessel is mounted on rollers and supported on sloping rails so that an unwashed unit may be pushed in at the bottom end and a clean one removed from the top. B. M. V.

**[Pneumatic] separating system.** H. KREISINGER, Assr. to INTERNAT. COMBUSTION ENG. CORP. (U.S.P. 1,949,279, 27.2.34. Appl., 3.3.31).—From a pneumatic grinding and separating system, that portion of air which is wasted to atm. is drawn off from a cyclone collector (*A*) which is additional to that used for collecting the ground material, the lower outlet of *A* delivering dust-laden air back to the mill. B. M. V.

**Centrifugal separator.** G. TER MEER, Assr. to A. T. OTTO & SONS (U.S.P. 1,947,068, 13.2.34. Appl., 14.4.32. Ger., 7.5.31).—A fan-shaped feed distributor which delivers the material evenly over the axial width of the centrifugal drum is described. B. M. V.

**Centrifugal apparatus.** A. PELTZER and A. PELTZER, JUN., Assrs. to MERCOR CENTRIFUGAL SEPARATOR CO., LTD. (U.S.P. 1,945,786, 6.2.34. Appl., 23.9.30).—A continuous centrifuge for two liquids is described in which part of the heavier liquid is recirculated as carrier liquid. B. M. V.

**Filtering or straining apparatus.** AUTO-KLEAN STRAINERS, LTD., and W. R. BELDAM (B.P. 416,097, 15.3.33).—In a strainer of the coiled-wire type, upon a layer of plain wire forming a coarse filter medium is superposed < 1 layer of stranded wire forming finer filter medium, wound, if desired, in < 2 turns per turn of the single wire. B. M. V.

**Filtering apparatus.** G. W. O'KEEFE, Assr. to FILTRATION ENGINEERS, INC. (U.S.P. 1,948,947, 27.2.34. Appl., 8.11.29).—A rotary, vac., external drum filter is operated with a gradually increasing vac. obtained by arranging the rotary-valve ports to open slowly. B. M. V.

**Screen [for liquids].** C. H. NORDELL (U.S.P. 1,948,125, 20.2.34. Appl., 11.4.32).—A screen for sewage and the like comprises a rotating drum built up of spaced annular discs on the outer edges of which cutting teeth are formed, which in conjunction with a stationary comb extending into the spaces comminutes the collected material and keeps the strainer clear. B. M. V.

**Fractionating apparatus.** R. W. BIELEFELDT (U.S.P. 1,948,500, 27.2.34. Appl., 28.9.31).—Bubbling caps are constructed so that, after bubbling, the gas descends through a curtain of falling liquid. B. M. V.

**Air cleaner.** W. W. LOWTHER, Assr. to DONALDSON CO., INC. (U.S.P. 1,949,212, 27.2.34. Appl., 20.7.31).—The air is admitted downwards through an axial conduit into a pool of oil, and the oily air passed upwards through screens (*S*) of meshwork of decreasing aperture and decreasing spacing between *S* and, finally, through fibrous material. *S* are conical so that oil in excess of that necessary to form a film will drain off. B. M. V.

**Self-cleaning dust collector.** E. F. ANNIS (U.S.P. 1,949,002, 27.2.34. Appl., 15.3.32).—Filter chambers are formed from a continuous band of material running in zigzag shape between walls at the edges, the gas being caused to pass through the same band twice. B. M. V.

**Dust separator.** J. B. TAYLOR, Assr. to BY-PRODUCTS RECOVERIES, INC. (U.S.P. 1,948,363, 20.2.34. Appl., 27.5.27).—The dust is collected between deeply corrugated plates which are dry but provided with barbed surfaces. The flow of gas is generally radially outwards, and therefore expanding; sections of the separator are cut off from the flow at intervals and jarred to dislodge the dust. B. M. V.

**Apparatus for filtration of air or gases.** H. A. BRASSERT, A. J. BOYNTON, and J. P. GRILLI, Assrs. to H. A. BRASSERT & CO. (U.S.P. 1,947,447, 20.2.34. Appl., 11.1.32).—A grounded, cylindrical filtering medium is provided with a cage of live electrodes on the inside, which is also the inlet side for dirty gas, and with irrigating sprays outside. B. M. V.

**Adsorption system.** C. F. ABBOTT and C. A. CONKLIN, 3RD, Assrs. to C. F. HOCKLEY (Receiver for SILICA GEL CORP.) (U.S.P. 1,948,779, 27.2.34. Appl., 30.6.31).—Two adsorbers (I, II) are operated always in series, but reversing from time to time, and with a condenser (*C*) in between. The gas, before passing to (I), is conditioned (heated) so as to remove most of the condensable vapour (*V*) previously collected in (I); this *V* is re-deposited by cooling in *C* and the gas thereby reconditioned so as to be properly stripped in (II). B. M. V.

**Obtaining a continuous mixture of liquid and gas.** C. COLEATTI (B.P. 416,038, 3.3.33).—A tall, closed, cylindrical vessel is provided with openings through the bottom: (1) inlet for liquid, (2) inlet for gas, (3) outlet for aerated liquid, and, through the top, (4) outlet for initial air and unabsorbed gas. (3) is hand-controlled to draw off aerated liquid as desired, (1) is provided with a weighted valve which opens when the internal pressure drops sufficiently, (2) and (4) are closed alternately (but never together) by needle

valves on the opposite ends of a long spindle which is moved upwards and downwards by a float having considerable free motion. The result is that the vessel fills itself with liquid and the gas, which is supplied at greater pressure than the liquid, enters only when (4) is closed and is thus dissolved under pressure, which is maintained until the vessel is nearly emptied; then the cycle starts again. B. M. V.

**Apparatus for catalytic oxidation of gases.** R. C. JEFFCOTT, Assr. to CALCO CHEM. CO., INC. (U.S.P. 1,949,122, 27.2.34. Appl., 5.7.29).—A catalyst, *e.g.*, for the oxidation of  $\text{SO}_2$ , is maintained at a lowering temp., *i.e.*, at a temp. gradient reverse to that which would be produced by the exothermic reaction. To effect this the catalyst is divided into separate masses (*M*); heat-transmitting ribs extend inwards between *M* and outwards from the wall of the container, the latter parts being surrounded by chambers in which individually regulable air currents (*A*) are maintained. Some of the hottest *A* are passed to the first *M* and the reacting gases guided towards the ribs by baffles. B. M. V.

**Apparatus for measurement of viscosity.** W. A. NIVLING, Assr. to AUTOMATIC APPLIANCE CO. (U.S.P. 1,945,822, 6.2.34. Appl., 13.7.26. Renewed 12.3.32).—A gear wheel or other pump of const. delivery forces the liquid into a closed vessel (*V*) having a const. outlet aperture (not of the sharp-edged type). The pressure of the air retained in *V* is a measure of  $\eta$ . B. M. V.

**Treating liquids. Catalysts.**—See VII. Catalysts. **Alloy for condenser tubes.**—See X. **Chlorinated rubber [insulation].**—See XIV. **Molasses feed.**—See XIX.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**Graphical classification of carbonaceous minerals: mineral oils.** H. BRIGGS (Proc. Roy. Soc. Edinburgh, 1934, 54, 115—120; *cf.* B., 1931, 659; A., 1932, 716).—Graphical relationships between the O and C contents of crude mineral oils are developed. It is concluded that coal and petroleum probably had a common origin in vegetable matter, and that the development of oil occurred through a fossil-resin stage. J. W. S.

**Products of the natural development of coal and oil.** H. BRIGGS (Proc. Roy. Soc. Edinburgh, 1934, 54, 121—134; *cf.* preceding abstract).—It is concluded that up to the semi-bituminous stage, coal is produced by a series of processes which expel  $\text{CO}_2$ ,  $\text{CH}_4$ , and  $\text{H}_2\text{O}$ , whilst for petroleum the processes eliminate  $\text{CO}_2$  and  $\text{CH}_4$ , but require the absorption of  $\text{H}_2\text{O}$ , throughout the processes of generation.  $\text{H}_2\text{O}$  is required for the further conversion of semi-bituminous coal into anthracite and finally graphite. J. W. S.

**Automatic instrument for sampling coal and anthracite.** A. V. KOLLEGAEV and P. L. RABINOVITSCH (Zavod. Lab., 1934, 3, 655—657).—Apparatus is described. R. T.

**Extraction of bituminous coal and its banded constituents.** G. KEPPELER and H. BORCHERS (Brennstoff-Chem., 1934, 15, 341—347; *cf.* B., 1934, 865).—Extraction with tetralin under pressure at 265° gave

20.8% of extract ("bitumen") from a coking coal as compared with 14.2% from a gas coal; the bright (I) and dull constituents (II) of the former yielded 25.4 and 12.8%, and of the latter 18.3 and 13.4%, of bitumen, respectively. The volatile matter contents of the extracted coals were not very much < those of the corresponding original coals, and similar yields of products were obtained by the low-temp. carbonisation of the coals before and after extraction, but the tars differed in physical constitution. The original coal, extracted coal, and extract exhibited only small differences in their ultimate composition. Solvent fractionation gave similar results with the bitumens extracted from the bright and dull coals, respectively. The gas coal was slightly more reactive towards Br (in  $\text{CCl}_4$  solution) than the coking coal, and (I) were more reactive than (II). Extraction had little effect on the amount of Br absorbed by the coals. Adsorption experiments with methylene-blue and with  $\text{H}_2\text{O}$  vapour indicated that extraction had brought about an increase in the "inner surface" of the coal. The origin of (I) and (II) is discussed; the occurrence of alternations in the type of the original vegetation, which may lead to the formation of alternate layers of (I) and (II) in the coal, is confirmed by observations of the structure of present-day "high-moor" peat deposits. A. B. M.

**Simultaneous determination of sulphur and ash in non-bituminous coals and coke by combustion in a stream of oxygen.** L. D. RASKIN (Zavod. Lab., 1934, 3, 614—616).—0.5 g. of material is burned in  $\text{O}_2$ , and the issuing gases are passed through 30 c.c. of 0.5%  $\text{H}_2\text{O}_2$ , the final  $\text{H}_2\text{SO}_4$  content of which is determined by titration with 0.05N-NaOH. The ash remaining after combustion is weighed. R. T.

**Gasification process for solid fuel.** M. K. GRODZOVSKI and Z. F. TSCHUCHANOV (Compt. rend. Acad. Sci. U.R.S.S., 1934, 3, 356—359).—In the combustion of activated coal and coke both  $\text{CO}_2$  and CO are primary products. The CO escaping further oxidation and the reaction  $\text{CO}_2 + \text{C} \rightarrow 2\text{CO}$  depend on the velocity of the air or  $\text{O}_2$  stream. H. J. E.

**Hydrogenation of bituminous coal.** K. GORDON (Proc. World Petroleum Congr., 1933, 2, 317—321).—The process developed by Imperial Chemical Industries, Ltd. is briefly outlined and the properties of the products are described (*cf.* B., 1932, 52; 1934, 530). A. B. M.

**Collin system of coke-oven heating.** N. J. BOWATER (Gas World, 1934, 101, Coking Sect., 103).—This system gives as low a free-space temp. as the Goldschmidt top channel. The increased yield of benzol at the Grimberg colliery, Ruhr, since the Goldschmidt system has been in use may be due merely to increased efficiency of the benzol washing plant. E. H. M. B.

**Determining the reactivity of coke.** W. J. MÜLLER and E. JANDL (Brennstoff-Chem., 1934, 15, 347—351; *cf.* B., 1931, 660).—The apparatus described previously has been modified to give a more uniform gas flow.  $\text{CO}_2$  passes from a const.-pressure reservoir through a control cock to a flow-meter (*M*) and thence through the reaction tube. The outlet gas ( $\text{CO}_2 + \text{CO}$ ) passes through a second *M*, a soda-lime tube, and

finally through a third  $M$  (CO). The  $M$  are of the type comprising a capillary tube combined with an inclined-tube manometer; before the gas enters each  $M$  its temp. is controlled by passing through a  $H_2O$ -jacketed cooler. The reaction tube (1 sq. cm. in cross-section) is packed with 10 c.c. of coke, 2–3-mm. size, and is heated at  $900^\circ$ . The technique of the method is described in detail. The reactivity ( $R$ ) of the coke (%  $CO_2$  reduced to CO) is readily calc. from the readings of  $M$ . Most cokes exhibited a const. val. for  $R$  after 20–30 min. gas flow at the reaction temp. The vals. varied from 27 to 75% for a series of metallurgical cokes. The apparatus can be made to record automatically.

A. B. M.

**Manufacture and use of active carbon, particularly for gas masks.** M. VÉROLA (Mém. Soc. Ing. Civ. France, 1934, 87, 282–286).—Charcoal from beech, poplar, fruit stones, and coconuts is chiefly used. Two methods of prep. are described.

W. J. W.

**Comparative tests for evaluating activated (decolorising) carbons.** K. SMOLENSKI and W. ZERO (Rept. Centr. Lab. Polish Sugar Ind., 1934, 64–95; Internat. Sugar J., 1934, 36, 354, 406).—Results are given for  $H_2O$  content, total and  $H_2O$ -sol. ash,  $pH$  val., decolorising power, and rate of filtration for various carbons.

J. P. O.

**Improvement of gas by removal of benzol with wash oil or active carbon.** L. MUSSO (L'Ind. Chimica, 1934, 9, 1173–1182).—Purification with active C (I) is better than with wash oil. Gas containing, in g./cu. m.,  $C_6H_6$  37.5,  $H_2S$  5.77, fixed org. S 0.4, total S 6.17, HCN 0.963, and  $C_{10}H_8$  0.463 showed, after purification with (I), 0.1, 0, 0.178, 0.178, 0.108, and 0 g./cu. m., respectively, of these impurities. The process yields a benzol suitable for motor fuel.

T. H. P.

**Analysis of dry refinery gases below pentane by simple batch-distillation.** J. HAPPEL and D. W. ROBERTSON (Ind. Eng. Chem. [Anal.], 1934, 6, 323–326).—A simplified method is described for calculation of a master-graph, whereby the composition of the gas is determined by a simple distillation.

R. S. C.

**Chemical control of dissolved acetylene manufacture.** O. LAZAR (J. S. African Chem. Inst., 1934, 17, 58–79).—Details of manufacture are described. Methods for determining  $CaC_2$ ,  $C_2H_2$ ,  $PH_3$ ,  $SiH_4$ ,  $NH_3$ , and  $H_2S$  are given.

A. G. P.

**Effect of carbonising conditions on yield and composition of tar.** D. R. PRYDE (Gas World, 1934, 101, Coking Sect., 108–110).—A review. The yield and composition of tar are shown to depend on the carbonising temp., the free space, the nature of the carbonising apparatus, the  $O_2$  content, and size of the coal used.

E. H. M. B.

**Reactions involved in hydrogenation-cracking of [coal] tar.** C. M. CAWLEY and C. C. HALL (Chem. & Ind., 1934, 806–811).—A summary of work already published (cf. B., 1932, 762; 1933, 260; 1934, 53). The main types of reaction are: (1) deoxygenation, including subsequent dehydration or condensation, e.g.,  $PhOH \rightarrow C_6H_6$ ,  $PhOH \rightarrow C_6H_{11}OH \rightarrow C_6H_{12}$ , and  $2PhOH \rightarrow Ph_2$ ; (2) hydrogenation prior to cracking,

e.g.,  $C_{10}H_8 \rightarrow C_{10}H_{12} \rightarrow PhBu^a \rightarrow PhEt$ ; and (3) cracking. If the speed of (2) is  $\ll$  that of (3) stable mols. are formed; if otherwise, destructive decomp. or polymerisation results.

H. A. P.

**Influence of paraffin wax on bitumen.** J. MANHEIMER (Proc. World Petroleum Congr., 1933, 2, 553–556).—Determinations of the physical and chemical properties, and microscopical examination, of a no. of samples of bitumen (I) have shown that paraffin wax (II) is an undesirable constituent thereof. On oxidation of (I) the (II) content decreases. Oxidation is regarded as an artificial ageing of (I), and an examination of its properties before and after 16 hr. oxidation in a current of air at  $100^\circ$  is suggested as a useful means of judging the val. of the material. Data relating to the properties of various (I) before and after oxidation are tabulated (cf. following abstract).

A. B. M.

**Determination of hard and soft asphalt, resins, and the refined oily constituents, including paraffin wax, in bitumen.** W. MAASS (Proc. World Petroleum Congr., 1933, 2, 557–572; cf. preceding abstract).—The methods of analysis are described (cf. B., 1933, 418) and the analyses of a no. of representative bitumens tabulated. Photomicrographs showing bitumen surfaces and the forms assumed by the paraffin wax when using different methods of separation are illustrated.

A. B. M.

**Tests on bituminous emulsions for road use.** TECHN. COMMEE. OF THE ROAD EMULSION & COLD BITUMINOUS ROADS ASSOC. (Proc. World Petroleum Congr., 1933, 2, 674–681).—The tests prescribed in Brit. Standards Inst. Spec. No. 434–1931 for (a) bitumen content, (b) emulsifier content, (c) quality of bitumen used, (d) suitability for spraying, (e) stability on storage, (f) viscosity, and (g) resistance to low temp. are described. A method for evaluating the general-performance stability of emulsions on the road, which consists in concentrating a sample of the emulsion by stirring it in a gentle current of air at  $18$ – $20^\circ$  until it coagulates and then determining its  $H_2O$  content (the lower this is the more stable is the emulsion), appears to give a useful indication of this property and has been adopted by the Technical Committee. Methods are also given for the determination of the dispersion of emulsions, by microscopical examination, but these are not yet sufficiently standardised for adoption. The methods of test adopted in Germany, United States, France, and Holland are critically reviewed.

A. B. M.

**Evaluation of German crude petroleum according to the production depth and geological formation.** H. STEINBRECHER and O. STUTZER (Petroleum, 1934, 30, No. 39, 1–3).—Crude petroleum obtained from different depths of the same geological formation, as well as from different formations, differ in constitution and physical properties. Results [ $d$ ,  $\eta$  (Engler), b.p., paraffin and asphalt contents] for 28 German crude oils are tabulated.

C. C.

**Theoretical comparison of petroleum distillation systems.** (A) P. J. ROELFSEMA (J. Inst. Petroleum Tech., 1934, 20, 901–902). (B) F. C. CUTTING (*Ibid.*, 902–909). (c) P. MEYER (*Ibid.*, 909–911).—(A) The method of applying the McCabe and Thiele calculations

to fractional distillation, as described by Meyer (B., 1933, 1042), is challenged; it is suggested that the % of material in the bottoms which could theoretically be included in the overhead is a more satisfactory numerical expression for the degree of fractionation.

(B) Data are presented to show that results calc., in the reference paper (*loc. cit.*), to be obtainable from continuous distillation may be considerably in error.

(C) A reply. H. S. G.

**Use of adsorption phenomena in purifying and refining petroleum products.** DUBRISAY (Ann. Off. nat. Comb. liquides, 1933, 8, 607—618; Chem. Zentr., 1933, ii, 3941).—The order of decreasing efficacy of adsorbents in removing tar from benzene was: animal charcoal, wood charcoal (I),  $\text{SiO}_2$  from  $\text{SiF}_4$ , kaolin dehydrated at  $500^\circ$ ,  $\text{Al}_2\text{O}_3$  (II), and kieselguhr. (I) and talc removed  $\text{NH}_2\text{Et}$ . (II) and  $\text{Fe}_2\text{O}_3$  removed  $\text{H}_2\text{S}$ . The order of decreasing efficacy in removing mercaptans was: (I), (II),  $\text{Fe}_2\text{O}_3$ ,  $\text{SiO}_2$ , talc, and kaolin. Hexadecene was removed only by  $\text{SiO}_2$ . H. J. E.

**Comparison of cracking and hydrogenation as methods of producing gasoline.** R. T. HASLAM, R. P. RUSSELL, and W. C. ASBURY (Proc. World Petroleum Congr., 1933, 2, 309—316).—A comparison of data relating to the cracking and to the hydrogenation of five representative gas oils shows that (a) both high- and low-temp. hydrogenation processes give substantially higher yields of gasoline than does cracking, (b) since only a caustic wash is required to refine the gasoline from the hydrogenation process, treating losses and losses of  $\text{C}_8\text{H}_{18}$  rating are negligible with these products; (c) although the  $\text{C}_8\text{H}_{18}$  nos. of the cracked gasolines are, in general,  $>$  those of the hydrogenated gasolines, the advantage is counterbalanced by the superior Pb-susceptibility and "non-fading" properties of the hydrogenated fuels. The economic position of the hydrogenation process is markedly affected by gasoline prices; when these are low hydrogenation offers no, or only small, advantages over cracking, but high prices are favourable to the former process. The low-temp. appears to be economically more advantageous than the high-temp. hydrogenation process. Gas oils and cycle stocks of low  $\text{NH}_2\text{Ph}$  point and high boiling range are the best initial materials for hydrogenation. Methods of operation wherein the two processes can be advantageously combined are discussed. A. B. M.

**Direct and indirect use of methane as motor fuel.** C. PADOVANI (Proc. World Petroleum Congr., 1933, 2, 795—798).—The possibility of using compressed  $\text{CH}_4$  as a motor fuel, or of converting it into liquid fuel, e.g., by direct thermal treatment or by conversion into a mixture of CO and  $\text{H}_2$  which would then serve as the initial material for the synthesis of alcohols or light hydrocarbons, is discussed with particular reference to conditions in Italy. A. B. M.

**Use of coal-tar oils in internal-combustion engines.** H. M. SPIERS and E. W. SMITH (Gas J., 1934, 207, 642—649).—The experiences of various users of coal-tar oils for motor fuel are summarised. For spark-ignition engines light creosote is washed to reduce tar acids, and  $\text{C}_{10}\text{H}_8$  is removed or else solvent naphtha

is added. A large choke on the carburettor and a high compression ratio are advantageous. Castor oil is the best lubricant. Petrol from a separate tank is used for starting and when the motor is idling. Tar oils are considerably cheaper than petrol, but maintenance charges seem to be higher and exhaust gases may be unpleasant. For stationary engines they are very satisfactory, but the spontaneous-ignition temp. are too high for use in compression-ignition engines without the addition of pro-knocks or ignition catalysts.

E. H. M. B.

**Dehydrating alcohol fuel with gypsum.** T. WALLIS (Petroleum, 1934, 30, No. 41, 3—4).—In a continuous process developed by the I. G., EtOH is dehydrated in the vapour phase by countercurrent treatment with powdered anhyd. gypsum. The latter is transformed into the hemihydrate, which passes to a drum in which  $\text{CaSO}_4$  is regenerated by hot air. 99.8—100% EtOH is produced. C. C.

**Determining the density of benzene in a benzene-spirit mixture.** B. LAMPE (Z. Spiritusind., 1934, 57, 252, 254).—50 c.c. of motor spirit are shaken with 50 c.c. of distilled  $\text{H}_2\text{O}$ , or, better, with aq.  $\text{CaCl}_2$  solution, in a stoppered cylinder. Any EtOH present passes into the aq. layer, and the increase in vol. of this  $\times 2$  gives the vol.-% EtOH. If the  $d$  of the upper layer is 0.87—0.88, it consists of benzol alone. If, however, colour or other tests show only benzene to be present in this layer, its  $d$  may be found from the original mixture by a method proposed by Kreuss (Böhm. Bierbrauer, 1934, 61, 269), dependent on the fact that a mixture of liquids of differing  $d$  will have an intermediate  $d$  depending on the proportions of the two constituents present, provided that no appreciable contraction takes place on mixing. The method is sufficiently accurate to distinguish between light and heavy benzene. I. A. P.

**Determination of lead soap in oils and greases.** H. LEVIN (Ind. Eng. Chem. [Anal.], 1934, 6, 333).—The sample is refluxed with  $\text{C}_6\text{H}_6$  and filtered hot from inorg. Pb salts (if any). AcOH (0.5 pt. by vol.) is added, the mixture refluxed for 1 hr., and the  $\text{Pb}(\text{OAc})_2$  formed removed by  $\text{H}_2\text{O}$ . Pb is determined in the aq. extracts as chromate. R. S. C.

**Investigation of industrial gases by the low-temperature technique.** E. BERL and W. FORST (Z. anal. Chem., 1934, 98, 305—326).—Hydrocarbon mixtures up to  $\text{C}_4$  may be rapidly and quantitatively separated by fractional distillation or (better) fractional condensation in high vac. Suitable design of apparatus and technique are described. J. S. A.

**Cyclic components of paraffin wax.** J. MÜLLER and S. PILAT (Nature, 1934, 134, 459).—Paraffin wax has been separated into four fractions, viz., (I) m.p.  $76^\circ$ ,  $\text{C}_n\text{H}_{2n+1}$ ; (II) m.p.  $63^\circ$ ,  $\text{C}_n\text{H}_{2n}$ ; (III) m.p.  $57^\circ$ ,  $\text{C}_n\text{H}_{2n-2}$  and (IV) m.p.  $50^\circ$ ,  $\text{C}_n\text{H}_{2n-3}$ . (I) is a mixture of true paraffins with some cyclic hydrocarbons (V) which are probably the chief constituents of (II), (III), and (IV).  $d$ ,  $n$ , and temp.- $\eta$  data provide further evidence of the presence of (V) in undistilled paraffin wax. L. S. T.

**Synthetic lubricating oils from condensation products of ethylene.** H. SCHILDWÄCHTER (Angew.

Chem., 1934, 47, 677—681).— $C_{10}H_8$  (85%), tetralin (27%), and coal-tar oils (33%) are converted into lubricating oils (I) when treated in presence of 5% of  $AlCl_3$  with  $C_2H_4$  under pressure (60 atm./20°) at 100—180°, 250—260°, and 290—330°, respectively; the yields are given in parentheses. (I) are similar in properties to standard motor oils, possessing low f.p. and high  $\eta$  for oils of moderate mol. wt. They are stable towards oxidation and high temp. and contain no asphalt. When submitted to the action of an electric discharge (suitable laboratory apparatus described),  $\eta$  increases still further. S. C.

**Carbon-forming tendencies of lubricating oils.** W. A. GRUSE (Proc. World Petroleum Congr., 1933, 2, 441—443).—The bulk of the C deposited in petrol engines is derived from the lubricating oil by (a) slow oxidation of crank-case oil to resins etc., (b) low-temp. carbonisation of these products in piston-ring grooves and on the piston, and (c) rapid cracking of these products and of unvolatilised oil on the surface of the combustion chamber. Attempts to correlate C formation with the results of oxidation (O), coking (C), and volatility tests (V) have not, however, been uniformly successful. O have not yet established any significance for ordinary service. Carefully controlled C indicate that there is a relationship between Conradson C residue and C formation in the engine per vol. of oil consumed. V indicate that the higher is the 90% distillation point the greater is the coke formation, but exceptions to this rule are possible. A. B. M.

**Properties of hydrogenated motor oils.** R. T. HASLAM, R. P. RUSSELL, and W. C. ASBURY (Proc. World Petroleum Congr., 1933, 2, 302—308).—The hydrogenation of lubricating oils results in the practically complete elimination of O, N, and S, the conversion of asphaltic and carbonaceous material into stable hydrocarbons, and the addition of H to unsaturated and aromatic hydrocarbons; there is also some tendency to split open and further hydrogenate ring- or naphthenic-type compounds. The viscosity indices are improved and may attain vals. = those of the best natural lubricating oils. The hydrogenated lubricants have high flash points, excellent colour, high A.P.I. gravities, and low Conradson C contents. Extensive engine tests have shown that the hydrogenated oils give satisfactory cold-starting performance, lower consumptions than the best natural lubricants, and low C and sludge formation. Tests on a machine designed to study bearing failures at high speed show these oils to have lubricating properties  $\ll$  those of castor oil blends for this type of lubrication. A. B. M.

**Oxidation of lubricating oils.** E. A. EVANS (Proc. World Petroleum Congr., 1933, 2, 460—461).—The stability of an oil to oxidation appears to be of more importance than "oiliness" in so far as aëro-engines are concerned. The oxidation of oils was studied by heating a small quantity in air in a rotating, sealed glass tube and subsequently determining the amount of  $O_2$  which has been absorbed. With well-refined mineral oils absorption of  $O_2$  was very low at  $< 125^\circ$ , but in presence of Fe rust appreciable oxidation occurred in 24 hr. The rate of oxidation at  $150^\circ$  was

tenfold that at  $125^\circ$ , and above  $150^\circ$ , even with the best oils, 1 g. depleted the  $O_2$  in 100 c.c. of air in 1—2 days. Above  $200^\circ$  the  $O_2$  was depleted in a few hr., whilst above  $250^\circ$  inflammation was liable to occur. Large increases in rate of oxidation were observed when 1% of Fe rust, Al or Ag powder, ZnO, etc. was added to the oil. The org. dopes, though effective below  $150^\circ$ , appeared to be of little use at higher temp. A. B. M.

**British Air Ministry oxidation test for lubricating oils.** F. H. GARNER, C. I. KELLY, and J. L. TAYLOR (Proc. World Petroleum Congr., 1933, 2, 448—457).—The test (Air Ministry Spec. D.T.D. 109) consists in passing air at 15 litres/hr. for 12 hr. through 40 c.c. of the oil contained in a boiling-tube supported in an oil-bath (B) ( $200^\circ$ ), and determining the resulting changes in  $\eta$  and coke no. It has been shown that no appreciable error is produced by (a) permitting a temp. variation of  $\pm 0.5^\circ$  instead of  $\pm 0.1^\circ$  as fixed in the specification, (b) an error of 10 min. in the time of oxidation, (c) slight variations in air rate and vol. of oil, (d) increased agitation of B, or (e) not sheathing the emergent length of the oxidation tube. The permissible variations in the dimensions of the different parts of the apparatus are defined. With certain types of oil the  $\eta$  ratio can be altered by mechanical treatment or varied thermal history, and a standard procedure is suggested which gives uniform results with such oils. Attention is directed to the sensitivity of the test to slight contamination, e.g., with Fe salts, which may occur if tins with soldered seams are used to contain the sample, and glass bottles are recommended for this purpose. A. B. M.

**Flash point of lubricating oils.** W. STEINITZ (Petroleum, 1934, 30, No. 41; Motorenbezt., 7, 5—7).—The val. of flash-point (I) determinations for the evaluation of lubricating oils (II) is discussed. An examination of (II) for bearings, steam-cylinders, compressors, and internal-combustion engines showed that of two oils for the same purpose, the one with lower (I) often proved superior in practice. C. C.

**Beneficial use of tin compounds in lubricants.** E. W. J. MARPLES (Tech. Publ. Internat. Tin Res. and Dev. Counc., Aug., 1934, C, No. 2, 5 pp.).—The deterioration of lubricating oils, due to sludge formation caused by oxidation, is inhibited by adding Sn oleate,  $SnPh_4$ , or certain other compounds of Sn. Compounds of Au, Hg, Cr, Ce, Bi, or Sb exert little effect, whilst compounds of Cu, Fe, Ag, V, and Mn accelerate the rate of deterioration. E. S. H.

**Lubricating grease.** H. S. GARLICK (J. Inst. Petroleum Tech., 1934, 20, 829—873).—A discussion of modern lubricating greases, their manufacture, characteristics, and testing. H. S. G.

**Employment of recovered used oils in power-driven vehicles.** E. GRAEFE (Petroleum, 1934, 30, No. 41; Motorenbezt., 7, 2—4).—The nature of used lubricating oils, with particular reference to motor oils, is discussed together with the economy which would result if such oils were again used after removal of solid impurities,  $H_2O$ , residual petrol, etc. C. C.

**Nomograms for gas industry. Crushing and grinding [of coal]. Changes in  $\eta$  of liquids.**—See I.

**Dielectric constants.**—See XI. [Fuel from] esterification of olive oils.—See XII. Anthracene oil as insecticide.—See XVI. Clarifying polluted [coal-washery]  $H_2O$ .—See XXIII.

## PATENTS.

**Carbonisation apparatus.** H. O. LOEBELL, Assr. to H. L. DOHERTY (U.S.P. 1,944,749, 23.1.34. Appl., 18.3.29).—A hollow drum (*A*) rotates within a closed retort (*B*). A heat-resistant, endless-belt conveyor (*C*) encircles *A* and an idler drum (*D*) mounted within *B* parallel to *A*. Coal is fed on to the lower flight of *C* by means of a conveyor trough so that, as *A* rotates, *C* compresses the layer of coal against the outer surface of *A*. The opposed surfaces of *A* and *C* are so shaped that the coal is compressed into flat briquettes. The tension in *C* and thereby the pressure to which the coal is subjected can be regulated by moving *D*. *A* is internally heated and *B* externally heated over the surface opposed to *A*. The briquettes are discharged from *A* into a trough and thence, by means of a screw conveyor, out of *B*. A. B. M.

**Apparatus for producing coke and gas.** W. A. RIDDELL, Assr. to FREDERICK IRON & STEEL Co. (U.S.P. 1,944,192, 23.1.34. Appl., 26.12.30).—Coal is fed from a hopper into a horizontal gasification chamber (*A*) by means of a screw conveyor. Air and steam are simultaneously introduced into *A*. The walls of *A* are perforated so that the gases formed therein pass through into an annular chamber (*B*) surrounding *A*, and thence into a gas accumulator (*C*). Surrounding *B* is an annular combustion chamber wherein part of the gas produced is burned to provide further heat for the carbonisation and gasification processes. The hot coke is discharged into a vertical pipe passing down through *C* into a quenching tank at the bottom thereof. Means are provided for continuously withdrawing the quenched coke. A. B. M.

**Manufacture of carbon black.** G. C. LEWIS, Assr. to COLUMBIAN CARBON Co. (U.S.P. 1,944,715, 23.1.34. Appl., 28.6.30).—A hydrocarbon flame is caused to impinge on a metallic surface which is maintained at a high temp., preferably by checking the loss of heat from those portions of the metal not acted on by the flame. A metallic plate backed by heat-insulating material may be used. A. B. M.

**Revivification of spent activated carbon.** R. G. DAVIS, Assr. to DARCO CORP. (U.S.P. 1,945,479, 30.1.34. Appl., 12.11.32. Cf. U.S.P. 1,873,998 and 1,875,223; B., 1933, 579).—The apparatus comprises a vertical retort (*A*), which may be externally or internally heated, surmounted by a mixing chamber (*B*), and communicating at its base with a collecting tank for the revived material. The spent *C* and preheated air are introduced into *B* and pass thence down *A*. The *C* ignites in *A*, the temp. and air supply therein being so regulated that adsorbed matter on *C* is burned without any substantial loss of the original activated *C*. The temp. is regulated by the introduction of steam into *A* at suitable points. A. B. M.

**Apparatus for making gas.** J. WHITEHART (U.S.P. 1,944,818, 23.1.34. Appl., 17.9.32).—The apparatus

comprises a supply of compressed air, two tanks (*A*, *B*) containing volatile liquid fuel and oil, respectively, and a mixing coil (*C*). The air and liquid fuel are supplied to a specially designed mixer, are passed thence through *C* to become thoroughly commingled, and the mixture is bubbled up through the liquid fuel in *A*. The gas withdrawn from *A* passes through the oil in *B*, whereby it is further cleaned and stabilised. A. B. M.

**Manufacture of carburetted water-gas.** G. J. NORDMEYER and T. W. STONE, Assrs. to KOPPERS Co. of DELAWARE and to WESTERN GAS CONSTRUCTION Co. (U.S.P. 1,947,792, 20.2.34. Appl., 16.5.31).—An apparatus comprising a generator (*A*), carburettor (*B*), and superheater (*C*), in which *B* and *C* are filled with chequerwork, is operated on the following cycle: (1) Forward air blast in *A*, secondary air admitted to *B*, gases to waste-heat boiler (*D*) or the like. (2) Backward air blast through *C*, *B*, *A* to remove *C* deposited during (3*b*), gases mostly removed to *D* through apertures in the wall of *A* not far below the top of the fuel bed, i.e., at a point where the CO content will be a min. (3*a*) Forward steam-run producing water-gas. (3*b*, simultaneous with 3*a*) Heavy oil is sprayed downwards upon the fuel bed in *A* and chequerwork in *B*. (4) A short steam back-run. (5) A short steam forward run. (6) Cycle (1) is repeated. B. M. V.

**Producer gas process using sodium carbonate.** A. H. WHITE and D. A. FOX (U.S.P. 1,948,085, 20.2.34. Appl., 26.3.31).—The generator may advantageously be operated at a lower temp. by the presence in the fuel of < 10% of a Na compound, which is stated to act, not as a catalyst, but as transfer agent in a cycle of reactions. The temporary presence of Na vapour is claimed in the ratio  $3CO : 2Na$ . B. M. V.

**Manufacture of oil gas.** C. ROSIER (U.S.P. 1,945,790, 6.2.34. Appl., 26.5.26).—Oil is gasified in presence of a catalyst, and further heated; air is separately preheated and injected into the oil vapour. B. M. V.

**Gas scrubbing.** H. R. FIFE, Assr. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,944,122, 16.1.34. Appl., 29.7.32).—When treating gases with an aq. solution of an amine, e.g., an ethanamine, the tendency of the solution to foam is suppressed by continuously circulating a portion thereof through activated *C*. It is advantageous also to add a small proportion of fatty oil to the solution. A. B. M.

**Treatment of coal-distillation gases.** S. P. MILLER, Assr. to BARRETT Co. (U.S.P. 1,944,523, 23.1.34. Appl., 4.12.28).—Gases from coal-carbonisation plants etc. are subjected to electrical pptn. while still at a high temp., deposition of non-fluid pitch or *C* on the precipitator (*P*), or carbonisation of pitch thereon, being prevented by continuously flushing the surface with tar or other suitable liquid medium. The gases may pass directly from the collector main to *P*, or may first be brought into intimate contact with tar or pitch to be distilled. The cleaned gases pass to the condensing system. A. B. M.

**Treatment of artificial gas.** W. L. SHIVELY, Assr. to KOPPERS Co. of DELAWARE (U.S.P. 1,945,001,

30.1.34. Appl., 20.1.31).—Gum deposition from coke-oven gas, carburetted water-gas, etc. which is transmitted over long distances under pressure is prevented by maintaining a high R.H. in the gas by introducing  $H_2O$  or steam into the high-pressure main at suitable points.

A. B. M.

**Removal of naphthalene and gum-forming constituents from gas.** A. R. POWELL, Assr. to KOPPERS Co. OF DELAWARE (U.S.P. 1,944,903, 30.1.34. Appl., 22.7.30).—The gas is scrubbed with a large vol. of oil, e.g., up to 3000 gals. per  $10^6$  cu. ft. of gas, the amount used being such that it is in equilibrium with the gas with respect to a predetermined content of light oil, is large in comparison with the vol. required for the removal of the  $C_{10}H_8$ , and is sufficient to remove substantially the whole of the gum-forming constituents.

A. B. M.

**Removal of ammonia and hydrogen sulphide from gases.** C. J. HANSEN, Assr. to KOPPERS Co. OF DELAWARE (U.S.P. 1,944,978, 30.1.34. Appl., 25.1.32. Ger., 24.1.31).—The gas is cooled and washed with  $H_2O$  to remove  $NH_3$ . The solution is worked up in known manner to give a more conc. (e.g., 12%) aq.  $NH_3$ , which is then treated with  $SO_2$ . The sulphite and bisulphite solution so formed is used to wash out part of the  $H_2S$  from the gas. The spent solution, containing  $NH_4$  thiosulphate (I) and S, is treated with  $H_3PO_4$  to give a mixture of  $NH_4$  phosphate (II) and polythionate (III) and S, and this solution is then used to wash out the residual  $H_2S$  from the gas, the (III) being thereby converted into (I) and S. The spent solution from this stage is treated with  $H_3PO_4$ , filtered, and heated under pressure to about  $150^\circ$  to give a mixture of (II) and  $(NH_4)_2SO_4$  suitable for use as a fertiliser (cf. U.S.P. 1,855,856; B., 1933, 324).

A. B. M.

**Treatment of gas to recover carbon dioxide.** M. SHOELD, Assr. to KOPPERS Co. OF DELAWARE (U.S.P. 1,945,002, 30.1.34. Appl., 5.5.31).—Hot aq.  $K_2CO_3$  is sprayed into a tower ( $T_1$ ) packed with contact material down which it passes countercurrent to gas, initially cold, containing  $CO_2$ . The solution absorbs the  $CO_2$  and at the same time is cooled. The resulting bicarbonate (I) solution is then passed into a second tower ( $T_2$ ) wherein it is brought into direct contact with steam to decompose the (I) with evolution of the absorbed  $CO_2$ , which is passed to storage. The hot solution is then recirculated. The process is so regulated that the amount of steam condensed in  $T_2$  is re-evaporated in  $T_1$  so that the requisite concn. of the solution is maintained automatically.

A. B. M.

**Production of tar acids.** W. C. COOPER, JUN., Assr. to BARRETT Co. (U.S.P. 1,944,681, 23.1.34. Appl., 7.1.31).—Phenoxide (I) solutions obtained by extracting tar acids (II) from tars or tar oils by means of alkali are treated with sufficient  $CO_2$  to convert the alkali into the normal carbonate without forming any substantial amount of bicarbonate. The crude (II) are separated from the aq. solution and, if desired, may be subjected to further treatment with  $CO_2$  and again separated from the aq. bicarbonate solution formed. The separated (II) are then washed repeatedly with  $H_2O$

to remove sol. carbonate and (I). When the wash- $H_2O$  is sufficiently conc. in (I) it is added to a further batch of the original (I) solution.

A. B. M.

**Bituminous emulsion.** L. KIRSCHBRAUN, Assr. to PATENT & LICENSING CORP. (U.S.P. 1,948,881, 27.2.34. Appl., 26.5.28).—Two emulsions (I, II) are made and mixed, (I) containing a liquefiable bitumen, clay, and  $H_2O$ , and (II) a bitumen of substantially the same m.p., soap or Na silicate, and  $H_2O$ , (II) being easily broken by electrolytes and (I) stable, coalescing only by removal of  $H_2O$ . The setting properties of the mixture (M) are adjustable by varying the proportion of (I) to (II), and M is less viscous than either constituent.

B. M. V.

**Removal of phenols from liquors containing same.** C. E. BRAUN, Assr. to BARRETT Co. (U.S.P. 1,937,941, 5.12.33. Appl., 25.1.30).—The phenolic solution is treated at  $60-80^\circ$  with coal-tar oil ( $d$  1.04—1.13) containing its original content of tar acids.

A. R. P.

**Producing a hydrocarbon and metallic sol.** J. C. ARNOLD (U.S.P. 1,933,836, 7.11.33. Appl., 11.12.31).—Metal is dispersed directly in hydrocarbon fluid, e.g., gasoline, by passing a high-frequency current across a gap in the fluid. The latter passes through a vertical tube made of insulating material, the electrodes comprising a series of metal baffle plates, preferably alloy, one metal being disruptible under the conditions obtaining.

C. C.

**Continuous distillation of hydrocarbons or hydrocarbon products.** A. WAGNER (B.P. 413,877, 31.7.33. Ger., 1.8.32).—Heated hydrocarbon oil at, e.g.,  $350^\circ/30$  atm. is introduced into the bottom of a fractionating column (C) through a vertical pipe (P) at the upper end of which is a conical valve (V) with an adjustable load. Surrounding P is another pipe and through the annular space superheated steam (or other gas) is introduced into C. The reduction in pressure when the oil passes V causes the oil to atomise and mix with the steam, thereby causing vaporisation. The vapours pass through a tubular guiding chamber and then up C.

D. K. M.

**Hydrogenating and distilling oil shale.** R. H. MCKEE (U.S.P. 1,941,809, 2.1.34. Appl., 12.10.29).—To obtain crude motor fuel and a combustible gas of high calorific val., finely-ground oil shale (or other carbonaceous material, e.g., lignite), preheated to  $300^\circ$ , is rapidly passed suspended in steam (or in hydrogenous gaseous fluid, e.g., high-pressure water-gas), through a Ni-Cr steel tube under pressure (1000—2500 lb. per sq. in.), the first half of the tube being maintained at  $900^\circ$ , and the second half at  $250-450^\circ$ . The Ni-Cr catalyses the reaction, but additional catalysts may be used to modify the end-products. Hydrogenation is facilitated by the water-gas formed from steam and the C residue from the shale.  $CO_2$  is a by-product.

C. C.

**Hydrogenation system [for petroleum oils].** W. B. PLUMMER, Assr. to STANDARD OIL Co. (U.S.P. 1,942,120, 2.1.34. Appl., 17.7.30).—Low-grade petroleum oils are converted into gasoline or lubricating oil etc. by heating with  $H_2$  at  $455^\circ/150-300$  atm. The hot gases and liquids are separated and uniformly

distributed to parallel catalyst tubes, 30—40 ft. long, 12 in. diam., made of a resistant metal lining over which thick-walled tube is shrunk. C. C.

**Purification of petrol and like oils and spirits.** REFINERS, LTD., and P. J. McDERMOTT (B.P. 413,412, 15.2.33).—Petrols etc. are refined by treatment with a mixture of fuller's earth,  $\text{Fe}_2(\text{SO}_4)_3$  (I), and free  $\text{H}_2\text{SO}_4$  (II) in which the ratio of (II) to (I) is  $> 0.35\%$  (0.45—0.80%). The spirit is mixed with the reagent at above  $45^\circ$ . C. C.

**Purification of liquid hydrocarbons with liquid sulphur dioxide or mixtures thereof.** EDELEANU GES. M.B.H. (B.P. 413,465, 2.5.33. Ger., 4.5.32).—The cost of the apparatus and of the operation of the Edeleanu plant is reduced and the manipulation simplified by rendering the extraction and evaporation processes automatic by using level regulators in combination with pressure- and temp.-regulating devices. C. C.

**Recovery of carbon disulphide and methyl alcohol from aqueous solutions obtained in purification of benzol with methyl alcohol and caustic alkali.** NAT. BENZOLE CO., LTD., W. H. HOFFERT, and E. G. HANCOCK (B.P. 416,404, 6.3.33. Cf. B.P. 414,644; B., 1934, 918).—The solutions are neutralised by (addition to) the calc. amount of acid ( $\text{H}_2\text{SO}_4$ ,  $\text{SO}_2$ ) and fractionally distilled, the first fractions containing the  $\text{CS}_2$  being collected under  $\text{H}_2\text{O}$ . H. A. P.

**Refining of hydrocarbons.** IMPROVED HYDRO-CARBON PROCESSES, LTD. (B.P. 413,719, 20.12.32. Addn. to B.P. 387,447; B., 1933, 340).—The hydrocarbons are oxidised by ozonised air in presence of a catalyst followed by treatment with a mixture of  $\text{H}_2\text{SO}_4$  and  $\text{FeCl}_3$  or  $\text{Fe}_2(\text{SO}_4)_3$ . The catalysts include the stearates, oleates, and resinates of Fe, Mn, or V, and also aldehydes or ketones which give unstable peroxides, e.g., acetaldehyde, citral, phorone, pinacolin. C. C.

**Refining a benzenoid hydrocarbon oil.** A. KEMPER, Assr. to KOPPERS CO. OF DELAWARE (U.S.P. 1,942,062, 2.1.34. Appl., 2.7.30. Ger., 26.4.30).—A continuous process for refining crude benzol comprises a series of tanks for treating with  $\text{H}_2\text{SO}_4$  ( $d$  1.70—1.81), settling, neutralising, and settling. By agitating the  $\text{H}_2\text{SO}_4$ -oil mixture in an additional tank with  $\text{H}_2\text{O}$  (2 vols. of  $\text{H}_2\text{O}$  per 1 vol. of  $\text{H}_2\text{SO}_4$ ), dil. acid is recovered free from resins. C. C.

**Desulphurising, decolorising, deodorising, and degumming hydrocarbons, and providing a protective buffer for apparatus.** H. T. DARLINGTON, Assr. to OIL CORP. OF AMERICA (U.S.P. 1,944,170, 23.1.34. Appl., 24.11.26. Renewed 23.3.33).—Desulphurising agents, e.g., oxides, hydroxides, or sulphates of Cu, Fe, Ni, and Ca, are colloiddally dispersed in the hydrocarbon liquid before or during cracking. At high temp., e.g., 315— $480^\circ$ , the oxides etc. are reduced to metal which is attacked by the S, thus preventing corrosive attack of apparatus and containers. Decolorising, deodorising, and degumming take place at the same time. C. C.

**[Gum inhibitor for] motor fuel.** A. O. JAEGER, Assr. to SELDEN CO. (U.S.P. 1,941,689, 2.1.34. Appl., 20.7.31).—The use of succinimide is claimed. H. A. P.

**Odourising liquefied petroleum gases.** F. E. FREY, Assr. to PHILLIPS PETROLEUM CO. (U.S.P. 1,944,175, 23.1.34. Appl., 7.10.29).—The liquefied gases, e.g.,  $\text{C}_3\text{H}_8$  or  $\text{C}_4\text{H}_{10}$ , are odourised with (1) volatile substances, e.g.,  $\text{C}_3\text{H}_6$ , (2) miscible materials of slightly higher b.p. than that of the liquid, e.g.,  $\text{Me}_2\text{S}$ , (3) volatile compounds of limited solubility, e.g.,  $\text{NH}_3$ . Sufficient material is added to odourise the leanest inflammable mixture of gas and air. To prevent an excess of odouriser in the delivered gas, a retaining agent is added, e.g., in cases (1) and (2) kerosene, and in case (3) a liquid of limited miscibility, e.g., glycol. C. C.

**Treatment of [oil-water] emulsions.** W. F. V. LOENEN, Assr. to L. BLAKE-SMITH (U.S.P. 1,944,479—80, 23.1.34. Appl., 21.5.32).—Continuous processes in which emulsions (I), e.g., petroleum emulsions, are resolved by treatment with granular solids (II) specifically heavier or lighter than (I) are claimed. (II) are released at a higher or lower level, respectively, to fall or rise by gravitation through (I) without additional agitation. They may be preferentially wetted by  $\text{H}_2\text{O}$ , or may consist of two substances, one preferentially wetted by  $\text{H}_2\text{O}$  and the other by oil. The solids which gradually separate are returned mechanically to the initial layer. Resolved products are removed, but an aq. layer may be maintained to scrub the returned solids. C. C.

**Production of aqueous dispersion of montan wax and other water-immiscible bodies.** O. F. NETZKE, Assr. to BENNETT, INC. (U.S.P. 1,940,431, 19.12.33. Appl., 7.12.29).—Free-flowing aq. dispersions of mixtures of montan wax (I) with asphalts, solid paraffins, or similar materials are produced by mixing hot dil. solutions of alkali (of concn.  $< 2\%$  NaOH or its equiv.) with the melted mixed materials. The proportion of (I) may be low, and no stabiliser other than the saponifiable products of (I) are used. Dispersions of (I) and paraffin are chilled immediately after formation. C. C.

**Obtaining hydrocarbon oils from heavier unrefined products by the action of high-pressure hydrogen.** H. H. SEMMES, Assr. to STANDARD-I.G. CO. (U.S.P. 1,940,653, 19.12.33. Appl., 30.1.30).—A plant and process are claimed whereby hydrocarbon oils containing S are hydrogenated at  $> 370^\circ / > 20$  atm. in presence of Ni, Co, Fe, or their oxides. When the catalyst becomes poisoned, the oil is diverted to a second reactor, the catalyst in the first being regenerated by roasting. For the production of gasoline, fractions boiling at  $> 200^\circ$  are recirculated. To obtain high-grade lubricating oils from Texas, Coastal, or Columbian crudes a large excess of  $\text{H}_2$  is used at  $< 455^\circ / > 50$  atm. (100—200 atm.). C. C.

**Processing of hydrocarbon oils.** J. C. BLACK (U.S.P. 1,945,508, 6.2.34. Appl., 21.1.30).—Hydrocarbon oil (I) is cracked at  $425$ — $480^\circ$ , lower-boiling constituents are removed by pressure regulation, and the higher-boiling portion is re-treated at  $480$ — $650^\circ$  with injection of cool (I), whereby cyclic hydrocarbons of high anti-knock val. are produced. C. C.

**Treatment of mineral oil distillates.** N. V. DE BATAAFSCHE PETROLEUM MAATS. (B.P. 414,237,

27.9.33. Holl., 3.10.32).—The yield and quality of the lubricating oil formed by treating cracked distillates of high unsaturated hydrocarbon content with anhyd.  $\text{AlCl}_3$  are improved by pretreating the distillate with about 1% of anhyd.  $\text{AlCl}_3$  at  $\leq 60^\circ$ , removing the sludge formed, and, if desired, redistilling, before effecting the main polymerising treatment. A. B. M.

**Treatment of hydrocarbons [petrolatum].** J. D. ZIELEY, Assr. to AMAREL CORP. (U.S.P. 1,944,483, 23.1.34. Appl., 7.7.31).—Hydrocarbon oil is atomised by superheated steam and the mixture passed into a tower (T) in which air and superheated steam are passed at the top and bottom. The temp. in the central zone of T is  $536^\circ$ . The mixture flows up an annular space formed by a circular curtain inside T, then successively in at the bottom and out at the top of two reaction towers into the first of which air is introduced, through a scrubber and fractionating column, to a condenser and gas separator. The system operates under a pressure of 60–70 lb. per sq. in. Petrolatum so treated yields a paraffin, and a series of low-boiling oils of oxygenated character. D. K. M.

**Treatment of hydrocarbon materials containing sulphurous impurities.** H. W. FISHER, Assr. to STANDARD-I. G. Co. (U.S.P. 1,944,639, 23.1.34. Appl., 20.11.29).—Hydrocarbon oil and  $\text{H}_2$  are passed at  $396\text{--}466^\circ$  and  $> 20$  (100–200) atm. through a tower packed with a catalyst, e.g., one or more oxides of Mo, Cr, W, alone or mixed with oxides of alkalis, alkaline earths, etc., whereby S present is converted into  $\text{H}_2\text{S}$ . After cooling and separating the gas, the oil is reheated to expel  $\text{H}_2\text{S}$  from solution. D. K. M.

**Treatment of hydrocarbon oils.** (A, B) J. C. MORRELL and G. EGLOFF, (C) J. C. MORRELL, (D) R. F. DAVIS, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,946,094–6 and 1,946,131, 6.2.34. Appl., [A, B] 16.2.31, [C] 25.3.32, [D] 11.2.31).—Vapours of low-boiling hydrocarbon distillates, e.g., from cracking, are subjected to the action of (A)  $\text{SO}_3$  and steam, or (B) phosphoric acids, together with gases containing  $\text{O}_2$ , e.g., air, ozonised air, etc., in presence of solid, non-metallic contact materials, e.g., fuller's earth. S and gum contents are reduced and colour is improved. (C) Hydrocarbons (I) of the gasoline range, particularly cracked (I), are refined in vertical towers by treatment in the vapour phase at  $120\text{--}315^\circ$  and under high pressure (up to several hundred lb./sq. in.) with an aq. solution of a borate, e.g., borax, steam being added to prevent undue concn. of the solution. (D) Dephlegmated cracked gasoline vapours are treated with dry  $\text{SO}_3$ , with or without inert gases, e.g.,  $\text{H}_2$ ,  $\text{CH}_4$ , or light hydrocarbon gases. Heavy products are separated and the vapours neutralised, treated with an earth, and rectified. C. C.

**Desulphurising hydrocarbon oils.** (A, B) F. W. SULLIVAN, JUN., and A. B. BROWN, (C) R. F. RUTHRUFF, Assrs. to STANDARD OIL Co. (U.S.P. 1,938,670–2, 12.12.33. Appl., [A] 15.12.28, [B, C] 5.7.29).—(A) Molten alkali metal (1–3 equivs. of the S in the oil) is dispersed in petroleum distillate containing gasoline, kerosene, and gas-oil fractions by means of a colloid mill and the resultant mixture is heated for 2–4 hr. at  $177\text{--}343^\circ$

(under pressure if the material is volatile). (B) Lower-boiling distillates (gasoline and kerosene fractions) of high S content, e.g., pressure distillate, are fractionally redistilled at  $93\text{--}163^\circ\text{F}$ ., at which point the bottoms are treated with alkali metal at  $176\text{--}343^\circ$   $> 150$  lb. per sq. in. The treated bottoms and original overhead are finally blended. (C) Hydrocarbon distillates of the gasoline and kerosene boiling ranges are heated with lump alkali metal, the proportion of which to S is  $> 1:1$  ( $> 3:1$ ), at  $> 200^\circ$   $> 150$  lb. per sq. in. Desulphurisation is improved by working in an inert atm., e.g.,  $\text{H}_2$  or  $\text{N}_2$ , and also by the addition of high-boiling, low-S oil. C. C.

**Neutralisation of [acid] hydrocarbon oils.** L. CALDWELL and R. T. HOWES, Assrs. to CALIFORNIA FIRST NAT. BANK OF LONG BEACH (U.S.P. 1,941,970, 2.1.34. Appl., 8.12.30).—Motor fuels of low gum content are obtained by neutralising acid-treated hydrocarbon oil with a granulated, substantially  $\text{H}_2\text{O}$ -insol. material (e.g.,  $\text{MgO}$ ) containing a dil. solution of  $\text{Mg}(\text{OH})_2$ . The neutralised oil (from this or other treatments) is vapourised and the vapours are treated with similar neutralising material. The condensed oil is treated with alkaline sweetening reagent, e.g., Na plumbite. C. C.

**Increasing the viscosity of mineral oils.** J. CRAIK, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 416,513, 17.3.33).—Thickened mineral oils which form gels at room temp. are obtained by dissolving  $\leq 2\%$  of a cellulose ester of a saturated fatty acid ( $\text{C}_{12}\text{--}\text{C}_{18}$ ) in the heated oil, e.g., cellulose stearate or palmitate. C. C.

**Improving hydrocarbon products, in particular lubricating oils.** J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 413,637, 13. and 26.1.33. Cf. B.P. 379,717; B., 1932, 971).—The properties of lubricating oils, lubricating greases, and paraffin waxes are improved by incorporating 0.5–10% of highly-polymerised vinyl compounds containing O, e.g., polymerised products of fatty acid vinyl esters, of vinyl ethers of BuOH, and of vinylcarboxylic acids such as acrylic acid derivatives. Alternatively, the monomeric compounds may be added to the hydrocarbon and the polymerisation performed *in situ* with the aid of accelerators (e.g.,  $\text{BF}_3$  or its double compounds), the polymerising agent being removed by subsequent refining. Treated lubricating oils have lower cold test and higher  $\eta$  index. C. C.

**Simultaneous production of high-grade motor fuels and lubricants from heavy hydrocarbons by the action of hydrogen.** R. T. HASLAM, Assr. to STANDARD-I. G. Co. (U.S.P. 1,944,236, 23.1.34. Appl., 25.1.30).—Gas rich in  $\text{H}_2$  is forced, at a rate  $> 10,000$  cu. ft. per barrel of oil fed, through heavy hydrocarbon oil, e.g., Texas, Coastal, Winkler, or Venezuelan crudes, at  $> 370^\circ$   $> 50$  atm. Lighter hydrocarbons, which are mainly unsaturated, are volatilised and the residue is treated again. The reaction zone may contain a catalyst immune from S poisoning, e.g., oxides of Cr, Mo, or W. Conditions are adjusted normally to yield 5–15% of high-grade naphtha. The lubricating oil obtained by refining the residue is equal to a Pennsylvanian oil. C. C.

**Manufacture of lubricating oils.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 415,526, 18.4.33. Addn. to B.P. 349,071; B., 1931, 832).—Aliphatic OH-compounds of high mol. wt ( $\neq C_{12}$ ), e.g., oleyl alcohol, stearyl glycol, etc., are treated with halogenating agents such as  $SOCl_2$ ,  $COCl_2$ ,  $PCl_3$  (whereby OH is substituted by halogen), followed by condensation, preferably with cyclic or olefinic compounds, e.g.,  $C_{10}H_8$ , tars, crude benzol, etc., in presence of  $AlCl_3$  or other condensing agents. 0.1–10% of the refined product is added to lubricating oil, grease, etc. to improve the set point and the temp.- $\eta$  curve. C. C.

**Production of lubricating oil.** U. B. BRAY, Assr. to UNION OIL CO. of CALIFORNIA (U.S.P. 1,944,491, 23.1.34. Appl., 10.1.33).—Lubricating oil is treated with a liquified paraffin, e.g.,  $C_2H_6$ , or casing-head gas under pressure, whereby asphalt is deposited. The oil is treated with acid and the latter and its reaction products are separated and then neutralised. The pressure is now released and part of the paraffin allowed to vaporise, thereby chilling the oil and depositing the wax, which is separated. D. K. M.

**Manufacture of green-bloom agents [for lubricating oils].** H. G. SMITH, Assr. to GULF REFINING CO. (U.S.P. 1,944,851, 23.1.34. Appl., 9.2.32).—A conc. bloom agent which when added to lubricating oil (0.2–5%) imparts a green fluorescence is prepared from  $AlCl_3$  sludges from the refining of hydrocarbon oils, especially those of a naphthenic character, by centrifuging or filtering to remove solid matter or by washing with dil.  $H_2SO_4$  followed by neutralisation. D. K. M.

**Purifying and regenerating used lubricating oil.** M. G. LEVI, A. MARIOTTI, and I. CIARROCCI (B.P. 413,537, 25.10.33. Italy, 26.11.32).—Used lubricating oil freed from  $H_2O$  is heated to 130–350° with solid anhyd.  $AlCl_3$  in a distilling apparatus with an agitator so that low-flash fractions are removed and asphaltic matter is pptd. simultaneously. The recovered oil is filtered with or without earth treatment. C. C.

**Making incandescence bodies for gas-lighting.** A. J. PRESTAGE, and SOUTH METROPOLITAN GAS CO. (B.P. 415,870, 19.6.33).

**Separation of materials. Recovering liquid from gravity separation.**—See I. Wetting etc. agents.—See III.  $H_2$  production. S from spent oxide.—See VII. Hydrogenation catalysts.—See X. Bituminous paint. Synthetic resin.—See XIII.

### III.—ORGANIC INTERMEDIATES.

**Preparation of acetaldehyde from acetylene (carbide).** H. THOMMEN (Chem.-Ztg., 1934, 58, 797–800).—A general description.

**Preparation of pure diethylamine by hydrochloric acid hydrolysis of *p*-nitrosodiethylaniline.** E. KATTSCHER (Anilinokras. Prom., 1934, 4, 340–342).—Price, Brazier, and Wood's method (J.S.C.I., 1916, 35, 147) gives 88–92% yields of 99%  $NHEt_2$ . R. T.

**Determination of benzene-mono- and -disulphonic acids.** A. I. KIPRIANOV and E. D. SITSCH (Zavod. Lab., 1934, 3, 623–625).—3 g. of material are

dissolved in 250 c.c. of  $H_2O$ ,  $H_2SO_4$  is determined in 25 c.c., and total acidity (I) in a second 25-c.c. portion by titration with 0.1N-KOH (phenolphthalein). The titrated solution is evaporated to dryness, and the residue of K salts is heated at 250° to const. wt. The % content of  $PhSO_3H = 135.1(S - 0.0157A)$ , and of  $C_6H_4(SO_3H)_2 = 101.8(0.0196A - S)$ , where  $S$  is the wt. of K salts less that of  $K_2SO_4$ , and  $A$  is (I) less that due to  $H_2SO_4$ . R. T.

**Preparation of aromatic amines from chloro-derivatives.** N. N. VOROSHOV, JUN. (Anilinokras. Prom., 1934, 4, 332–340).— $PhCl$  reacts with aq.  $NH_3$  in presence of  $Cu_2O$  as follows:  $PhCl + Cu(NH_3)_2$  (I)  $\rightarrow PhCl, Cu(NH_3)_2$  (II); (II) +  $NH_3 \rightarrow NH_2Ph + HCl$  + (I); (II) +  $OH^- \rightarrow PhOH + Cl^-$  + (I); (II) +  $NH_2Ph \rightarrow NHPH_2 + HCl$  + (I). Of these reactions, the slowest is that of formation of (II), whence it follows that the velocity of reaction in homogeneous systems is a function of the concn. of the catalyst and of  $PhCl$ , but not of  $NH_3$ . R. T.

**Determination of aniline in presence of nitro-anilines.** A. P. TERENTIEV and L. A. SCHTSCHETININA (Anilinokras. Prom., 1934, 4, 359–362).—The solution, containing *o*-, *m*-, and *p*- $NH_2 \cdot C_6H_4 \cdot NO_2$  and  $NH_2Ph$ , is diazotised at 0°, excess of 30%  $NaOH$  is added, and the resultant solution titrated with 0.05N- $\beta$ - $C_{10}H_7 \cdot OH$ , which reacts only with  $PhN_2Cl$ . R. T.

**Influence of conditions of nitration of formanilide on the yield and relative isomeric composition of nitroformanilide.** J. T. EIDUS, A. P. ALECHINA, and M. V. ARISTARCHOVA (Anilinokras. Prom., 1934, 4, 362–368).—The ratio of *p*- (I) to *o*- (II) -nitroformanilide rises from 2.5 when 90%  $H_2SO_4$  is used in the nitration mixture to 52 with 6% oleum; at the same time the total yield falls by  $> 50\%$ , partly owing to formation of  $NH_2 \cdot C_6H_3(NO_2)_2$ . The best yield (90%) is obtained when 98%  $H_2SO_4$  is used. Increasing the proportion but not the concn. of  $H_2SO_4$  does not affect the yield of (I), but greatly reduces that of (II) in the paste; the reverse is the case when the proportion of  $H_2SO_4$  is  $<$  the optimum. The total yield and its composition are little affected by increasing the proportion of  $HNO_3$  by up to 50%. The best yields of (I) are obtained by nitration at 0°. The proportion of (II) in the product rises from 8.7% at  $-10^\circ$  to 20% at  $15^\circ$ . R. T.

**Diethylaniline and benzylethylaniline.** N. G. LAPTEV (Anilinokras. Prom., 1934, 4, 342–349).—In the process of removal of  $NHPhEt$  (I) from  $NPhEt_2$  (II) as  $CH_2Ph \cdot NPhEt$  (III) by adding  $CH_2PhCl$  (IV) (U.S.P. 1,884,772; B., 1933, 720) 3–5% of (I) remains uncombined when the theoretical amount of (IV) is added; the remaining (I) is best eliminated by ethylation with  $EtBr$  at  $90^\circ$ . The presence of  $CHPhCl_2$  in (IV) greatly reduces the yields of (III). (IV) does not, in presence or absence of  $CH_2Ph \cdot OH$ , yield  $NH_4$  derivatives (V) with (II), and  $> 2\%$  of (V) with (III). The mixture of (I) and (II) should contain  $\geq 1$ –2% of  $NH_2Ph$ , as otherwise a considerable proportion of (I) remains, even when a 10% excess of (IV) is taken. (II) is separated from the benzylation mixture by adding sufficient aq.  $H_2SO_4$  to combine with (II) only, when (II) alone is present in the aq. layer, impure (III) remaining

undissolved. The (III) layer is extracted with aq.  $H_2SO_4$ , when unchanged (IV) and tarry products remain undissolved. The (III) thus obtained is freed from traces of (II) by steam-distillation. R. T.

**Vacuum distillation of  $\beta$ -naphthol.** M. J. ILLIUKÉ-VITSCH and L. E. TSCHERNETZKAJA (Anilinokras. Prom., 1934, 4, 350—356).—At  $160^\circ/70$  mm.  $\beta$ - $C_{10}H_7\cdot OH$  (I) can be distilled with a loss due to decomp. of  $> 1.5$ —3%; the % loss rises with rising temp. of distillation and with the initial content of impurities in the (I), Fe salts being the most harmful in this connexion. R. T.

**Rapid determination of 1:2:4-diazonaphthol-sulphonic acid and of certain of its derivatives.** G. I. OSTROSHINSKAJA (Anilinokras. Prom., 1934, 4, 368—371).—A modification of Gutmann's method (A., 1912, i, 397) is described. R. T.

**Filtration of gypsum in the production of amino-S-, -C-, and *p*ert-nitrosulphonic acids.** N. M. KAS-ATKIN and E. A. IVANOV (Anilinokras. Prom., 1934, 4, 371—375).—The rate of filtration of suspensions of gypsum arising in the production of the above acids is diminished when the solutions are kept before or after neutralisation with  $CaCO_3$ , when  $> 10\%$  excess of  $CaCO_3$  is present, and, in the case of S- and C-acids, when they are heated during or after neutralisation. R. T.

**Dissolved  $C_2H_2$ . Hydrogenation-cracking of tar.  $CH_4$  as motor fuel. Synthetic lubricating oils.**—See II. Determining S in org. compounds.—See V. Dielectric constants.—See XI. Determining Pr<sup>OH</sup> in beverages. Citric fermentation.—See XVIII. Citric acid from whortleberries.—See XIX. Determining aldehydes in oils.—See XX. Distilling phenols from  $H_2O$ .—See XXIII.

See also A., Oct., 1091, Prep. of keten. 1096, Synthesis of thio-*o*-toluidine. 1097, Prep. of *p*-indophenol and derivatives. 1106, Prep. of camphene and camphorquinone. 1109, Prep. of 2:5-dimethylpyrrole.

#### PATENTS.

**Manufacture of substances suitable as wetting, cleansing, dispersing, softening, and like agents.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 416,379, 6.3.33 and 5.3.34).—Aliphatic olefines or alkyl halides containing  $\leq C_8$  are condensed with an aromatic hydrocarbon (e.g.,  $C_6H_6$ , PhMe,  $C_{10}H_8$ ) in presence of an acid condensing agent ( $AlCl_3$ ,  $ZnCl_2$ ) or a surface-active catalyst and the products sulphonated either during or after condensation. Examples of starting materials are the chlorides or olefines from the higher *n*-aliphatic alcohols or from chlorination or cracking of mineral oils or natural waxes. The products are claimed to be stable to hard  $H_2O$ . H. A. P.

**Manufacture of condensation products from alcohols [methylisobutylcarbinol] and phenols.** BRIT. INDUSTRIAL SOLVENTS, LTD., H. LANGWELL, and C. B. MADDOCKS (B.P. 416,505, 17.3.33).— $CHMeBu^i\cdot OH$  (1 mol.) and PhOH or cresols (slightly  $> 1$  mol.) are heated at approx.  $130^\circ$  in presence of aq.  $ZnCl_2$  (from a previous condensation) incompletely dehydrated by heating at  $210^\circ/1$  atm. or  $130^\circ/vac$ . H. A. P.

**Production of organic products from 7:18-stearic glycol [ $\alpha,\lambda$ -dodecanediol].** F. GUENTHER and K. SAFTIEN, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,942,812, 9.1.34. Appl., 12.4.32. Ger., 29.4.31).—The glycol is dehydrated to  $\Delta^x$ - or  $\Delta^y$ -octadecenol by heating it or its org. esters (chloroacetate, acetate) with  $H_2SO_4$ ,  $H_3PO_4$ , org. sulphonic acids, or halogenoacetic acids ( $CH_2Cl\cdot CO_2H$ ) at  $100$ — $200^\circ$  ( $200^\circ$ ). The product is converted into H sulphate ( $ClSO_3H$  in  $Et_2O$  at  $15^\circ$  or  $C_2HCl_3$ ) or  $H_2$  phosphate ( $P_2O_5$  in  $Et_2O$ ), which are detergents, wetting, and emulsifying agents. H. A. P.

**Recovery of organic acids [from alkali salts].** J. C. WOODRUFF, G. BLOOMFIELD, and I. J. KRCHMA, Assrs. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,946,419, 6.2.34. Appl., 27.4.31).—The salt is treated with  $CO_2$  under pressure in an aq.-org. solvent in which it is sol. and  $NaHCO_3$  is insol. Suitable solvents are 80—97% aq.  $COMe_2$  and  $MeOAc$  (72%)— $MeOH$  (22%)— $H_2O$  6%. H. A. P.

**Production of methylamine from dimethyl ether.** L. C. SWALLEN, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,946,245, 6.2.34. Appl., 11.10.29).— $Me_2O$  (1 mol.) and  $NH_3$  (1—2 mols.) are passed over  $Al_2O_3$ -kieselguhr at  $300$ — $450^\circ$  ( $400^\circ$ ) at a space velocity of 100—2000 (150) vols. per hr. per vol. of catalyst. The product is claimed to contain 70% of  $NH_2Me$  mixed with  $NHMe_2$  and  $NMe_3$ . H. A. P.

**[Manufacture of] alkylolamine [hydroxyalkylamine] derivatives of fatty acids.** J. G. KERN and C. J. SALA, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,946,079—80, 6.2.34. Appl., [A, B] 21.3.30).—The prep. and use (as dispersing and emulsifying agents) of soaps from hydroxyalkylamines [ $NH(C_2H_4\cdot OH)_2$ ,  $N(C_2H_4\cdot OH)_3$ ,  $NH_2\cdot CH_2\cdot CH(OH)\cdot CH_2\cdot OH$ ] and (A) stearic, ricinoleic, and (A, B) oleic acids are claimed. H. A. P.

**Manufacture of cyanohydrins.** TRIPLEX SAFETY GLASS Co., LTD., L. V. D. SCORAH, and J. WILSON (B.P. 416,007, 1.3.33).—HCN (1 mol.) is added slowly to  $COMe_2$  (1 mol.) maintained at its b.p. and containing a small amount (0.5%) of basic catalyst ( $C_5H_5N$ ,  $C_5H_{11}N$ , quinoline), boiling is continued until equilibrium is reached (0.5—1 hr.), and the liquid is cooled, acidified (50% excess  $H_2SO_4$ ), and distilled in vac. Alternatively, the product is dehydrated *in situ* to  $CH_2\cdot CMe\cdot CN$  by gradual addition of  $C_5H_5N$  and  $P_2O_5$ . H. A. P.

**Use of catalyst for nuclear chlorination of benzene compounds.** W. C. STOESSERT and F. B. SMITH, Assrs. to DOW CHEM. Co. (U.S.P. 1,946,040, 6.2.34. Appl., 1.10.31).—In the chlorination of PhCl, Ph<sub>2</sub>, or mono-alkylbenzenes (PhMe) the formation of the *p*-isomeride is favoured by use of a mixed catalyst comprising S and  $SbCl_3$  (and superficially chlorinated Pb). H. A. P.

**[Preparation of] ethylene glycol phthalate.** C. L. GABRIEL and L. C. SWALLEN, Assrs. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,946,202, 6.2.34. Appl., 24.1.28).—Liquid esters of approx. mol. wt. 500 are obtained by heating *o*- $C_6H_4(CO_2)_2O$  with  $C_2H_4(OH)_2$  and a catalyst ( $H_2SO_4$ ) in  $C_6H_6$  as entraining liquid for the  $H_2O$  formed. They are converted into resins of higher mol. wt. by further heating (to  $150^\circ/vac$ ). H. A. P.

**Manufacture of amino-aromatic ketones.** E. C. BRITTON and F. BRYNER, Assrs. to DOW CHEM. CO. (U.S.P. 1,946,058, 6.2.34. Appl., 27.7.32).—The corresponding nuclear-halogenated ketone is heated with aq.  $\text{NH}_3$  ( $\leq 2$  equivs.) and a Cu compound ( $\text{Cu}_2\text{O}$ ) at  $170\text{--}300^\circ$  ( $190\text{--}250^\circ$ ). Examples are the prep. of  $p\text{-NH}_2\cdot\text{C}_6\text{H}_4\cdot\text{COMe}$ ,  $4\text{-NH}_2\cdot\text{C}_6\text{H}_4\cdot\text{COPh}$ , and  $4:4'\text{-CO}(\text{C}_6\text{H}_4\cdot\text{NH}_2)_2$  from the corresponding Cl-compounds.

H. A. P.

**Manufacture of 1:4-diaminoanthraquinones.** SOC. CHEM. IND. IN BASLE (B.P. 416,245, 17.1.34. Switz., 18.1.33).—A 1:4-quinonoid derivative of anthraquinone is heated with 1:4-diamino-2:3-dihydroanthraquinone (I) or its substitution products. Thus, e.g., (I) with anthraquinone-1:4-quinoneimine in 90%  $\text{H}_2\text{SO}_4$  at  $90^\circ$  gives 1:4-diamino- (II) and 1-amino-4-hydroxyanthraquinone and with anthraquinone-1:4-quinone-di-imine gives (II) alone.

H. A. P.

**Manufacture of heterocyclic sulphonic acids.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 416,291, 23.4.34).—2-Thiol-benz- or -naphth-oxazoles or -thiazoles are oxidised in alkaline solution, or the 2-halogeno-compounds are heated with an alkali sulphite. Examples include the prep. of benzthiazole-2-sulphonic acid, its 4-OEt, 4-NHBz, 4- $\text{NO}_2$ , 5- $\text{SO}_3\text{H}$ , 3:4-benzo-, and 4-Cl-6-Me derivatives, benzoxazole- and  $\beta$ -naphth-oxazole-2-sulphonic acids (as Na salts).

H. A. P.

$\text{C}_{10}\text{H}_8$  from gas. Tar acids. Removing phenols from liquors. MeOH from benzol purification.—See II. Intermediates for azo dyes.—See IV.  $\text{CNCl}$ .—See VII. Hydrogenation catalysts. Soldering fluxes.—See X.  $\text{EtCO}_2\text{H}$  fermentation.—See XVIII.

#### IV.—DYESTUFFS.

[Production of] leuco-crystal-violet by pressure synthesis. T. R. LISTON and W. M. DEHN (Ind. Eng. Chem., 1934, 26, 1073—1074).—The effects of catalysts (I), temp. (II), and solvents (III) on the yields of  $\text{CH}(\text{C}_6\text{H}_4\cdot\text{NMe}_2\cdot p)_3$  obtained by direct interaction of  $\text{NPhMe}_2$  and CO (water-gas containing 15.5% CO) under pressures of 246—351 kg./sq. cm. are tabulated. Best yields (63%) are obtained when (I) is anhyd.  $\text{AlCl}_3$ ,  $\text{FeCl}_3$ , or  $\text{AlCl}_3\text{-Cu}_2\text{Cl}_2$ , (II) is  $17^\circ$  for mixing and  $25^\circ$  for reaction, and (III) is  $\text{PhCl}$ .  $\text{PhNO}_2$  as (III) causes oxidation, and HCl and  $\text{H}_2\text{O}$  inhibit the reaction. No comparable advantage accrues from the use of pure CO.

J. W. B.

Colouring matter in pine kraft pulps.—See V.

See also A., Oct., 1097, Colour of monosubstituted  $\beta$ -naphtholazo dyes. 1099,  $\text{CPh}_4$  dyes. 1108, Synthesis of ceroxonones. Pechmann's dye. 1114, Phthalocyanines. 1115, Oxacyanines. Prep. of thiazole-azo dyes.

#### PATENTS.

**Manufacture of dye pastes.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 415,742, 30.12.32).—Acetate silk dyes of aminoanthraquinone, azo, and indophenol classes (containing  $\leq 1$  basic group) are ground with polyhydric alcohols [glycerol, polyglycerols,  $\text{C}_2\text{H}_4(\text{OH})_2$ , polyglycols], thioalkylene glycols, or hydroxyalkylamines until no increase of colour val. on printing occurs.

H. A. P.

**Production of azo dyes [for leather and acetate silk].** I. G. FARBENIND. A.-G. (B.P. 414,446, 25.2.33. Ger., 6.5.32. Addn. to B.P. 384,800; B., 1933, 185).—An alkyl or alkoxy derivative of 4-nitro-*m*-phenylene-diamine (I) is monodiazotised and coupled with a coupling component. Examples are: 6-Me derivative of (I)  $\rightarrow$   $\text{NPhEt}_2$  (orange on acetate silk); 6-OMe-derivative of (I)  $\rightarrow$  H-acid (brown on leather). C. H.

**Manufacture of azo dyes.** SOC. CHEM. IND. IN BASLE (B.P. 414,483, 15.5.33. Switz., 15.5.32).—Aryl esters of aniline-*o*-sulphonic acids, carrying a  $\text{NO}_2$ - or acylamino-group in position 4, are diazotised and coupled with a coupling component; the  $\text{NO}_2$ - or acylamino-group may subsequently be converted into  $\text{NH}_2$ , and a new acyl group may be introduced. Examples are:  $\text{NO}_2\text{-C}_6\text{H}_3(\text{NH}_2)\cdot\text{SO}_3\text{Ph} \rightarrow \gamma$ -acid (violet-blue on wool);  $p\text{-C}_6\text{H}_4\text{Cl}$  ester  $\rightarrow \gamma$ -acid (blue-red). C. H.

**Manufacture of azo dyes [containing chromium] and intermediates thereof.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 414,381, 1.2.33).—A 5-aminosalicylic acid is treated with a  $\text{Cr}^{+++}$  compound, e.g.,  $\text{CrCl}_3$ , in a solvent ( $\text{H}_2\text{O}$  and/or org. solvent);  $\leq 1$  mol. of a non-diazotisable org. base [ $\text{C}_5\text{H}_5\text{N}$ ,  $\text{C}_2\text{H}_4(\text{NH}_2)_2$ ,  $\text{NPhEt}$ ,  $\text{NPhMe}_2$ ] may be present and enters into the complex. The products may be tetrazotised and coupled with 2 mols. of a coupling component; the org. base and/or the Cr may be removed from the resulting dye complexes. Examples are:  $\text{Cr-C}_5\text{H}_5\text{N}$  complexes of 5-aminosalicylic acid (I) and its 3-Me derivative; Cr-triethanolamine complex of 3-sulphosalicylic acid (II); (I)  $\rightarrow$  2 mols. of salicylic acid (III) (yellow on wool; redder on chroming); Cr-complex of 5-aminosalicylic acid  $\rightarrow$  2 mols. of R-acid (red); (II)  $\rightarrow$  2 mols. of (III), treated with acid to remove  $\text{C}_5\text{H}_5\text{N}$  and Cr (yellow). C. H.

[Lactamisable] azo dyes and application thereof. IMPERIAL CHEM. INDUSTRIES, LTD., S. COFFEY, M. LAPWORTH, and W. A. SEFTON (B.P. 415,753, 30.11.32. Cf. B.P. 377,739; B., 1932, 1021).—Compounds of the type  $o\text{-NH}_2\cdot\text{C}_6\text{H}_4\cdot\text{X}\cdot\text{CR}'\text{R}''\cdot\text{CO}_2\text{H}$  ( $\text{X}=\text{O}$  or R and R' and R'' are H or alkyl) are used as coupling components (I), or, alternatively, corresponding amines in which the *o*-(lactamisable)- $\text{NH}_2$  is latent, are used as diazo components, the final dyes being free from additional solubilising groups and convertible by lactamisation on the fibre into  $\text{H}_2\text{O}$ -insol. pigments. The use of  $o\text{-NH}_2\cdot\text{C}_6\text{H}_4\cdot\text{S}\cdot\text{CH}_2\cdot\text{CO}_2\text{H}$  and 4-alkyl- or 4-alkoxy-2-aminophenoxyacetic acids is disclaimed. Thus, e.g., 2-nitro-4-aminophenylthioglycollic acid is diazotised and coupled with 2:3-hydroxynaphthoic arylamides, pyrazolones,  $m\text{-C}_6\text{H}_4(\text{NH}_2)_2$ , etc., and the products are reduced with  $\text{Na}_2\text{S}$ . Other examples of (I) are 5:2:1- $\text{NH}_2\cdot\text{C}_6\text{H}_3(\text{NO}_2)\cdot\text{S}\cdot\text{CH}_2\cdot\text{CO}_2\text{H}$  and its 4-Cl, 4-OMe, and 4-Me derivatives, 2-nitro-4-aminophenyl-thiolactic, -glycollic, and -lactic acids, and 4-chloro-2-nitro-5-aminophenylthioisobutyric acid. H. A. P.

**Manufacture of disazo dyes.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 416,053, 6.3.33).—Acid wool and silk dyes of excellent fastness to washing and fulling are prepared by coupling tetrazotised 4:4'- $\text{CO}(\text{C}_6\text{H}_4\cdot\text{NH}_2)_2$  (I) and derivatives with hydroxy-naphthoyl- or -naphthalenesulphon-arylamides or Bz or

ArSO<sub>2</sub> derivatives of aminonaphthols or their sulphonic acids,  $\leq 1$  SO<sub>3</sub>H being present in the final dye. Examples are: (I)  $\rightarrow$  1:8:3:6-NHBz·C<sub>10</sub>H<sub>4</sub>(OH)(SO<sub>3</sub>H)<sub>2</sub> (II) (blue-red); (I)  $\rightarrow$  2:5:7-NHBz·C<sub>10</sub>H<sub>5</sub>(OH)·SO<sub>3</sub>H or  $\rightarrow$  2:3-OH·C<sub>10</sub>H<sub>6</sub>·CO·NHPh (yellow-red); 1:8:4:6-NHBz·C<sub>10</sub>H<sub>4</sub>(OH)(SO<sub>3</sub>H)<sub>2</sub> (III)  $\leftarrow$  (I)  $\rightarrow$   $\beta$ -C<sub>10</sub>H<sub>7</sub>·OH (blue-red); 1-2':5'-dichlorobenzamido-8-naphthol-4:6-disulphonic acid (V)  $\leftarrow$  (I)  $\rightarrow$  2:6-OH·C<sub>10</sub>H<sub>6</sub>·SO<sub>3</sub>H, 1:8:3:6-*p*-C<sub>6</sub>H<sub>4</sub>Me·SO<sub>2</sub>·NH·C<sub>10</sub>H<sub>4</sub>(OH)(SO<sub>3</sub>H)<sub>2</sub>  $\leftarrow$  3:3'-Cl<sub>2</sub>-derivative of (I)  $\rightarrow$  2:8:6-NH<sub>2</sub>·C<sub>6</sub>H<sub>5</sub>(OH)·SO<sub>3</sub>H, (II)  $\leftarrow$  (I)  $\rightarrow$  2:6-OH·C<sub>10</sub>H<sub>6</sub>·SO<sub>2</sub>·NHPh (all red); 3:3'-(OMe)<sub>2</sub>-derivative of (I) (IV)  $\rightarrow$  (III) (violet), and 2:8:6-NHBz·C<sub>10</sub>H<sub>5</sub>(OH)·SO<sub>3</sub>H  $\leftarrow$  (IV)  $\rightarrow$  (V) (blue-violet).  
H. A. P.

**Manufacture of vat dyes of anthracene series.** W. W. GROVES. FROM I. G. FARBENIND. A.-G. (B.P. 416,385, 10.3.33).—Anthracene-1:4:9:10- or -1:5:9:10-tetracarboxylic acids or their anhydrides are condensed with an *o*-diamine or its salts or with an *o*-nitroamine followed by reduction. Examples are the condensation of the crude acid or anhydride obtained by oxidising (KClO<sub>3</sub> and HCl) the condensation product of aceanthrene with CH<sub>2</sub>(CN)<sub>2</sub> with *o*-C<sub>6</sub>H<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub> and 4:1:2-OEt·C<sub>6</sub>H<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub> (both dark blue).  
H. A. P.

**Manufacture of anthraquinone [vat] dyes.** Soc. CHEM. IND. IN BASLE (B.P. 414,529, 2.9.33. Switz., 15.9.32).—An 8-halogeno-1-acylaminoanthraquinone is halogenated in position 4, condensed with 2 mols. of a 1-aminoanthraquinone, and carbazolated with ClSO<sub>3</sub>H, AlCl<sub>3</sub>, etc. Examples are: 4:8-dichloro-1-benzamidoanthraquinone, m.p. 213—214°, with 1-aminoanthraquinone, carbazolated (brown); 4:8-dichloro-1-urethano- (m.p. 205—206°), 8-chloro-4-bromo-1-benzamido- (m.p. 215°), and 4:8-dichloro-1-acetamido- (m.p. 218—219°)-anthraquinones.  
C. H.

**Manufacture and application [as vat dyes] of aroylaminoanthraquinones.** IMPERIAL CHEM. INDUSTRIES, LTD., and N. H. HADDOCK (B.P. 416,425, 11.3.33).—Aminoanthraquinones are condensed with the chlorides of bis(carboxy)phenylimides of naphthalene- (I) or perylene-peritetracarboxylic acid (II). Thus, *e.g.*, (I) and (II) are heated with *m*-NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·CO<sub>2</sub>H at 180—200°, and the products converted into their chlorides by SOCl<sub>2</sub> in PhNO<sub>2</sub> at 105—110°, and these condensed with 1-amino-4-methoxy- (scarlet) or 1-amino-4-benzamido- (bluish-red) and 1-aminoanthraquinone (yellow), respectively.  
H. A. P.

**[Manufacture of diamino]anthraquinone derivatives.** IMPERIAL CHEM. INDUSTRIES, LTD., and F. LODGE (B.P. 416,020, 4.3.33).—A mono- or dialkylated (arylated etc.) 1-aminoanthraquinone-2-sulphonic acid is halogenated, and the product heated with a SO<sub>3</sub>H-free primary or *sec.*-amine; *e.g.*, K 4-bromo-1-methylaminoanthraquinone-2-sulphonate is heated with *p*-C<sub>6</sub>H<sub>4</sub>Me·NH<sub>2</sub>, NaHCO<sub>3</sub>, and Cu(OAc)<sub>2</sub> at 80° to give a blue wool dye.  
H. A. P.

**Manufacture of 1:4-diamino-2-aryloxanthraquinone-3-sulphonic acids.** I. G. FARBENIND. A.-G. (B.P. 416,433, 14.3.33. Ger., 15.3.32. Addn. to B.P. 363,027; B., 1932, 333).—A 2:3-dihalogeno-1:4-diaminoanthraquinone is heated with a phenol (excess) and an acid-binding agent (K<sub>2</sub>CO<sub>3</sub>) at 150—160° and

the monoaryl ether produced (without isolation) heated with aq. Na<sub>2</sub>SO<sub>3</sub> under pressure (at 140—145°). The prep. of 1:4-diamino-2-phenoxyanthraquinone-3-sulphonic acid, a blue acid wool dye, is described.

H. A. P.

**Manufacture of [benzoquinone] dyes.** I. G. FARBENIND. A.-G. (B.P. 415,749, 27.1.33. Ger., 27.1.32. Addn. to B.P. 313,094; B., 1931, 337).—The process of the prior patent is applied to dihalogenodi(acylaryl-aminoarylamino)benzoquinones or corresponding carbazoles obtained, *e.g.*, by condensation of chloranil (I) with an aminoacyldiarylamide or a 9-acylaminoanthraquinone, or, alternatively, acylation is carried out during the condensation. Examples of starting materials are the 9':9'-bis-Bz, -Ac, -*p*-C<sub>6</sub>H<sub>4</sub>Me·SO<sub>2</sub>, -NHPh·CO, and -CO-derivatives of 3:6-dichloro-(or -dibromo)-2:5-bis-3':3'-carbazylylaminoanthraquinone and the condensation product of (I) with 4-NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·NBzPh (2 mols.). Sulphonation of the products gives blue to violet wool dyes.  
H. A. P.

**Solid vat dye preparations.** I. G. FARBENIND. A.-G. (B.P. 414,426, 9.2.33. Ger., 10.2.32).—A vat dye (*e.g.*, tetrabromoindigo, Algol-yellow, dibromodimethoxydibenzanthrone) is mixed with Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> and Na<sub>3</sub>PO<sub>4</sub>, with or without a wetting agent.  
C. H.

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

**Testing of waterproof fabrics.** F. D. TOYNE (Ind., Chem. 1934, 10, 380).—H<sub>2</sub>O (60 0.1-g. drops/min.) falls through 6 ft. on to a 4-in. square of the cloth, which is stretched by four 20-g. weights over the mouth of a funnel; this delivers into a burette, and the time required to pass 5 ml. is recorded as the "proof-val."  
J. G.

**Recent advances in the manufacture of cellulose from wheat straw with chlorine gas.** (Pomilio patents.) P. PIZZINI (Papier-Fabr., 1934, 32, 417—419).—The operation of a plant recently installed in Santiago is described.  
D. A. C.

**Production of cellulose from larch and cedar.** L. P. ZHEREBOV, L. V. GORDON, V. N. KOMAROVSKI, and G. A. KAN (Trans. Centr. Inst. Sci. Res. Forest Chem. U.S.S.R., 1933, 2, 8—38).—Extracted larch cooked for 1 hr. at 160—170° with 14% liquor (NaOH + Na<sub>2</sub>S) afford 53—54% of kraft cellulose; conditions for obtaining bleachable stock and soft pulp are recorded. Unextracted larch produced lower yields. The sulphate pulp is not easily bleached. Larch can be used for the industrial production of paper. Siberian cedar produced 55% of sulphate kraft pulp and 41% of bleached with a higher consumption of Cl<sub>2</sub> than for larch.  
KOMAROVSKI

**Utilisation of rot-damaged larch in the production of cellulose.** L. P. ZHEREBOV and V. N. KOMAROVSKI (Trans. Centr. Inst. Sci. Res. Forest Chem. U.S.S.R., 1933, 2, 8—38).—Injury caused by a rot-retarding cellulose, retards the mechanical properties of the pulp. The effect on the yield of cellulose, and its effect on the yield of cellulose, and affects the bleaching, and affects the softness of the pulp.  
KOMAROVSKI

**Cellulose cooking.** A. KREISSLER (Zellulose, 1934, No. 9, 17—23).—Innovations in the process is given, including a short

superficial description of the Lurgi circulation system and method of liberating residual free  $\text{SO}_2$  from spent liquors.

D. A. C.

**Maturation of cellulose solutions.** G. TOCCO and E. CERBARO (Boll. Staz. Sperim. Ind. Carta Fibre Tess., 1934, 29, 456—459; cf. B., 1931, 876, 1087).—The maturation (*M*) of cellulose esters dissolved in metallic thiocyanate solutions is connected with hydrolysis of the esters, this being rapid with the formate and much slower with the acetate, solutions of which keep for months without apparent change, even of  $\eta$ . The  $\eta$  falls slightly to a min. and later rises appreciably as coagulation approaches. *M* is accelerated by the acid liberated, and by inorg. salts,  $\text{PhOH}$ , or  $\text{CH}_2\text{O}$ , and is retarded by  $\text{NH}_3$  and other alkalis, or by alkali formates or acetates. Solutions in  $\text{Ca}(\text{CNS})_2$  coagulate more rapidly than those in  $\text{NaCNS}$  or  $\text{KCNS}$ . The thiocyanates hydrolyse also other esters, such as alkyl formates and acetates. Ethyl- and benzyl-celluloses do not dissolve in thiocyanate solutions. The results support Berl's theory (B., 1926, 943—4) of the *M* of cellulose solutions.

T. H. P.

**Vistra.** W. ELLER (Chem.-Ztg., 1934, 58, 809—812).—Vistra (viscose staple fibre) is spun on ordinary cotton machinery; the uses of the fabrics are outlined. It is especially suitable for spinning in admixture with cotton or wool, and large economies could be effected by replacing 30% of the latter.

A. G.

**Rayon and insects.** E. DEBENEDETTI (Boll. Staz. Sperim. Ind. Carta Fibre Tess., 1934, 29, 460—467).—Rayon may be damaged by attack of: (1) insects or their larvæ which feed on proteins and normally attack wool, silk, etc., such attack being exceptional, or (2) insects which feed on cellulosic materials, including parasites of wood, paper, and vegetable residues, in general, and actually consume the rayon. *Ptilinus fuscus* (fam. Anobiidae) and *Criptomagus* were found on damaged rayon. Means of defence are considered.

T. H. P.

**Chromium-nickel alloys for sulphite [digester] systems.** C. C. SNYDER and V. W. WHITMER (Paper Trade J., 1934, 99; T.A.P.P.I. Sect., 150—152).—The Mo type of Cr-Ni steel alloys called 18-8-SMO (Cr 18, Ni 10, Mo 3, C 0.06%) has been found in a no. of Pacific Coast pulp mills to be better than 18% Cr-8% Ni without Mo; 20% Cr-10% Ni and 25% Cr-10% Ni alloys are less satisfactory. Weld decay (carbide pptn.) is prevented by the 3% Mo content, and this alloy contains also traces of C, Mn, P, and S.

H. A. H.

**Factors which affect the yield of sulphite pulp.** G. J. C. POTTER and F. H. YORSTON (Pulp and Paper Canada, 1934, 35, 526—529).—A survey of recent literature, indicating chiefly the many phases of the subject not yet fully investigated.

H. A. H.

**Theory of absorption applied to the sulphite [liquor] tower [for wood-pulp manufacture].** W. L. BEUSCHLEIN and F. H. CONRAD (Paper Trade J., 1934, 99; T.A.P.P.I. Sect., 119—122).—Factors affecting the absorption of  $\text{SO}_2$  by  $\text{CaO}$  in the sulphite-liquor tower are investigated theoretically. A method is given for indicating the effect of tower-operating charac-

teristics. Some pressure-composition curves are presented for the system  $\text{CaO-SO}_2\text{-H}_2\text{O}$  at 15° and 25°.

H. A. H.

**Determination of sulphur in sulphite waste liquor and organic compounds. Potassium permanganate method.** R. N. POLLOCK and A. M. PARTAUSKY (Ind. Eng. Chem. [Anal.], 1934, 6, 330—331).—The substance is evaporated with  $\text{NaOH}$  and  $\text{KMnO}_4$  and then heated at 500°.  $\text{SO}_4^{2-}$  is determined as usual in the melt.

R. S. C.

**Pulping of Douglas fir by ammonia-base liquor.** H. K. BENSON, R. P. ERWIN, J. R. HENDRICKSON, and J. A. TERSHIN (Paper Trade J., 1934, 99; T.A.P.P.I. Sect., 131—133).—A series of small-scale experimental cooks, using  $\text{NH}_4\text{HSO}_3$  in place of the usual  $\text{Ca}(\text{HSO}_3)_2$ , were carried out, whereby old, young, and medium growth in Douglas fir (the last after pre-resin extraction with aq.  $\text{NH}_3$ ) were compared with commercial spruce and hemlock (all in chip form). Numerous physical and chemical tests on the resulting pulps showed that young fir is equal in quality to the more commonly used woods, but old fir is distinctly inferior. The use of  $\text{NH}_3$  in place of  $\text{Ca}$  as base results in higher  $\alpha$ -cellulose vals. for all the pulps. The resin content of fir need only be reduced to the extent to which it occurs in other coniferous woods; complete removal is unnecessary. The very low lignin content of young fir wood makes the pulp valuable when a high degree of purity is required.

H. A. H.

**Developments in processing of resinous wood waste.** R. C. PALMER (Chem. Met. Eng., 1934, 41, 456—460; cf. B., 1930, 655).—A brief review is given of some of the recent chemical and engineering developments in the processing of the raw material, together with references to various patents relating thereto.

D. K. M.

**Production of straw pulp at low pressure.** M. P. RESH, A. P. PETROV, and V. A. PINCHUK (Bumazhn. Prom., 1933, 12, No. 11, 26—29).—Rye and wheat straw cooked by the sulphate and sulphate-sulphite process at 4 atm. produced good-grade writing paper.

CH. ABS.

**Accelerated cooking of straw pulp.** M. P. RESH and A. P. PETROV (Bumazhn. Prom., 1933, 12, No. 12, 23—30).—An easily bleachable pulp was produced by treating rye and wheat straw, or a mixture, with 1%  $\text{NaOH}$  at 90—95° for 15—30 min., and then cooking for 1 hr. at 5—6 atm.

CH. ABS.

**Colouring matter in pine kraft pulps.** W. F. HOLZER (Paper Trade J., 1934, 99; T.A.P.P.I. Sect., 135—147).—Following G. M. Kuettel (Diss., Univ. Wisconsin, 1933), the idea that S dyes are responsible for the colour of kraft pulp is investigated, and further evidence adduced for the view that S derivatives of phlobotannin are responsible. Some qual. reactions are given. Other variables being const., the S content depends on the sulphidity of the cooking liquor, and the colour of both unbleached and bleached pulp varies directly with the S content. Similar compounds have been extracted from black liquor. An attempt to investigate the oxidation-reduction of the colouring matter by Clark's electrometric titration method was unsuccessful,

and by analogy it is suggested that the substance is acidic.

H. A. H.

**Optical studies of [chemical] wood pulps: influence of beating, loading materials, cooking, and calendering.** H. RESS (Papier-Fabr., 1934, 32, 361—367, 377—384, 385—392, 401—407, 409—414).—A photometric method of obtaining the brightness ( $B$ ) (reflective power) and transparency ( $T$ ) of pulps, by which the effect of beating and loading on a large variety of bleached and unbleached chemical wood pulps, bleached rag, esparto, straw, Manila pulps, etc., is investigated. Measurements were made over (1) an opaque layer of the same sheets as the sample, (2) an abs. black surface (velvet), (3) a standard white. The resultant curves are practically parallel, although (1) gave the highest and (3) the lowest vals.  $B$  of the bleached and unbleached wood pulps decreases with beating to a similar degree, whereas that of rag and Manila pulps remains practically const.  $T$  similarly increases. Increase in the loading content raises  $B$  according to the "brightening power" of the loading, of which blanc fixe (I) has the greatest.  $T$  similarly decreases according to the covering power of the loading. Beating produces a deeper shade of fluorescence when viewed under a quartz lamp, which is ascribed to the effect of the visible ultra-violet rays present. The fluorescence colours of the loading have no relation to the colours produced when present in the paper, although increase in loading causes an increase in intensity, (I) having the greatest power. The relation between the change in optical properties and physical changes in fibre structure is discussed on the basis of the most recent theories of hydration. D. A. C.

**Determination of fixed resin in [chemical] pulp.** V. HOTTENROTH (Papier-Fabr., 1934, 32, 367—368).—Graap's method (B., 1934, 753) is criticised on the grounds that  $(\text{HCO}_2)_2\text{Ca}$  is not completely insol. in  $\text{Et}_2\text{O}$ , and, further, that volatilisation of the  $\text{H}_2\text{C}_2\text{O}_4$  at  $130^\circ$  for 12 hr. would also remove some of the more volatile constituents of the resin extract. D. A. C.

**Removal of samples of oil from oil-impregnated paper.** J. D. PIPER (Ind. Eng. Chem. [Anal.], 1934, 6, 380—381).—The centrifugal method described is preferred to extraction methods for determining the nature of the oil. E. S. H.

**Increasing the usefulness of less expensive paper fibres with zinc sulphide pigments.** F. A. STEELE (Paper Trade J., 1934, 99; T.A.P.P.I. Sect., 149—150).—The optical advantages of  $\text{ZnS}$  pigments are not confined to fine papers, but can be used economically in the manufacture of unbleached papers, especially those intended for waxing, and boxboard liners.

H. A. H.

**Preservation of newspaper records.** B. W. SCRIBNER (U.S. Dept. Commerce, Nat. Bur. Stand. Misc. Publ. No. 145, 1934, 10 pp.).—A large no. of newspapers published in the United States between 1830 and 1900 were examined and attempted methods for their preservation are described. For retarding decay the use of Japanese tissue or transparent cellulose acetate covering appears to be effective. The properties are given for papers considered satisfactory for permanent newspaper editions, although for large-scale reproduction the

use is suggested of photographic glass plates containing the miniature print which can be reproduced on film slides for projection.

D. A. C.

**Differentiation between cellulose-destroying fungi parasitic on paper.** A. SARTORY, R. SARTORY, J. MEYER, and H. BÄUMLI (Compt. rend., 1934, 199, 439—442).—*Cladosporium herbarum* (I) was most active in decomposing cellulose when N was supplied as  $\text{NO}_3^-$ , *Aspergillus fumigatus* (II) when  $\text{NO}_3^-$  or gelatin, and *Fusarium cæruleum* (III) when peptone or gelatin was used. The addition of glucose to gelatin used as source of N diminished activity of (I) and (II) but increased that of (III).

L. D. G.

**Absorption of dyes by cellulose etc.**—See VI.  
**Testing [paper] for electrical industries.**—See XI.

See also A., Oct., 1064, Viscosity of cellulose ester solutions. 1099, Separating sterols from wool wax.

#### PATENTS.

**Artificial fabrics.** G. L. SCHWARTZ, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,945,537—8, 6.2.34. Appl., [A, B] 31.5.30. Renewed 19.1.33).—A web made from pulp, substantially similar to that obtained by dispersing paper in cold 8—35%  $\text{NaOH}$  solution for  $> 30$  min. and washing, is treated (A) with a solution of starch and casein or other colloidal matter, or (B) with rubber latex.

B. M. V.

**Production of film base from seaweed.** T. GOHDA (B.P. 416,826, 22.1.34).—Matured brown alga is dissolved in aq. alkali ( $\text{Na}_2\text{CO}_3$  and aq.  $\text{NH}_3$ ) and the filtered solution neutralised by addition of dil. mineral acid. After separation, washing, and drying, the product is dissolved in aq.  $\text{NH}_3$ , in the form of a jelly, dried in thin sheets, and treated with  $\text{Ac}_2\text{O}$ , glacial  $\text{AcOH}$ ,  $\text{ZnCl}_2$ , and furfuraldehyde until sol. in  $\text{COMe}_2$ . The acetylated product is washed with  $\text{H}_2\text{O}$ , dried, dissolved in a mixture of  $\text{COMe}_2$ ,  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ , and  $\text{C}_6\text{H}_6$ , and allowed to mature for a few days for production of films by known processes.

F. R. E.

**Manufacture of cellulose ethers.** J. CRAIK, L. RUBENSTEIN, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 416,590, 18.3.33).—Semi-moist soda-cellulose, after ageing at room temp. if desired, is treated at  $< 10^\circ$  ( $0-5^\circ$ ) with  $< 1$  ( $\frac{1}{4}$ ) mol. proportion of  $\text{Me}_2\text{SO}_4$  per  $\text{C}_6\text{H}_{10}\text{O}_5$  unit. The product, which is insol. in  $\text{H}_2\text{O}$  and incompletely sol. in aq. alkali of any concn. at room temp., may be brought into solution, before or after separation (by acidification or diluting with  $\text{H}_2\text{O}$  and washing) from the reaction mass, by treatment with aq. alkali, cooling below  $0^\circ$  until crystals of ice appear, and thawing the magma to a solution which is permanent at room temp.

F. R. E.

**Reduction of the viscosity of cellulose nitrate.** R. K. ESKEW, Assr. to DUPONT VISCOLOID Co. (U.S.P. 1,946,067, 6.2.34. Appl., 25.3.32).—Nitrocellulose in the undissolved state is treated with a solution of  $\text{C}_2\text{H}_5\text{N}$  or its homologues until the desired reduction in  $\eta$  is obtained, and then, preferably in presence of a small amount of acid, is washed with  $\text{H}_2\text{O}$ , treated with an aldehyde ( $\text{CH}_2\text{O}$ ,  $\text{MeCHO}$ ) to remove  $\text{C}_2\text{H}_5\text{N}$  etc., and again washed.

F. R. E.

**Manufacture of paper pulp.** A. G. MACINTYRE, Assr. to A. E. STALEY MANUFG. CO. (U.S.P. 1,945,862, 6.2.34. Appl., 7.8.30).—Wood is cooked either by acid or alkaline processes, with addition of a dispersing colloid (converted starch) other than that contained in the wood. F. R. E.

**Paper manufacture.** J. TRAQUAIR and F. G. RAWLING, Assr. to MEAD CORP. (U.S.P. 1,942,622, 9.1.34. Appl., 27.4.28).—Pulp is continuously bleached with  $\text{Cl}_2$  gas by feeding at a consistency of 15% into a vertical tower (*T*), the gas being introduced near its lower end. The pulp and gas travel together down *T* until, just prior to reaching a  $\text{H}_2\text{O}$ -sealed compartment at the bottom of *T*, the major portion of the gas is led off, the remainder being removed from the pulp by means of air jets. The pulp is discharged by slushing with  $\text{H}_2\text{O}$  into a storage tank (*S*), the rate of discharge being controlled by the level in *S*. The pressure in *T* controls the flow of gas in it. D. A. C.

**Recuperating rosin size already applied to papers.** S. YANO (U.S.P. 1,949,000, 27.2.34. Appl., 16.9.32).—After drying or other treatment detrimental to the rosin the paper is steamed at 55–100°. B. M. V.

**Impregnation [fireproofing] of porous material with solid substances.** A. WINOGRADOW (U.S.P. 1,945,714, 6.2.34. Appl., 22.7.30).—Fibre board (*I*) is immersed in a solution (*II*) of  $(\text{NH}_4)_2\text{SO}_4$ ,  $\text{NH}_4\text{H}_2\text{PO}_4$ ,  $(\text{NH}_4)_2\text{HPO}_4$ , or  $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$  which is subjected to vac. or pressure. After removal from (*II*), (*I*) is squeezed to reduce its vol. and remove sufficient solution so as to leave substantial air pockets, and is finally dried. F. R. E.

**Stapling of continuous filaments.** I. G. FARBER and A.-G. (B.P. 416,636, 22.2.34. Ger., 24.2.33).

**[Stretching] treatment of yarns [in warp form].** BRIT. CELANESE, LTD., R. W. MONCRIEFF, and G. W. HARRISON (B.P. 416,087, 10.3.33).

**Decorticating, hackling, combing, splitting, etc. of sisal and like fibrous leaves, stalks, etc.** W. J. HOLLIER (B.P. 416,045, 6.3.33).

**Filter papers [of special shape].** H. W. BENTZ, and MELITTA-WERKE A.-G. (B.P. 416,299, 11.12.33. Addn. to B.P. 400,077 and 415,929).

**Manufacture of [continuous sheets of] paper.** W. H. MILLSPAUGH (B.P. 416,792, 21.3.33).

**Resinous compositions.**—See XIII.

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

**Stripping of [dyed] cotton goods.** C. W. WILSON (Amer. Dyestuff Rep., 1934, 23, 527–529).—Methods for stripping direct, basic, and vat dyes are discussed. Special apparatus is described in which fabric dyed with indigoid (*I*) or anthraquinone (*II*) dyes is stripped while completely submerged in a reducing liquor (*III*). (*III*) contains glucose,  $\text{NaOH}$ , and  $\text{C}_5\text{H}_5\text{N}$  for (*I*), and glucose,  $\text{NaOH}$ , Decamine A (I.C.I.),  $\text{Na}_2\text{S}_2\text{O}_4$ , Turkey-red oil, and  $\text{C}_5\text{H}_5\text{N}$  for (*II*). Alternatively, (*I*) may be stripped by boiling in a solution of  $\text{K}_3\text{Fe}(\text{CN})_6$  and  $\text{NaOH}$ . A. J. H.

**Indigosol blue IBC.** G. RUDOLPH (Kunstseide, 1934, 16, 304–305).—The application of this new, very fast,  $\text{H}_2\text{O}$ -sol. vat dye (I.G.) to cotton and viscose rayon by dyeing and padding methods, using the  $\text{NaNO}_2$  process, is described fully. A. J. H.

**Indigosol dyes for foulard dyeing and reserves under such dyeings.** L. CABERTI (Boll. Staz. Sperim. Ind. Carta Fibre Tess., 1934, 29, 480–483).—In dyeing with indigosol dyes (*I*) when vat reserves are used, either steam or nitrite development may be used. When (*I*) not readily oxidised are employed, such dyes as indanthrene-blue may be attacked by the hot  $\text{HNO}_2$ , subsequent treatment in a dil. hyposulphite bath being then required. Formulae for the dye baths and procedures are given for both steam and nitrite development, and also directions for using various chrome and "Rapid Fast" dyes as reserves. T. H. P.

**Application of Sirius dyes to rayon.** G. RUDOLPH (Kunstseide, 1934, 16, 268–270).—Practical details of bleaching and dyeing methods are given. A. J. H.

**Effect of  $p_H$  on adsorption of dyes by cellulose and fillers.** L. FRIEDMAN and D. V. KUYKENDALL, JUN. (Paper Trade J., 1934, 99; T.A.P.P.I. Sect., 147–149).—The important effect of  $p_H$  on the adsorption of two basic and two acid dyes on cellulose,  $\text{Al}_2\text{O}_3$ , and a no. of fillers is demonstrated. No rosin size was present in these experiments. The adsorption of methylene-blue on cellulose, kaolin, and talc follows the Freundlich adsorption law. H. A. H.

**Spray-printing [of fabrics].** H. W. ELLINGER (Kunstseide, 1934, 16, 270–271).—The possibilities and advantages of spray-printing as a means for decorating silk and rayon materials in colours of various degrees of fastness are discussed. For most purposes it is satisfactory to spray (through stencils) solutions of pigments in  $\text{EtOH}$ , but patterns having the highest fastness require a steaming treatment of the printed fabric. A. J. H.

**[Printing] lustre effects on [viscose] rayon.** H. METZL (Textilber., 1934, 15, 460).—Fabric is impregnated with a solution containing  $\text{Na}_2\text{WO}_4$  (200 g.) and Ludigol (8 g.) per litre, dried, overprinted with a vat-dye printing paste containing  $\text{K}_2\text{CO}_3$ , Rongalite C, glycerol, and suitable thickening agents, dried, steamed, impregnated (without previous rinsing) with 10% aq.  $\text{BaCl}_2$  or treated in a winch machine with a more dil. solution, then thoroughly washed (a previous oxidation with dil. acidified  $\text{NaBO}_3$  may be required for certain vat dyes), soaped at 40°, and dried, to give fast-coloured effects on a matt ground. By replacing the vat dye paste by a gum Senegal thickening a white lustrous pattern is obtained on a matt ground. A. J. H.

**Autoxidation as a factor in the deterioration of cotton fabrics impregnated with varnishes.** BOGATIREV, LUBIMOVA, and SOBOLEVA (Textilber., 1934, 15, 457–460).—The progress of deterioration, during storage at various temp., of cotton fabrics impregnated with linseed oil varnishes (*I*) containing Co, Pb, or Fe driers was followed by measuring the changes in tensile strength,  $\eta$  (in cuprammonium solution), and oxy-cellulose content of (*I*). Deterioration of the cotton is accelerated by the presence of a drier and a high temp.

during storage; the effect of the driers decreases in the above order.

A. J. H.

## PATENTS.

**Dyeing of leather.** SOC. CHEM. IND. IN BASLE (B.P. 415,924, 5.12.33. Switz., 21.12.32).—Leather is coloured in shades of brown to black by cupriferos azo dyes of the type amino-phenols or -naphthols or their alkyl ethers, or *o*-hydroxy- or -amino-benzoic acids  $\rightarrow$   $m$ -C<sub>6</sub>H<sub>4</sub>(OH)<sub>2</sub> (I) and derivatives  $\leftarrow$  aminoazo compounds, e.g., 2:1:4-NH<sub>2</sub>·C<sub>6</sub>H<sub>3</sub>(OH)·SO<sub>3</sub>H  $\rightarrow$  (I)  $\leftarrow$  4:4'-NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·N<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·SO<sub>3</sub>H (red-brown). H. A. P.

**Dyeing of chrome leathers with basic dyes.** IMPERIAL CHEM. INDUSTRIES, LTD., and G. S. J. WHITE (B.P. 416,016, 4.3.33).—The leather is first mordanted with a complex heteropoly-acid (phospho-tungstic, -molybdic, or -tungstomolybdic acid) and then dyed in the usual way with the basic dye. H. A. P.

**Delustring of natural and artificial silks.** CHEM. FABR. VORM. SANDOZ (B.P. 415,822, 11.3.33. Switz., 12.3. and 25.5.32).—BaWO<sub>4</sub> or BaMoO<sub>4</sub> is pptd. within the fibres by impregnation with a H<sub>2</sub>O-sol. salt of W or Mo followed by treatment with aq. BaCl<sub>2</sub>. Patterned lustre effects may be obtained by using printing methods. The delustred effects are fast to washing and the softness of the textile material is not impaired. A. J. H.

**Treatment [de-electrification] of textile materials.** BRIT. CELANESE, LTD., D. FINLAYSON, and E. L. GREENWOOD (B.P. 416,028, 2.3.33).—Hydroxyalkylamines and their derivatives, or OH-esters or  $\omega$ -amino-alkylamides of higher aliphatic acids (cf. B.P. 388,426—7 and 388,769; B., 1933, 343), are applied (continuously) to the fabric from a solution in a volatile solvent (5—25% triethanolamine oleate in light petroleum). H. A. P.

**Wetting etc. agents.**—See III. **Dye pastes.** **Lactamisable azo dyes.** **Applying aroylamino-anthraquinones.** **Vat dye preps.**—See IV. **Colouring rubber.**—See XIV. **Coloured photographic material.**—See XXI.

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

**Deterioration of sodium hypochlorite solutions.** W. L. DAVIES (Analyst, 1934, 59, 619—620).—10% aq. NaOCl stored in brown or blue glass bottles at 16—18° lost 50% of its available Cl in 390—420 days. E. C. S.

**Composition and properties of superphosphate.** I. **Determination of water in superphosphate and relation between free water and free acid.** W. L. HILL and K. D. JACOB (J. Assoc. Off. Agric. Chem., 1934, 17, 487—505).—The principal constituents of superphosphate (*S*) and of aq. *S*, and the relations between the hygroscopic, or free, H<sub>2</sub>O (*H*) and the free acid (*A*), and between the composition of the aq. *S* and the H<sub>2</sub>O determination, are discussed. Results are given of drying, distillation, and extraction methods (using EtOH or Et<sub>2</sub>O) for H<sub>2</sub>O determination. *H* is best determined by Et<sub>2</sub>O extraction, which also permits the simultaneous determination of *A*. H<sub>2</sub>O of crystallisation can then be determined by drying the Et<sub>2</sub>O-extracted sample at 120°. E. C. S.

**Acidimetric determination of P<sub>2</sub>O<sub>5</sub> in technical phosphoric acid, aqueous extracts of superphosphates and phosphorites, and in the filtrates after precipitation.** L. N. LOBANOV (Zavod. Lab., 1934, 3, 598—604).—The solution is titrated with 0.1*N*-NaOH ( $\alpha$ -naphtholphthalein), and back-titrated with 0.1*N*-HCl (Me-orange): 1 ml. of acid  $\equiv$  7.1 mg. of P<sub>2</sub>O<sub>5</sub>. The results are 10% low in presence of 30% of Fe<sub>2</sub>O<sub>3</sub> or Al<sub>2</sub>O<sub>3</sub>. Minor modifications of the method for special purposes are described. R. T.

**Differentiation between light and heavy magnesium oxide.** H. WALES and G. L. KEENAN (J. Assoc. Off. Agric. Chem., 1934, 17, 534—536).—The proposed U.S.P. XI sedimentation test cannot be depended on to distinguish between light and heavy MgO. An accurately standardised wt.-bulk relationship test may be used, but microscopical examination may provide the best means of distinguishing between the two types. E. C. S.

**Rapid determination of aluminium in clay.** S. S. SHUKOVSKAJA and M. I. VOLINETZ (Zavod. Lab., 1934, 3, 616—619).—0.1 g. of dry clay is fused with 2 g. of NaOH, the melt extracted with H<sub>2</sub>O, the extract filtered, and Al determined in the filtrate by 8-hydroxyquinoline pptn. R. T.

**Determination of sulphates in cryolite.** A. A. BORKOVSKI and N. A. PORFIRIEV (Zavod. Lab., 1934, 3, 588—591).—1 g. of cryolite is heated during 30 min. at 100° with 25 c.c. of a saturated solution of H<sub>3</sub>BO<sub>3</sub> in conc. HCl, the cooled mixture is filtered, and SO<sub>4</sub><sup>2-</sup> determined in the filtrate + washings as BaSO<sub>4</sub>. R. T.

**Analysis of hydrogen by Schilling's method.** L. I. TURBIN (Zavod. Lab., 1934, 3, 619—623).—Tables are given for use with Schilling's method. R. T.

**Sn compounds in lubricants.**—See II. **Utilising residues from AlCl<sub>3</sub> manufacture.** **Refractories for Na<sub>2</sub>S furnaces.**—See VIII. **Analysis of fertilisers.**—See XVI. **Hypochlorites as germicides.**—See XXIII.

See also A., Oct., 1068, **Prep. of colloidal MnO<sub>2</sub>**, 1077, **Poisoning of NH<sub>3</sub> catalysts by O<sub>2</sub>**, 1082, **Synthesis of NH<sub>3</sub> under high pressures**, 1083, **Prep. of Na tungstate**, 1085, **Determining K as perchlorate**.

## PATENTS.

**Manufacture of hydrofluosilicic acid.** W. PENFIELD, ASSR. to PENNSYLVANIA SALT MANUFG. CO. (U.S.P. 1,938,533, 5.12.33. Appl., 17.2.31).—The gases evolved from the decomp. of phosphate rock are condensed to 25% aq. H<sub>2</sub>SiF<sub>6</sub> by passing them up a tower filled with horizontal, perforated trays over which 25% H<sub>2</sub>SiF<sub>6</sub> is allowed to trickle. A. R. P.

**Treatment of liquids. [Production of aqueous ammonia.]** W. L. SPALDING, ASSR. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,948,948, 27.2.34. Appl., 26.5.30).—In the prep. of aq. NH<sub>3</sub> the partly formed solution (I) is recirculated through a tank and cooler (C), and a gas containing NH<sub>3</sub> is injected before C in such a manner as to cause the circulation; the waste gases are bubbled through fresh H<sub>2</sub>O, thus forming (I). (Cf. U.S.P. 1,785,792; B., 1931, 789.) B. M. V.

**Manufacture of caustic alkalis [soda].** MATHIESON ALKALI WORKS, Assees. of N. C. HILL (B.P. 414,497, 15.6.33. U.S., 13.7.32).—In the causticisation of aq.  $\text{Na}_2\text{CO}_3$  with CaO about 2–5% of MgO is added to the mixture to effect more complete pptn. of the Fe and Al.  
A. R. P.

**Manufacture of sodium carbonate monohydrate.** W. H. ALLEN, W. A. GALE, and C. F. RITCHIE, Assrs. to AMER. POTASH & CHEM. CORP. (U.S.P. 1,937,937, 5.12.33. 31.1.30).—Solutions of  $\text{Na}_2\text{SO}_4$  and  $\text{Na}_2\text{CO}_3$  resulting from the treatment of natural alkali deposits are heated with NaCl, which ppts. the  $\text{Na}_2\text{SO}_4$  as burkeite; the mother-liquor is then heated with  $\text{NH}_3$  to cause  $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$  to crystallise.  
A. R. P.

**Thermic production of phosphorus from crude phosphates.** S. D. GOOCH and F. P. KERSCHBAUM, Assrs. to PEMBROKE CHEM. CORP. (U.S.P. 1,938,557, 5.12.33. Appl., 2.6.30. Ger., 11.6.29).—Phosphate rock (I), clay, sand, and a small amount of carbonaceous material is sintered in a Dwight-Lloyd machine and the sinter smelted in a blast furnace with 1 pt. of coke to 1.5–3 pts. of (I), using air preheated to 700° and running the furnace at such a rate that the coke consumption is 200–800 kg./hr./sq. m. of hearth.  
A. R. P.

**Preparation of anhydrous iron chlorides.** C. F. PRUTTON, Assr. to DOW CHEM. Co. (U.S.P. 1,938,461, 5.12.33. Appl., 11.2.32).—Aq.  $\text{FeCl}_2$  is treated with  $\text{Cl}_2$  to produce  $\text{FeCl}_3$  and the solution boiled with scrap Fe to obtain  $\text{FeCl}_2$  again. Part of this solution is returned to the first step and the remainder evaporated at 120° to produce  $\text{FeCl}_2 \cdot 2\text{H}_2\text{O}$  crystals, which can be dehydrated at 230° without decomp. Treatment of the resulting anhyd.  $\text{FeCl}_2$  with dry  $\text{Cl}_2$  affords dry  $\text{FeCl}_3$ .  
A. R. P.

**Extraction of alumina from aluminous materials.** J. J. CRAWFORD (U.S.P. 1,938,173, 5.12.33. Appl., 16.2.31).—The material is extracted with HCl to give a solution of  $\text{AlCl}_3$  and  $\text{FeCl}_3$ , which is mixed with NaCl and treated with  $\text{NH}_3$  and  $\text{CO}_2$  to ppt. a mixture of  $\text{Al}(\text{OH})_3$ ,  $\text{Fe}(\text{OH})_3$ , and  $\text{NaHCO}_3$ , leaving aq.  $\text{NH}_4\text{Cl}$  from which the  $\text{NH}_3$  is recovered by boiling with CaO. The washed ppt. is calcined and fused, and the product leached with  $\text{H}_2\text{O}$  to give aq.  $\text{NaAlO}_2$  and leave  $\text{Fe}_2\text{O}_3$ .  
A. R. P.

**Manufacture of [aluminium] fluoride materials.** W. H. GITZEN and G. H. WAGNER, Assrs. to ALUMINUM Co. of AMERICA (U.S.P. 1,937,885, 5.12.33. Appl., 29.1.31).—Dry HF is passed up a tower through which  $\text{Al}_2\text{O}_3$  slowly descends, at such a rate that pure  $\text{AlF}_3$  is discharged at the lower end and no HF escapes from the upper. Admixture of Na compounds with the  $\text{Al}_2\text{O}_3$  affords  $\text{Na}_3\text{AlF}_6$ .  
A. R. P.

**Manufacture of catalytic materials.** HOUDRY PROCESS CORP., Assees. of E. HOUDRY (B.P. 416,025, 3.2.33. U.S., 23.3.32).—An activated hydrosilicate of Al, containing < 70%  $\text{SiO}_2$ , as well as oxides of Fe (> 3%), Ca, and Mg, the total oxides being > 10%, is moistened with an inert liquid and moulded into units of uniform wall thickness, which are then heated at > the normal temp. of regeneration by oxidation. The activity of the product may be increased by chemical

treatment before moulding or after heating. Other catalytic material (oxides of Co, Ni, Cu, Mn, V, Mo) may be mixed with the Al hydrosilicate, each catalyst working independently, but reciprocally with the other. A high degree of activity in spite of frequent regenerations is ensured.  
W. J. W.

**Manufacture of barium fluosilicate and its application.** GRASSELLI CHEM. Co. (B.P. 414,110, 28.1.33. U.S., 29.1.32).— $\text{BaSiF}_6$  containing < 0.4%  $\text{Ba}(\text{HF}_2)_2$  is prepared by suspending the commercial product in boiling  $\text{H}_2\text{O}$  and adding aq.  $\text{Ba}(\text{OH})_2$  sufficient to convert the  $\text{Ba}(\text{HF}_2)_2$  into  $\text{BaF}_2$ . The resulting neutral product is a suitable insecticide for cucumber plants.  
A. R. P.

**Deodorisation of carbon dioxide.** R. H. MCKEE (U.S.P. 1,937,832, 5.12.33. Appl., 7.3.30).—Industrial  $\text{CO}_2$  containing odorous impurities is mixed under pressure with about 0.01 wt.-% of  $\text{O}_3$ .  
A. R. P.

**Production of cyanogen chloride.** P. DIETERLE, Assr. to NAT. ANILINE & CHEM. Co., INC. (U.S.P. 1,938,324, 5.12.33. Appl., 14.3.27).—CNCl for use in the production of diphenylguanidine is made by passing  $\text{Cl}_2$  at –5° to –10° into a suspension of powdered NaCN in  $\text{CCl}_4$  containing a small quantity of glacial AcOH.  
A. R. P.

**Hydrogen production.** R. WILLIAMS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,938,202, 5.12.33. Appl., 12.9.30).—A 2 : 1 vol. mixture of steam and  $\text{CH}_4$  is passed at 300–550°/1–50 atm. over a catalyst (I) composed of a 9 : 1 mixture of CaO and finely-divided Ni; (I) is occasionally regenerated by heating to > 800° to expel  $\text{CO}_2$ .  
A. R. P.

**Recovery of sulphur from spent oxide.** W. G. RAGG, J. A. WEIL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 414,074, 26.1.33).—Spent oxide containing > 40% of S with > 2% of tarry matter (I) is heated at 320–350° for 1 hr. to insolubilise (I) and, after cooling, the S is extracted with  $\text{CS}_2$ .  
A. R. P.

**Production of phosphorus oxychloride.** I. A. DAVIES, and IMPERIAL CHEMICAL INDUSTRIES, LTD. (B.P. 416,084, 9.3.33).—A finely-divided alkaline-earth orthophosphate is mixed with finely-ground coal and conc. aq.  $\text{NH}_4\text{H}_2\text{PO}_4$ , and the suspension is evaporated, granulated, and carbonised at approx. 700°. The intimate mixture of C and alkaline-earth metaphosphate thus obtained is treated with  $\text{Cl}_2$  at 500–750°. Reaction is thereby stimulated and the retarding action of  $\text{CaCl}_2$  eliminated.  
W. J. W.

**Systems for producing and storing liquid oxygen.** LINDE AIR PRODUCTS Co., Assees. of F. P. KITSON (B.P. 416,965, 2.1.34. U.S., 5.1.33).—See U.S.P. 1,936,059; B., 1934, 833.

**Catalytic oxidation of gases.**—See I.  $\text{NH}_3$ ,  $\text{H}_2\text{S}$ , and  $\text{CO}_2$  from gases.  $\text{CS}_2$  from benzol purification.—See II. Glass-metal joints [for  $\text{Cl}_2$  tubes].—See VIII. Acid-resistant Pb alloys.—See X. Electrolytic cells.—See XI. Ti pigments.—See XIII.

## VIII.—GLASS; CERAMICS.

**Production of iron-free glass.** I. I. KITAGORODSKI and L. S. LANDE (Compt. rend. Acad. Sci. U.R.S.S., 1934, 3, 267–273).—Of the methods used for purifying

the sand, heating in a stream of HCl gas was most successful. After 30 min. treatment at 900°, the  $\text{Fe}_2\text{O}_3$  was diminished from 0.32–2.07% to 0.04–0.03%; and with preheating at 600° and pulverising, the final product contained < 0.007%  $\text{Fe}_2\text{O}_3$ . J. G. A. G.

**Testing of and a specification for glass-house pot clays.** J. H. PARTRIDGE (J. Soc. Glass Tech., 1934, 18, 128–142; cf. B., 1932, 982).—Based on the results of tests of the resistance to thermal shock, to corrosion, and to “creep” under (tensile) stress at high temp. of various glass pot clays (I), and on the correlation with their service behaviour, the following specification of properties required in (I) is suggested: (1) the linear shrinkage, on firing to the highest temp. employed in glass melting, should be < 2%; (2) no cracking should occur after 30 repetitions of the thermal shock test, in which specimens (6 cm. sq.  $\times$  15 cm.) are heated to 1320°, withdrawn from the furnace, and, with the lower 7.5 cm. surrounded and so kept hot by insulating material, the upper half is cooled by an air stream for 5 min.; (3) to obviate “bursting” of pots, the (I) should extend  $\geq$  0.01–0.06 mm. per day when tested under service conditions of load and temp.; (4) to assess the resistance to corrosion, trial foundings should be made in crucibles of (I); the glass produced should be of good colour, and free from “cord.” The true porosity of pots (after use) made from (I) should be  $\geq$  20%. Tests showed that the rate of creep of refractories at high temp. was increased by the presence of grog, this effect being greater with coarse than with fine grog, and suggested that the rate of creep of aluminous ( $>$  27%  $\text{Al}_2\text{O}_3$ ) was  $>$  that of siliceous (approx. 20%  $\text{Al}_2\text{O}_3$ ) clays. A. L. R.

**Determination of the solubility of soda-lime glasses.** O. G. BURCH (Bull. Amer. Ceram. Soc., 1934, 13, 200–204).—The various tests now in use are discussed and a simple modification is described. 10 g. of crushed, graded (40–50-mesh), and (EtOH) washed glass are treated in a Pyrex flask with 50 c.c. of 0.02N- $\text{H}_2\text{SO}_4$  at 89° for 4 hr. The amount of alkali dissolved is determined by titration (Me-red) with 0.02N-NaOH. J. A. S.

**Casting slip.** E. SCHRAMM and F. P. HALL (J. Amer. Ceram. Soc., 1934, 17, 262–267).—Observations were made on the  $\text{H}_2\text{O}$  permeability and content of the cast, the action of various dispersing agents, and the attack of the mould by the slip. Fine-grained clays, due to their close particle packing, have a lower rate of casting and  $\text{H}_2\text{O}$ -permeability and give drier (denser) castings. Dilution and dispersion of a slip increases the closeness of packing, but flocculation and higher density, having the reverse effect, give a slower casting rate and less dense and wetter castings. Shrinkage from the mould increases with the  $\text{H}_2\text{O}$  retention and is greater with denser slips. Cracking of casts from thin slips is due to shortness of the casts and not to excessive shrinkage. The most important factor controlling dispersion is the  $p_{\text{H}}$  of the medium. Tannic, gallic, and other org. acids have the advantage of being protective colloids; these stabilise the slips and require less alkali to give the same fluidity. Tannic acid greatly decreases the attack of the mould by the alkaline dispersing agents. J. A. S.

**Effects of the variety of flint used on the properties of a hotel china body.** C. L. THOMPSON (J. Amer. Ceram. Soc., 1934, 17, 268–271).—Pebble (I), sand (II), rock (III), and tripoli flints (IV) were used in a series of cone 10 bodies. The  $\text{H}_2\text{O}$  adsorption, the linear expansion to 300°, and the impact strength were little influenced by the nature of the flint. Bodies (I) and (II) were, respectively, the least and most easily crazed in the  $\text{H}_2\text{O}$ -dip test, whilst the series of bodies varied from opacity to good translucency in the order: (I), (IV), (II), and (III). J. A. S.

**Durability of tableware decorations.** C. J. KOENIG and A. S. WATTS (J. Amer. Ceram. Soc., 1934, 17, 259–262).—The usual American hotel dish-washing preps. consist of 0.2–1.5% solutions of  $\text{Na}_3\text{PO}_4$ ,  $\text{Na}_2\text{CO}_3$ , neutral and alkaline soaps, etc. used at 40–80°. In Germany the conditions are less severe (0.25% and 60°). The laboratory tests on American and European overglaze decorations included the action of 0.33% solutions of washing agents at 65° for 15 hr., 3% HCl and 4% AcOH at room temp. for 5 and 24 hr., respectively, and with boiling 1%  $\text{Na}_2\text{CO}_3$  for 0.5 hr. All vitreous wares were equally more durable than was semi-porcelain. Acids had a selective action on the colours (Co blues were very readily attacked); alkalis were harmful to all decorations, probably due to their attack on the glassy phase. Vegetable and fruit juices had no harmful action. The newer type of cleaning agent depends on its emulsifying (as distinct from saponifying) action and is less harmful. The average American washing conditions have a noticeable effect after 15 days' service, but the attack can be greatly reduced by limiting the temp. to 60° and by using an emulsifying agent. J. A. S.

**Effect of de-airing on the fired properties of a terra-cotta body.** R. L. CLARE (Bull. Amer. Ceram. Soc., 1934, 13, 223–224).—The de-airing of a grogged body increased the dry and fired strengths (5% and 4%, respectively), decreased the warping (20%) and adsorption (2%), and did not affect the total shrinkage. J. A. S.

**De-airing process for sewer-pipe and conduit manufacture.** R. C. ZEHM (Bull. Amer. Ceram. Soc., 1934, 13, 224–226).—The plant and its operation are briefly described. J. A. S.

**Ceramic utilisation of residues in the manufacture of aluminium chloride from kaolin.** P. P. BUDNIKOV and M. J. SOLOMONOV (Keram. Rundsch., 1934, 42, 46; Chem. Zentr., 1934, i, 1858).—The residue after treating kaolin (I) with CO and  $\text{Cl}_2$  [approx. 60% of the (I) used] contains  $\text{SiO}_2$  88–96,  $\text{Al}_2\text{O}_3$  12–4%, CaO traces. It is suitable for manufacturing whiteware of good quality. H. J. E.

**New high-grade refractory materials.** Z. UNENO (J. Fuel Soc. Japan, 1934, 13, 91–93).—The manufacture and properties of “Corhart” electro-cast aluminous (e.g., 75%) refractories, which are highly resistant to corrosion by slags, glass, cement, and metals, are described. The main properties of “Corhart Black” (the electro-cast product) and “White” (made by bonding the pulverised cast material) are, respectively: refractoriness, cone 38 (approx. 1850°), cone 36 (1795°); refractoriness under load (2 kg. per sq. cm.)  $>$  1740°.

1400—1560°; linear thermal expansion at 1000° 0.53% for both; thermal conductivity [kg.-cal. per sq. m. per hr. per °C. (for 1-cm. thickness ?)] 3.29 and 1.67 at 300°, 2.95 and 1.40 at 1300°; crushing strength 1000—1500, 350—750 kg. per sq. cm.; porosity 1, 10—20%;  $d$  3.3—3.4, 3.0—3.2; hardness ("Black") 42 (Martin's scale).  
A. L. R.

**Choice of chemically resistant refractory materials for sodium sulphide furnaces.** P. P. BUDNIKOV and S. A. JICHAREVITSCH (Trans. Ceram. Soc., 1934, 33, 368—378).—The suitability of various Russian grogged clay, quartz grog,  $\text{SiO}_2$ , and "Black Dinas" bricks, and MgO, chromite-MgO, and carbonaceous bricks for use in  $\text{Na}_2\text{S}$  furnaces was tested by filling cavities in 75-mm. cubes cut from the bricks with a mixture (I) of powdered  $\text{Na}_2\text{SO}_4$  and coal (3:2 pts. by wt.); after being heated for 1 hr. at 1200° the cubes were cut in two and examined microscopically. The refractoriness of the powdered bricks when mixed with 15% of (I) was also determined. The resistance of grogged bricks to attack by (I) increased as the  $\text{Al}_2\text{O}_3$  content rose, and was influenced by the burning treatment, structure, homogeneity,  $d$ , and the quantity of fluxes. Except for the "Black Dinas," which merit further industrial tests, the  $\text{SiO}_2$  bricks showed considerable corrosion in the slagging test, and marked lowering of refractoriness when mixed with (I). MgO and chromite-MgO bricks showed the greatest resistance to attack by (I), the carbonaceous brick also being highly resistant. The min. requirements of a satisfactory refractory are suggested as:  $\text{Al}_2\text{O}_3$   $\leq$  40%,  $\text{H}_2\text{O}$  absorption  $\geq$  8%, crushing strength  $\leq$  180 kg. per sq. cm., refractoriness  $\leq$  cone 33 (about 1730°), no deformation under load (2 kg. per sq. cm.) below 1320°, and  $\geq$  0.5% shrinkage on firing for 1 hr. at 1400°.  
A. L. R.

**Al in clay.**—See VII.

See also A., Oct., 1081, True  $d$  of kaolin. 1082, Corrosion figures on glass.

#### PATENTS.

**[Suction-gathering] glass [tank] furnace.** L. D. SOUBIER, Assr. to OWENS-ILLINOIS GLASS Co. (U.S.P. 1,949,380, 27.2.34. Appl., 5.11.29).—A curtain composed of separately adjustable refractory slabs, for adjusting the opening under the jack arch between the melting furnace and the forehearth, is described.  
B. M. V.

**Fining of borosilicate glasses.** R. HADDAN. From CORNING GLASS WORKS (B.P. 416,473, 27.6.34).—Borosilicate glasses ( $\text{SiO}_2$   $\leq$  70,  $\text{B}_2\text{O}_3$   $\leq$  5%) of lower gas content and higher ultra-violet transmission than those produced by fining with  $\text{As}_2\text{O}_3$  and  $\text{KNO}_3$  are obtained by adding  $\leq$  1% of NaCl or other chlorides (e.g., of K, Al, or Zn) as fining agent to the batch, and melting at  $>$  1400°.  
A. L. R.

**Glass to metal joint.** J. C. BAKER, Assr. to WALLACE & TIERNAN Co., INC. (U.S.P. 1,937,865, 5.12.33. Appl., 7.3.32).—In making such joints in  $\text{Cl}_2$  tubes the metal is sealed into the glass with fused AgCl.  
A. R. P.

**Coating of surfaces of vitreous or vitrified bodies with metals, alloys, or metallic compounds.** SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE

ST.-GOBAIN, CHAUNY & CIREY (B.P. 413,900, 2.10.33. Fr., 17.7.33).—Metals, such as Ag, Zn, Cu, brass, etc., are sprayed from a Schoop pistol on to enamelled Fe at 250°, hard porcelain at 400°, or glass at 200—400° during cooling from the tempering temp.  
A. R. P.

**Ceramic acoustical material.** R. ERICSON, J. S. OFFUTT, and J. R. PARSONS, Assrs. to UNITED STATES GYPSUM Co. (U.S.P. 1,948,878, 27.2.34. Appl., 22.3.32).—Clay slip is deflocculated, a foam-forming agent is added, then a reflocculating agent which acts after a definite time (e.g., alum), ligneous fibres are incorporated, and the mass is moulded, allowed to set in the mould, dried, and fired.  
B. M. V.

**Refractory article.** T. M. CAVEN, Assr. to CORNING GLASS WORKS (U.S.P. 1,949,038, 27.2.34. Appl., 10.2.27).—The article consists of coarse grains of  $\text{Al}_2\text{O}_3$  and a binding film of  $\text{AlPO}_4$  produced by the addition of  $\geq$  2% of  $\text{H}_3\text{PO}_4$ .  
B. M. V.

**Manufacture of grinding bodies.** DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 416,614, 10.10.33. Ger., 21.8.33).—Abrasive grains (e.g., of SiC, corundum, emery, or glass) are agglomerated by impregnation with binding material (e.g., clays or synthetic resins) and suitable heat-treatment, and are then disintegrated; by moulding (with the addition of more binder if necessary) and heat-treating the resultant granules, highly porous (e.g.,  $\leq$  50%) grinding bodies are produced, in which the size of the pores is much  $>$  that of the abrasive grains.  
A. L. R.

**Manufacture of abrasive material.** CARBORUNDUM Co., LTD. From CARBORUNDUM Co. (B.P. 416,730, 20.3.33).—By wetting grains of abrasive material (e.g., SiC or fused  $\text{Al}_2\text{O}_3$ ) with a thin slip of fusible material (e.g., clay, cryolite, feldspar, or their mixtures, with fluxes if desired), removing excess slip, and firing to the vitrification temp., a rough lustreless surface is produced on the granules, which promotes increased bonding.  
A. L. R.

#### IX.—BUILDING MATERIALS.

**Compound flame furnace for the burning of cement.** N. C. KYRIACOU (Cement, 1934, 7, 309—312).—The requirements and efficiency are described.  
T. W. P.

**Puzzuolanic cements.** R. G. FRANKLIN and A. E. J. VICKERS (Soc. Chem. Ind., Chem. Eng. and Road Groups, Oct. 12, 1934, 4 pp.).—In testing the suitability of burnt clay as a puzzuolana, the temp. of burning should be adjusted to give the best results in the salt-resistance test (I) on the resulting puzzuolanic cement mortar. This temp. differs from that giving the best result on strength tests (II). A mix of 60 Portland cement: 40 puzzuolana gave better results than an 80:20 ratio in test (I). Data are given on tests (I) and (II) with different mixes.  
T. W. P.

**Setting and hardening of Portland and aluminous cements with relation to their resistance to water and mineral sulphates.** A. V. HUSSEY (Chem. & Ind., 1934, 838—847).—The constitution and hydration of Portland (I) and aluminous cements (II) are discussed. The  $\text{H}_2\text{O}$  requirement for the hydration of (II) is  $>$  that of (I), and care is required to

prevent loss of  $H_2O$  by evaporation during curing. Hydrated (II) develops excess  $Al_2O_3$  (gel) but no free  $CaO$ ; high early strengths are attained. The chemistry of the action of acid and sulphate waters on cement is discussed. T. W. P.

**Analysis of mortars, mortar mixtures, and cement mixtures with reference to their technical value.** F. OCCHIPINTI (Atti Congr. naz. Chim., 1933, 4, 759—772; Chem. Zentr., 1934, i, 2024).—Analysis of hardened samples or samples after long storage gives little information as to the original mixture. H. J. E.

**Shrinkage and temperature changes in concretes.** H. RABOZEE (Ann. Trav. Pub. Belge, 1934, 35, 171—197; Road Abs., 1934, No. 551).—Shrinkage ( $S$ ) on small test-specimens is most rapid at early ages and is due to loss of  $H_2O$ ; after 2—3 months with dry-air storage and const. temp.,  $S$  ceases.  $S$  of small specimens is not a guide to movements in large masses.  $S$  of reinforced concrete is  $<$  that of plain concrete. T. W. P.

**Shrinkage in cements, mortars, and concretes.** R. DUTRON (Ann. Trav. Pub. Belge, 1934, 35, 243—249, 347—429; Road Abs., 1934, No. 52).—Shrinkage and expansion depend on the hydration reactions of the cement constituents and the physical causes due to capillarity. The factors and conditions are discussed from the experimental data. T. W. P.

**Clinker cooling studies.** H. M. LARMOUR (Rock Products, 1934, 37, No. 7, 40—44).—The clinker from the kiln is reheated in a reducing atm. to a point approaching the clinkering temp. Details of the method and test data on the resulting cements are given. Increased ease of grinding of the clinker is claimed. T. W. P.

**Effect of trass cement on large masses of concrete.** K. BIEHL (Cement, 1934, 7, 288—290).—As compared with ordinary Portland cement, trass cement shows lower heat of hydration and the resulting concrete has an increased resistance to chemical influences, a high early strength, and low permeability, rendering it suitable for use in mass concrete. T. W. P.

**Chemical studies of wood preservation. I. Problem and plan of attack.** R. R. WILLIAMS. II. **Sampling poles for chemical analysis.** R. E. WATERMAN and C. O. WELLS (Ind. Eng. Chem. [Anal.], 1934, 6, 308—310, 310—313).—I. A crit. discussion. II. A method of sampling is recommended. E. S. H.

**Durability of posts and results of preservative treatment.** D. G. CARTER, H. T. BARR, and J. B. WOODS (Arkansas Agric. Exp. Sta. Bull., 1933, No. 287, 16 pp.).—Creosote was the most effective preservative. Promising results were obtained with waste lubricating oils.  $ZnCl_2$  was satisfactory in field tests. Cu compounds pptd. in the wood were highly toxic to destructive fungi. A. G. P.

**Rotary kilns.**—See I. **Emulsions for roads.**—See II. **Al in clay.**—See VII. **Refractories.**—See VIII.

See also A., Oct., 1080—1, **Effect of F' on thermal synthesis of Ca silicates.**

## PATENTS.

**Making concrete, mortar, and like materials waterproof and non-porous, and also strengthening them.** H. G. LLOYD (B.P. 416,391, 15.3.33).—Concrete foundations, permeable to  $H_2O$ , are treated first with  $Ca(OH)_2$  (I) solution, followed by  $CO_2$ , to form  $CaCO_3$  in the pores of the concrete.  $CaCO_3$  or pozzuolanic materials may be pumped in with the (I). T. W. P.

**Manufacture of a [seamless] pipe or conduit [of fibrous material] and compositions therefor.** C. MASTRANGELO, G. GAMBERINI, and G. ROSSI (B.P. 416,565, 8.12.32. Addn. to B.P. 394,344).—Compositions suitable for extrusion are claimed, consisting principally of fibrous material (*e.g.*, asbestos or cellulose), fillers (clays, cement, mineral sulphates, oxides, etc.), and a binder (copal, diluted in linseed oil and containing a drier, or glue solution). The products, after drying, are hardened by heat if necessary. The dried product may be rendered impermeable by dipping in a bath of K silicate, which is then vitrified by heating; residual hygroscopic tendencies are removed by a second heating in hot oils, which are then oxidised by heat-treatment. A. L. R.

**Production of artificial marble.** J. COLLETT (B.P. 416,774, 9.3.34).—A heat-resisting, non-porous product is obtained by adding to a mixture of equal wts. of ordinary white cement and marble dust (pigmented if required) 5% of a powder consisting of  $CaCO_3$  75,  $CaC_2O_4$  10,  $Na_2B_4O_7$  3, and maize or starch 12%. The mass is then mixed with a suitable binding agent (*e.g.*,  $H_2O$ ) and allowed to set. A. L. R.

**Production of roads and like surfaces, wall coverings, and moulded blocks.** INTERNAT. LATEX PROCESSES, LTD., D. F. TWISS, and E. W. B. OWEN (B.P. 416,742, 8.8.33).—Surfacing material for roads etc. is produced by mixing an aq. dispersion of rubber with cement (*e.g.*, ciment fondu or Portland cement) and a hydrophilic colloid (*e.g.*, Na alginate or gelatin) under conditions such that the latter is converted by chemical action into an irreversible gel prior to setting of the cement. Fibrous material and disintegrated vulcanised rubber may also be incorporated. D. F. T.

**Preserving wood.** H. W. WALKER, AssT. to PENNSYLVANIA LUBRICATING Co. (U.S.P. 1,948,551, 27.2.34. 19.4.30).—The preserving effect of an oil or creosote composition is improved by addition of 0.1—5.0% of  $NHPh_2$  and  $AsCl_3$  in approx. equiv. proportions. B. M. V.

**Fireproof porous materials.**—See V. **Acoustical material.**—See VIII.

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

**Potentiometric titration of iron in ores.** B. A. SOSNOVSKI (Zavod. Lab., 1934, 3, 605—606).—The ore is dissolved according to Zimmermann's method, and 150—200 ml. of solution, containing 15—20 ml. of conc.  $HCl$ , are electrotitrated with standard  $K_2Cr_2O_7$  (e.m.f. 0.45 volt until the galvanometer reading becomes zero, when the e.m.f. is raised to 0.55 volt). R. T.

**"Shorter" process for surface hardening [of steel].** T. SWINDEN (Engineering, 1934, 138, 337).—Heating is effected by an  $O_2$ - $C_2H_2$  burner and quenching by a jet of  $H_2O$ , both mechanically controlled. When specimens of varying composition are treated the surface hardness varies directly with the C content; Cr, Ni, Mo, and Mn have no effect. Microscopical study of transverse sections shows only slight variation with composition. The process is satisfactory in surface hardening over a wide range of steels, and, so far as this property is concerned, the use of alloy steels offers no advantage. C. I.

**Structural changes in hardened and tempered steel.** J. ŠPLIČAL (Chem. Listy, 1934, 28, 208—213, 225—227).—A review of literature. R. T.

**Magnetic method of testing results of thermal treatment of high-speed steel instruments.** A. GELFENBEIN and M. DECHTJAR (Zavod. Lab., 1934, 3, 626—631).—The austenite content and hardness of high-speed steels vary inversely with their magnetic inductance (I). A simple apparatus is described for measuring (I). R. T.

**Determination of chromium in steels containing vanadium and molybdenum.** W. ERHARD (Mitt. Forsch.-Anst. Konz. Gutehoffnungshütte, 1934, 2, 268—270; Chem. Zentr., 1934, i, 1844).—The steel is dissolved in  $HNO_3$  +  $KClO_3$  or in HCl. To the diluted solution aq.  $NaOAc$  and then aq.  $Pb(OAc)_2$  are added, and the pptd.  $PbCrO_4$  and  $PbMoO_4$  filtered and washed. The ppt. is treated with 15 c.c. of 1:1 HCl, 100 c.c. of  $H_2O$  are added after 15 min., together with 1 g. of KI, and the liberated I is titrated with aq.  $Na_2S_2O_3$ . H. J. E.

**Svoboda method of determining sulphur [in iron and steel] by combustion.** E. I. FOGELSON, N. V. KALMIKOVA, and M. I. PINIKIROVSKIE (Zavod. Lab., 1934, 3, 607—612).—Minor improvements of the Svoboda method are suggested; applying these, the method gives results identical with those given by Schulte's method for grey and malleable cast Fe, slightly higher for white cast Fe, considerably higher for high-speed steels, and slightly lower for C steels. R. T.

**Iodometric determination of sulphur in steel and cast iron by combustion.** G. G. BOGDANOV (Zavod. Lab., 1934, 3, 613—614).—Kassler's method (B., 1933, 750) is recommended as being extremely rapid and sufficiently accurate. R. T.

**The corrosion problem.** H. C. CHANDLER (J. New England Water Works Assoc., 1934, 48, 345—349).—The use of cement-lined pipes and treatment of the  $H_2O$  with  $Ca(OH)_2$  (I) is recommended as a preventive of corrosion in mains. In the discussion it is pointed out that the increased cost of (I), the extra soap required, and the reduced efficiency caused by boiler scale when capitalised would more than pay for the lining of the pipe system. C. J.

**Rusting of iron.** W. PALMAER (Nordiske Kjemiker, 1932, 4, 183—236; Chem. Zentr., 1934, i, 1877).—Rusting is discussed on the galvanic element theory. Analyses of rust show the hydrates  $2FeO \cdot H_2O$ ,  $Fe_2O_3 \cdot H_2O$ , and  $Fe_3O_4 \cdot 1.5H_2O$  to be formed. The rate

of rusting at  $20^\circ$  in presence of aq.  $CaCl_2$  has been measured by the  $O_2$  consumed. It increases with the conductivity of the salt solution. H. J. E.

**Phosphate rust-protecting processes.** W. OVERATH (Metallges. Rev., 1934, No. 9, 12—16).—A brief description is given of improvements effected in the Parker process. D. A. C.

**Rapid analysis of converter copper slag.** S. J. FAINBERG (Zavod. Lab., 1934, 3, 583—585).—The finely-powdered slag is sieved (200-mesh); approx. 5.5% of the powder remains on the sieve, and consists of metallic Cu. The powder is heated with 40 c.c. of 50%  $H_2SO_4$  and 5 g. of  $Na_2SO_3$  to evolution of  $SO_3$  fumes (I), 1 c.c. of conc. HCl is added, and the solution again heated to evolution of (I), cooled, diluted to 100 c.c., 1 c.c. of HCl added, and the solution boiled and filtered. The residue (II) ( $SiO_2$  +  $PbSO_4$ ) is boiled with 5% HCl to remove  $PbSO_4$ , and the residual  $SiO_2$  collected, dried, and weighed. Cu, Al, Fe, Zn, and Sn are determined by the usual methods in the filtrate from (II). The complete analysis occupies  $\approx$  12 hr. R. T.

**Determination of bismuth in copper.** L. C. NICKOLLS (Analyst, 1934, 59, 620—621; cf. B., 1934, 150, 764).—The pptn. method, using  $Fe(OH)_3$  as coprecipitant, gives substantially accurate results with  $10^{-5}$  g. of Bi. E. C. S.

**Analysis of "Bandrat" babbitt metal.** I. U. MARTINTSCHENKO (Zavod. Lab., 1934, 3, 660—661).—1 g. of metal is dissolved in 10 ml. of conc.  $HNO_3$ , the solution evaporated to dryness, the residue boiled with 70 ml. of  $H_2O$  and filtered, and the ppt., containing Sn and Sb, ignited and weighed. 6 ml. of 50%  $H_2SO_4$  are added to the filtrate, and the ppt. of  $PbSO_4$  is collected, ignited, and weighed. The filtrate is diluted to 200 ml., Cu, Cd, and Zn are determined electrolytically in 100 ml., and As by Ledebur's method in the remaining 100 ml. R. T.

**Effect of surface strain on solid solubility [of metals].** A. PHILLIPS and R. M. BRICK (Met. & Alloys, 1934, 5, 204—205).—Strips of Al-Cu alloy (5% Cu) of perfectly homogeneous composition were given a dissolution treatment for 60 hr. at  $545^\circ$ , quenched in cold  $H_2O$ , polished, heat-treated for varying times at  $410$ — $530^\circ$ , and then etched with HF and studied microscopically. In all cases the second heat-treatment resulted in the surface plane containing  $CuAl_2$  far in excess of that to be expected under equilibrium solubility. It is suggested that the strains set up by cold treatment reduced the solubility of Cu in Al with formation of the ppt. This is only slowly redissolved, and in the meantime during the second heat-treatment Cu migrates outwards to the surface. C. I.

**Silicon-zinc-copper alloys.** T. R. EDMUND (Z. ges. Giessereipraxis, 1934, 55, 33—35; Chem. Zentr., 1934, i, 2033).—Cu containing 10—14% Zn and 4—5% Si has many important properties common to brass and bronze. With const. Zn content the hardness increases and the expansion decreases with Si additions up to 5%. Sn and Ni impurities are detrimental. H. J. E.

**Rapid potentiometric determination of copper in alloys.** H. B. HOPE and M. ROSS (Ind. Eng. Chem. [Anal.], 1934, 6, 316—318).—Cu is pptd. as CuCNS by adding excess of KCNS, the ppt. is filtered off, and the excess of CNS' titrated potentiometrically with  $KIO_3$ .

E. S. H.

**Bearing metals of lead hardened with alkali and alkaline-earth metals.** L. E. GRANT (Met. & Alloys, 1934, 5, 191—195; cf. B., 1934, 965).—Alloys of Pb with Ca etc. are made commercially by electrolysing a mixture of  $CaCl_2$  and  $BaCl_2$  in an Fe pot over molten Pb. A central graphite anode is used with an e.m.f. of 10 volts. Analyses and physical properties of 4 bearing metals (I) made in this way are given. One is stabilised to oxidation by the presence of 0.04% Li. Certain difficulties in melting and casting are discussed. Practical trials with "Satco" metal (Sn 2.40, Ca 0.15, K 0.07%) are described. These alloys are harder than the Sn-base (I) generally used, but will not carry so high loads or function so well at high speeds. Further, under service conditions Satco is corroded by free fatty acids in the lubricant.

C. I.

**Analysis of cassiterite ores.** F. A. FERJANTSCHITSCH (Zavod. Lab., 1934, 3, 581—583).—The powdered ore, containing  $\geq 0.25$  g. of Sn, is ignited to complete oxidation of sulphides, boiled with 20 c.c. of conc. HCl to dissolve  $Fe_2O_3$ , 5 c.c. of conc.  $HNO_3$  are added, and heating is continued to complete dissolution of tungstates, when the solution is diluted and filtered. The residue is washed with  $H_2O$  and aq.  $NH_3$ , dried, and fused with 1:1  $Na_2CO_3$ -S mixture, the aq. extract of the melt is boiled with AcOH, and the ppt. of  $SnO_2$  is fused with KOH at 280—320°. The aq. extract of the melt is made feebly acid and Zn is added, when Sn is completely pptd. after 30 min. Excess of HCl is then added, the solution boiled to dissolution of Sn and excess Zn, 10 c.c. of 0.1N-KI and 1 g. of  $NaHCO_3$  are added to the cooled solution, followed immediately by excess of 0.1N- $KBrO_3$ , and the I liberated according to the following equations is titrated with 0.1N- $Na_2S_2O_3$ :  $BrO_3' + 3Sn'' + 6H' \rightarrow Br' + 3Sn'''' + 3H_2O$ ;  $BrO_3' + 5Br' + 6H' \rightarrow 3H_2O + 6Br$ ;  $Br + KI \rightarrow KBr + I$ . The mean error is  $\geq 0.1\%$ .

R. T.

**Tin research and development.** D. J. MACNAUGHTAN (Misc. Publ. Internat. Tin Res. and Dev. Council., July, 1934, No. 1, 10 pp.).—A lecture. E. S. H.

**Chlorate staining agents [for metals or alloys].** H. KRAUSE (Mitt. Forsch.-Inst. Proberamt. Edelmet., 1933, 7, 17—24, 66—70; Chem. Zentr., 1934, i, 1875).—A crit. discussion of current methods. The effect of  $NH_4NO_3$  additions to the colour bath has been investigated.

H. J. E.

**Origin of odour and taste of metals.** E. RAUB (Angew. Chem., 1934, 47, 673—675).—Ag, Cu, and alloys of these (brass, bronze, alpukka) develop an unpleasant odour and taste when used for culinary purposes. This is due to the formation of metallic complexes of mercaptans and thio-ethers, which are very tenaciously held by the metal surface. The film may be removed by dil. acids, nascent H, or by warming to 200—300°, but the usual polishes and  $Na_2CO_3$  are useless for this purpose.

S. C.

**Testing of metals for creep on torsion.** M. M. ORLOV (Zavod. Lab., 1934, 3, 652—654).—The torsion method is recommended.

R. T.

**Electrodeposition of chromium from aqueous chromic acid solutions containing hydrofluoric acid.** E. MÜLLER and H. DRECHSEL (Z. Elektrochem., 1934, 40, 707—713).—Advantages are claimed for a solution in which the usual  $H_2SO_4$  is replaced by HF. The current efficiency, throwing power, and appearance of the deposit are compared.

E. S. H.

**Electrolytic winning of zinc from roasted blende with ammoniacal ammonium sulphate solution.** G. GRUBE and J. G. GRUNENFELDER (Z. Elektrochem., 1934, 40, 677—685).—Zn can be deposited with an efficiency of 90% from solutions of  $Zn(NH_4)_2SO_4$  (I), with a c.d. 0.02—0.04 amp. per sq. cm. at 25°, using a stainless-steel anode and an Al cathode. Equilibrium potentials of Zn in these solutions, and the equiv. conductivities of the solutions, have been measured. Two extractions of roasted blende with aq. (I) + aq.  $NH_3$  succeed in winning 90% of the Zn. Most of the impurities are removed by stirring with granulated Zn, and the solution is then electrolysed as described, giving 99.95% Zn with a current efficiency of 90%.

E. S. H.

**Electrolytic determination of copper in aluminium alloys.** J. A. KLATSCHKO and G. F. BURLAK (Zavod. Lab., 1934, 3, 585—588).—1 g. of alloy is dissolved in a mixture of 20 c.c. of  $H_2O$ , 1 c.c. of conc.  $H_2SO_4$ , and 3 c.c. of conc.  $HNO_3$ ,  $H_2O$  is added to 150 c.c., and a current of 0.5—2 amp./2—3 volts is passed during 10 min. at 80°, using a rotating spiral anode (1200 r.p.m.).

R. T.

**Welding joints. Cooling mixtures [for steel].—**See I. Cr-Ni alloys for sulphite digesters.—See V. Refractories.—See VIII.

See also A., Oct., 1064, Systems Al-Zn, Ru-Hg, Pb-Tl, Pb-Tl-Cd, and Cd-Li. Au<sub>2</sub>Pb. 1065, Systems Mg-Sn and Li-Tl. 1067, Flotation of Pb glance powder. 1077, Electrolysis of MeOH solutions of Zn and Cd chlorides. 1080, Prep. of Ca-Si alloys. 1082, Prep. of Ce and its alloys.

## PATENTS.

**Ore concentration [by flotation].** A. C. MUNRO (U.S.P. 1,937,837, 5.12.33. Appl., 9.6.30).—Claim is made for a combination of the Forrester rougher type of flotation machine with a rake classifier which removes the coarser portion of the tailings for regrinding.

A. R. P.

**Beneficiation of [banded iron] ores.** A. J. JONES (U.S.P. 1,937,822, 5.12.33. Appl., 12.9.31).—The ore in coarse lumps is passed together with coal through an inclined rotary kiln heated at the upper end to bright redness and the gases liberated are taken from the lower part of the furnace and utilised for firing the upper part. This procedure results in reduction of the outer surface of the Fe-ore bands and facilitates subsequent crushing and separation of the gangue.

A. R. P.

**Manufacture of malleable iron.** W. COTTERILL (U.S.P. 1,938,516, 5.12.33. Appl., 25.8.30).—Cast Fe

is heated for 2—4 hr. at 930—980° in an atm. of  $N_2$  and  $CO_2$  (from complete combustion of carbonaceous fuel) to cause the graphite to dissolve in the Fe, then cooled to 650° to produce pptn. of finely-divided C while admitting 1—3%  $O_2$  into the gas mixture to oxidise a portion of the temper C. A. R. P.

**Preparation of [hydrogenation] catalytic agents [iron, cobalt, or nickel].** E. R. W. DE MAHLER and C. IMERETINSKY (B.P. 414,370, 1.11.32).—A solution of an Fe, Co, or Ni halide in an org. solvent is treated with a more electropositive metal; e.g.,  $FeCl_3$  in  $Et_2O$  is reduced with Mg ribbon to give an active Fe powder catalyst, and  $NiCl_2$  in boiling xylene is reduced with Zn powder. The ppts. are washed with  $Et_2O$  in the absence of  $O_2$ . A. R. P.

**Production of magnetic alloy powders [containing nickel and iron] and magnetic cores therefrom.** STANDARD TELEPHONES & CABLES, LTD. From HERAEUS VACUUMSCHMELZE A.-G. (B.P. 413,526, 29.9.33).—A solution of  $FeCl_2$  and  $NiCl_2$  containing the metals in a 1:4 ratio is evaporated to dryness, the  $H_2O$  of crystallisation expelled by heating at 110°, and the residue heated in  $H_2$  at 600° to obtain an Fe-Ni alloy powder for the manufacture of dust cores. Co salts may be introduced into the solution when a ternary alloy is required. A. R. P.

**Coating of iron and steel.** J. L. BRAY (U.S.P. 1,948,505, 27.2.34. Appl., 18.1.32).—The metal is hot-galvanised, cooled below the m.p. of Zn, and dipped in Pb containing Zn in amount < the saturation quantity [0.1—1.2 (0.5)% Zn], the content being held within limits by addition of Pb from time to time. B. M. V.

**Testing of steel.** L. E. HOWARD, ASSR. to SIMONDS SAW & STEEL Co. (U.S.P. 1,937,820, 5.12.33. Appl., 17.12.29).—Soft spots in steel may be detected by sand-blasting with a uniform coarse-grained sand and examining the depths of the scores produced by oblique illumination. A. R. P.

**Hardening of metals and alloys [steels].** I. BUDOWSKI and H. BARDT (B.P. 414,108, 27.1.33. Ger., 16.7.32).—Fe and steel articles are painted with a mixture of peat coke or similar carbonaceous material, Fe, Co, or Ni powder, and a powdered org. salt of an alkali or alkaline-earth metal, e.g., acetates, formates, oxalates, or carbonates. The coated articles are then heated at > the A3 point of the metal until a sufficient thickness of hard layer is produced. A. R. P.

**Case-hardening of iron, steel, and alloy steels.** ROESSLER & HASSLACHER CHEM. Co. (B.P. 414,092, 27.1.33. U.S., 27.1.32).—Claim is made for a case-hardening bath comprising a fused mixture of alkali chloride with < 5% of an alkali cyanide and > 20% of a  $B_2O_3$  compound (preferably 1% of  $B_2O_3$ ). A. R. P.

**Austenitic corrosion-resisting [nickel-chromium steel] alloys.** F. M. BECKET and R. FRANKS (B.P. 414,211, 6.7.33).—Inter-cryst. breakdown at 500° of Ni-Cr steels with Cr 12—30, Ni 6—30, and C < 0.3 (< 0.12)% is prevented by addition of Nb + Ta in amount > 10 times the C. The Ta:Nb ratio is preferably 1:3. [Stat. ref.] A. R. P.

**Corrosion-resisting ferrous alloy adapted for forging and casting purposes.** SOC. ANON. DES HAUTS-FOURNEAUX, FORGES, & ACIERIES DE POMPEY (B.P. 413,895, 15.9.33. Fr., 26.9.32).—The alloy contains Fe with C < 0.05, Mn 0.2—0.6, Si 0.2—0.6, P < 0.02, S < 0.015, Cu 0.3—0.6, and W 0.01—0.15% and is made by refining Fe under a CaO slag until the C is almost completely oxidised, removing the slag and replacing it with a new CaO slag, adding Mn, W, and Cu, and casting into chill or sand moulds. A. R. P.

**Steel alloy.** J. P. GILL, ASSR. to VANADIUM ALLOYS STEEL Co. (U.S.P. 1,938,221, 5.12.33. Appl., 16.3.32).—A die for use in die-casting Al-base alloys consists of steel containing C 0.2—0.5 (0.28), Si 0.1—1.5 (1.27), Mn 0.1—1 (0.23), Cr 3—8 (4.76), Mo 1.5—2.75 (1.5), W 0.5—2 (1), and Ni 0.5—2.0 (1)%. A. R. P.

**Production of [chromium and manganese] metal and alloys with very small contents of carbon.** G. ANDERSEN (B.P. 413,570, 16.12.33. Norw., 31.12.32).—In the manufacture of low-C steels containing Cr and/or Mn the charge of ore, limestone, and ferrosilicon is heated at 1000—1100° in an oxidising atm. to expel  $CO_2$  and burn off any C before being added to the main bath of molten metal. A. R. P.

**Metallurgical process. [Refining of brass.]** W. F. ZIMMERLI, ASSR. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,937,934, 5.12.33. Appl., 3.2.32).—Addition of Na, preferably as a 2% Na-Zn alloy, to molten brass results in castings of a more uniform grain structure. A. R. P.

**Manufacture of copper alloys.** H. W. BROWNSDON, M. COOK, H. J. MILLER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 413,332—3, 13.1.33).—(A) Age-hardenable Cu alloys are prepared by melting Cu,  $\alpha$ -brass, or  $\alpha$ -bronze with a 1:3—4 Al-Ni alloy to give a product containing Ni 4—10 and Al 1—3%. (B) Alloys of Cu with Zn 0—37, Sn 0.5—15, Al 0.5—5 (1—3), and Ni 1.5—15% (Al:Ni = 1:3—4) are hardened by quenching from 900° and ageing at 300—600°. A. R. P.

**Corrosion-resistant and malleable [copper-zinc] alloys.** OESTERR. DYNAMIT NOBEL A.-G. (B.P. 414,212, 7.7.33. Austr., 8.7.32).—Cu-Zn alloys with 31—40% Cu ( $\gamma$ -brass) are rendered malleable by addition of 3—9.5% Ni and/or Co, preferably < 5% Co or < 7% Ni. The added metal(s) should not be > the limit of solid solubility. A. R. P.

**[Refining] metals [copper and nickel] and alloys [bronze].** DEUTS. GOLD- u. SILBER-SCHNEIDANSTALT VORM. ROESSLER, ASSR. of R. RIEDELBAUCH and J. COLLAS (B.P. 413,881, 9.8.33. Ger., 9.8.32).—P or B is introduced into the molten metal or alloy by means of a flux consisting of a mixture of phosphate or borate with an alkali ferrocyanide (as reducing agent) and an alkali silicate or  $SiO_2$  as a diluent. A. R. P.

**Copper-base alloys [for condenser tubes].** D. K. CRAMPTON, ASSR. to CHASE COMPANIES, INC. (U.S.P. 1,938,172, 5.12.33. Appl., 24.3.33).—Claim is made for an alloy of Cu 75—95 (80), Al 0.5—4 (2), Sn 0.25—2 (1), and Zn > 24 (17)%. A. R. P.

**Metal [antimonial lead] refining process.** A. E. HALL (U.S.P. 1,938,101, 5.12.33. Appl., 11.8.31).—The metal is fractionally crystallised by a modification of the Pattinson process. Antimonial Pb (4% Sb) yields as end-products alloys with (i) Pb 99.6, Sb 0.4%, and (ii) Pb 91, Sb 9%. The process can be used also for the treatment of Pb containing Sn, Cu, As, Bi, Cd, Mg, Ca, Na, or Zn. A. R. P.

**Lead alloys resistant to corrosion by acids.** Soc. MIN. ET METALLURG. DE PENARROYA (B.P. 414,606, 7.3.34. Fr., 7.3.33).—The Pb is alloyed with Cu 100—1500, Te 100—1000, and Ni or similar metal 0—100 g. per metric ton. The additions produce no hardening effects, but greatly increase the resistance of the Pb to conc. H<sub>2</sub>SO<sub>4</sub>. A. R. P.

**Separating and recovering metals and alloys.** C. B. WHITE (U.S.P. 1,938,239, 5.12.33. Appl., 7.8.33).—In the liquation of a metal of low m.p. (I) from an alloy of it with a metal of high m.p., the alloy is heated on a slightly inclined, vibrating screen through which (I) drips into a collecting trough. A. R. P.

**Hot-working and more particularly melting and casting of magnesium, magnesium waste, and magnesium alloys.** OESTERR. AMERIKAN. MAGNESIT A.-G. (B.P. 414,577, 28.12.33. Austr., 7.1.33).—The metal is washed with a hydrocarbon oil which volatilises during subsequent heat-treatment or melting and provides a non-oxidising atm. around the metal. A. R. P.

**[Manganese-magnesium-copper]-aluminium alloys.** H. W. CLARKE and L. ATTCHISON (B.P. 413,865, 29.6.33).—Claim is made for Al alloys containing Mn 0.7—1.5 (0.83), Mg 1.4—1.8 (1.48), and Cu 3.5—4.5 (4.41)%, together with the usual impurities of Fe (0.3) and Si (0.18%). The extruded alloys are quenched from 480° and aged at room temp. A. R. P.

**Soldering fluxes [for aluminium alloys].** ALUMINIUM, LTD. (B.P. 413,519, 7.9.33. U.S., 29.12.32).—Claim is made for mixtures of fluxing salts with solid chlorinated Ph<sub>2</sub> (45—75), solid chlorinated C<sub>10</sub>H<sub>8</sub> (10—20), and *p*-C<sub>6</sub>H<sub>4</sub>Cl<sub>2</sub> (10—35%) diluted with any suitable volatile org. solvent. A. R. P.

**Manufacture of aluminium bronze powder.** ALUMINIUM, LTD., Asses. of F. C. ARTHUR (B.P. 414,041, 23.1.33. U.S., 13.5.32).—See U.S.P. 1,920,234; B., 1934, 329.

**[Composite] protective coating of pipes particularly for marine use.** F. B. DEHN. FROM HUME PIPE (FAR EAST), LTD. (B.P. 416,466, 3.4.34).

**Hg-vapour generators.**—See I. Glass-metal joints. Coating alloys etc.—See VIII.

## XI.—ELECTROTECHNICS.

**Féry-Carbone dry tin accumulator.** C. J. V. FÉRY (Tech. Publ. Internat. Tin Res. and Dev. Council, April, 1934, C, No. 1, 5 pp.).—The cell comprises a Sn negative electrode and a PbO<sub>2</sub> positive electrode, packed in a finely-divided ceramic material saturated with H<sub>2</sub>SO<sub>4</sub>. It evolves no gas during discharge or storage, and can be sealed and kept in an inverted or other position. The e.m.f. is 1.9 volts. E. S. H.

**Oxide insulation of aluminium-plated cables.** V. PLOTNIKOV, N. GRATZIANSKI, and Z. DEMTSCHENKO (Mem. Inst. Chem. All-Ukrain. Acad. Sci., 1934, 1, 111—118).—The cable, coated with a layer of Al < 0.03 mm. thick, is immersed in 2% NaOH, washed with H<sub>2</sub>O at 50°, and oxidised by passing through a bath containing 3—4% H<sub>2</sub>C<sub>2</sub>O<sub>4</sub> at 25—30°, with a current of 2—4 amp. and 120 volts per sq. dm. of surface of the wire. R. T.

**Applications of *p*<sub>H</sub> values in raw-material testing [for light electrical engineering industries].** ANON. (Ind. Chem., 1934, 10, 387).—Existing simple colorimetric methods are described. The limited meaning of *p*<sub>H</sub> val. as applied to solid materials (e.g., paper) is indicated, and a uniform method of extraction of the solid, involving 2 g. of sample/100 c.c. of H<sub>2</sub>O and 10 c.c. of test solution/0.5 c.c. of indicator, is suggested, conditions of extraction and filtration being varied according to the material. Papers for fixed condenser dielectrics should give *p*<sub>H</sub> 6.0—7.5, coil and cable papers 8.5, interleaving coil papers 3.0—3.5, and waxed linings or wrappings 4—9. J. G.

**Determination of dielectric constants, and their importance in commerce and in the laboratory.** R. BÜLL and O. ZWECKER (Chem.-Ztg., 1934, 58, 801—802).—A direct-reading instrument (the dielmeter) for the determination of dielectric const. (ε), using the ordinary heterodyne method, is described. The importance of ε in the determination of small amounts of H<sub>2</sub>O in oils, the rapid determination of the purity of esters and essential oils, and the control of distillation processes is emphasised. A. J. M.

**Röntgenographic determination of residual strains.** M. SHELDON, G. KURDJUMOV, and A. PROTOPOPOV (Zavod. Lab., 1934, 3, 631—640).—A description of known methods. R. T.

**Titration Fe in ores. Testing thermal treatment of steel. Analysis of babbitt metal. Determining Cu in alloys. Pb bearing metals. Cr-plate. Zn from roasted blende. Determining Cu in Al alloys.**—See X. Electrical consts. of soil.—See XVI. Oxidation-reduction potential in beer.—See XVIII. Proofing cabinet. Hg-vapour lamp. Tests for "breakdown" in potatoes.—See XIX.

See also A., Oct., 1060, Conductivity of Te, and (1065) of Mg-Sn and Li-Tl alloys. 1077, Electrolysis of MeOH solutions of Zn and Cd chlorides.

## PATENTS.

**Electrolytic cells.** I. G. FARBENIND. A.-G. (B.P. 416,495, 15.3.33. Ger., 16.3.32).—Small diaphragm chambers, each containing one coherent electrode, are arranged within a large chamber containing one electrode of fine material. J. S. G. T.

**[High-pressure] electrolytic cells.** R. ARMBRUST (B.P. 416,900, 21.3.34).—Cells for the production of H<sub>2</sub> and O<sub>2</sub> etc., having bi-polar series electrodes (*E*), are claimed. *E* have arched or funnel-shaped bottoms, in the form of vessels or cylinders of gradually decreasing size, inserted one inside another, with interposed diaphragms. J. S. G. T.

**Electrolytic device [condenser].** P. ROBINSON, Assr. to SPRAGUE SPECIALTIES Co. (U.S.P. 1,938,464,

5.12.33. Appl., 17.4.31).—The condenser comprises a Cr-plated Al container (I) containing a corrugated Al electrode and a suitable borax electrolyte. (I), which acts as cathode, may be coated with V or Mn instead of being Cr-plated. A. R. P.

**Preparation of fluorescent screen substances for cathode-ray excitation.** A. C. COSSOR, LTD., Assees. of (BARON) M. VON ARDENNE (B.P. 414,597, 22.2.34. Ger., 22.2.33).—CaWO<sub>4</sub> or ZnS is impregnated with minute amounts of Cu or other metal by cathodic sputtering in vac. A. R. P.

**Manufacture of photoelectric tubes.** W. KLUGE, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,938,431, 5.12.33. Appl., 18.5.31. Ger., 21.5.30).—The interior of the tube (I) is partly coated with Ag. (I) is evacuated, and O<sub>2</sub> admitted at < 1 mm. pressure, and a pulsating glow discharge is produced in (I) to form a yellow film of Ag<sub>2</sub>O on the Ag. A small quantity of K is placed in (I), which is heated while a voltage is applied to the terminals to coat the Ag with K and sensitise the tube. A. R. P.

**Television receiving lamp [valve].** D. D. KNOWLES, Assr. to WESTINGHOUSE ELECTRIC & MANUFACTURING Co. (U.S.P. 1,948,720, 7.2.34. Appl., 5.2.29).—The atm. in the valve comprises a noble gas and H<sub>2</sub> (*e.g.*, Ne 90, H<sub>2</sub> 10%) and the space between the plate-like electrodes is > the mean free path of an electron. B. M. V.

**Production of finely-divided magnetic bodies.** C. E. EVERY-CLAYTON. From F. KRUPP A.-G. (B.P. 416,761, 29.11.33).—A mass composed of individual magnetic substances is subjected to a deformation process, *e.g.*, rolling, hammering, forging, or drawing. J. S. G. T.

**Examination of objects by means of X-rays.** F. LANGE and A. BRASCH (B.P. 416,482, 13.2.33. Ger., 13.2.32).—A voltage < 500,000 volts is used to generate X-radiation so that direct and scattered radiation can be separated by simple filtering. J. S. G. T.

**Treating coal gas. Hydrocarbon-metal sol.**—See II. **Magnetic powders.**—See X.

## XII.—FATS; OILS; WAXES.

**Double decompositions between acids and salts.** C. BERGELL (Allgem. Oel- u. Fett-Ztg., 1934, 31, 145—151).—Powdered dried soap, exposed to a current of CO<sub>2</sub>, became saturated in about 5 hr., the total acidity (*A<sub>t</sub>*) of the soap being 15% (of the fatty content), of which 7% represented acidity due to fatty acids (*A<sub>f</sub>*), and the remainder bicarbonate; on keeping in air saponification gradually occurs, equilibrium being reached at about 1% of free fatty acids. *A<sub>t</sub>* is best determined by dissolving the soap in a small measured excess of abs. EtOH-alkali, and back-titrating with alkali after cooling. For the determination of *A<sub>f</sub>*, the soap is dissolved in neutralised Et<sub>2</sub>O-EtOH, in which bicarbonates are insol., and titrated with EtOH-KOH. E. L.

**Examination of refinery residues [soap stock].** F. WITTKA (Allgem. Oel- u. Fett-Ztg., 1934, 31, 359—360; cf. B., 1932, 312).—The author's previous contention is reaffirmed, *viz.*, that in the case of soap stocks from low-grade, impure oils, hydrolysis of neutral

glycerides may occur when the sample is boiled with acid, although this may not happen in the case of high-class samples of the type examined by Better and Sinskin (B., 1934, 683); the author's modified test is, therefore, more reliable for general use in all cases than the older method. E. L.

**Fat-extraction apparatus.** A. D. HOLMES and M. G. PIGOTT (Ind. Eng. Chem. [Anal.], 1934, 6, 384).—Minor alterations of the apparatus described by the Joint Rubber Insulation Committee (Ind. Eng. Chem., 1917, 9, 310) are described. E. S. H.

**Photochemical studies of rancidity.** M. R. COE and J. A. LECLERC (Oil & Soap, 1934, 11, 189—190; cf. B., 1934, 414, 683, 894).—Rancidity (*R*) and peroxide (*P*) tests on maize and cottonseed oils and lard, irradiated through coloured filters, gave results similar to those reported before. *R* and *P* vals. developed more rapidly in oils exposed to white light (I) (glass filter) than to blue light (II), but after prolonged irradiation the *P* vals. in case (II) exceeded those obtained from (I). E. L.

**Continuous process of [fatty] oil extraction.** H. H. BIGHOUSE (Chem. Met. Eng., 1934, 41, 482—483).—Seeds, *e.g.*, cottonseed, crushed to the required fineness, are continuously fed through a seal on to a revolving plate which distributes them evenly into the annulus (I) between a vertical cylindrical vessel and a concentric internal cylindrical housing (II), steam-jacketed along its lower part, for a vertical screw conveyor. The latter elevates the seeds from the conical bottom of (I) up through (II) and discharges them down a chute. A solvent at a suitable temp. flows down (II) and up through (I), extracting oil countercurrently. Solvent is recovered from the extracted seed and the oil. D. K. M.

**Air-blown linseed stand oil.** F. STECHELE (Farbe u. Lack, 1934, 447—448).—The thickening of blown linseed oil (I) is accelerated when the air blow is sufficient to remove the volatile acids formed; the material of the vessel also has an accelerating effect in the following decreasing order: Al, Zn, Cu, Fe, glass. At high temp. loss of O<sub>2</sub> takes place and subsequent drying of the oil is affected; excessive oxidation yields a solid which is insol. in most solvents. (I) can be distinguished from polymerised linseed oil (II) in that (a) O<sub>2</sub> is evolved at 200°, and (b) it is partly sol. in EtOH; the min. amount of EtOH required to produce turbidity in a C<sub>6</sub>H<sub>6</sub> solution of the oil is a measure of the relative amount of O<sub>2</sub> which has been absorbed. (I) is compatible with nitrocellulose and most resins; it dries more rapidly than (II), requires less drier, and there is reduced tendency for wrinkling of the film, which is, however, deficient in H<sub>2</sub>O-resistance. S. M.

**Esterification of fatty acids, especially the ethylation and methylation of "sulphur" olive oils of high acidity.** G. BIANCHINI (L'Ind. Chimica, 1934, 9, 1167—1170).—When esterified with MeOH or EtOH in presence of HCl, highly acid oleines or olive oils yield products of greatly reduced acidity, especially if an accelerating salt is used. When further purified either by treatment with glycerol at 170° in a vac. and in presence of a catalyst, or by mixing with castor oil,

these products yield oils which are cheaper and better for illuminating purposes than commercial olive oil or neutral seed oils, and were so used on the Italian State railway stations during 1924–1927. T. H. P.

**Surface tension of oils.** E. CANALS and (MLLE.) M. E. FLOUS (*J. Pharm. Chim.*, 1934, [viii], 20, 241–243; cf. A., 1933, 671; B., 1933, 76).—Over the range 16–142°,  $\gamma$  of olive oil varies directly with the temp. H. G. R.

Some additional factors affecting the accuracy of the free fatty acid determination as applied to cottonseed analysis. R. S. MCKINNEY and G. S. JAMIESON (*Oil & Soap*, 1934, 11, 191–192; cf. B., 1934, 893).—The methods employed for hulling the seed and grinding the meats appreciably affect the figures obtained for free fatty acid content. Although the amount of meats obtained by hulling with rolls (A) or with a Bauer mill (B) may be the same, the meats from treatment B yield less oil (of a higher acid val.) than those from A; the retained oil from B (e.g., 9%, compared with 2.5% from A) had a much lower acid val. than the extracted oil. It is recommended that seed samples be dried at 130° for 2 hr. (to destroy lipase and facilitate separation of the oil), treated as in B, and the meats ground in the Wiley mill. (Cf. report of A.O.C.S. Seed Comm., *ibid.*, 198–199.) E. L.

**Interpretation of cottonseed oil-mill products analysis.** E. FREYER (*Oil & Soap*, 1934, 11, 196; cf. B., 1934, 803).—A correction. E. L.

**Rapid determination of free fatty acids in liquid oils.** J. A. BAQUÉ (*Allgem. Oel- u. Fett-Ztg.*, 1934, 31, 358).—Adopting an average  $d$  of 0.917 for calculation, commercial routine determinations can be made rapidly, and with sufficient accuracy, by using a known vol. of oil (5 c.c., conveniently measured out from a calibrated syringe). E. L.

**Determination of the thiocyanogen value, according to H. P. Kaufmann[’s method], of some typical Indian oils, and its application in determining the percentage of oleic, linoleic, and linolenic acids.** I. N. N. GODBOLE, K. C. TRIGUNAYAT, A. DATT, and U. DATT (*Allgem. Oel- u. Fett-Ztg.*, 1934, 31, 143–145; cf. B., 1934, 462).—Hanus I vals. and Kaufmann SCN vals. of linseed and several semi- and non-drying Indian oils have been determined. E. L.

**White lupin flour and oil.** M. F. LAURO (*Oil & Soap*, 1934, 11, 196).—Flour prepared from skinned beans contained H<sub>2</sub>O 4.25, oil 13.65, protein (N  $\times$  6.25) 47.81, ash 2.77, insol. ash 1.48, crude fibre 3.72, carbohydrates (by diff.) 27.8, starch 0.0%. The bitter principle is easily removable. The light petroleum-extracted, semi-drying oil (12.4%) had  $d_{4}^{20}$  0.9193 (0.9180 for another sample), I val. (Wijs) 96.9, sap. val. 190.9, acid val. 7.4, unsaponifiable matter 1.25%, titer 31°, phosphates (as P<sub>2</sub>O<sub>5</sub>) 0.17%. A second commercial sample of beans gave similar results. E. L.

**Passion fruit seed oil.** G. S. JAMIESON and R. S. MCKINNEY (*Oil & Soap*, 1934, 11, 193).—Seeds of *Passiflora edulis* contained 7.92% of H<sub>2</sub>O and 18.17% of oil suitable for edible or technical purposes. The

clarified expressed oil did not deposit “stearine” at 10° and had:  $d_{4}^{25}$  0.9207,  $n_{D}^{25}$  1.4737, sap. val. 190.4, I val. (Hanus) 140.4, SCN val. 81.2, Reichert–Meissl val. 0.11, Polenske val. 0.21, Ac val. (André–Cook) 8.10, unsaponifiable matter 0.62% (I val. 146.2), saturated acids (corr.) 8.88%, unsaturated acids (corr.) 84.31%. The oil contains palmitic 6.78, stearic 1.76, arachidic 0.34 (Me ester fractionation), oleic 19.0, linoleic 59.9, and linolenic acid 5.4% (thiocyanometric analysis). E. L.

**Composition of the oil from *Persea indica*.** M. COVELLO and M. ROSANO (*Atti Congr. naz. Chim.*, 1933, 4, 702–704; *Chem. Zentr.*, 1934, i, 2056).—The kernel from the berries (11.43% on the whole fruit) gave on extraction with Et<sub>2</sub>O 37.5% of an oil having  $d_{4}^{15}$  0.952,  $n_{D}^{15}$  1.4514, solubility in 95% EtOH 45:100. H. J. E.

**Technical preparation and application of synthetic waxes, artificial waxes, and wax-like compounds.** R. STRAUSS (*Chem.-Ztg.*, 1934, 58, 837–839).—A review.

**Lubricating grease. Determining Pb soap in greases.**—See II. Oils, fats, and waxes as anti-septics.—See XX.

See also A., Oct., 1084, **Determination [of fat in chocolate] by extraction.** 1090, **Unsaponifiable matter from oils of elasmobranch fish.** **Prep. of  $\beta$ -monoglycerides.** 1126 and 1145–6, **Vitamins.** 1130, **Sunflower oil.**

#### PATENTS.

**Reducing the oil content of soya.** L. W. HAAS and H. O. RENNER, Assrs. to J. R. SHORT MILLING CO. (U.S.P. 1,947,200, 13.2.34. Appl., 15.6.32).—Fresh soya beans are ground (to pass 20–40-mesh) and the H<sub>2</sub>O content is adjusted to 10–11% (by spraying, or drying at < 65.5°) before cold-pressing at 8000–16,000 lb./sq. in., and the cakes are ground and dried at < 50°, preferably under vac.; the vitamins in the oil, and the enzyme in the meal, which bleaches carotene (e.g., in flour), are unimpaired by the process. E. L.

**Treatment of oleaginous seeds, fruits, and other vegetable material.** J. SCHMIDT (B.P. 416,378, 13.2.33).—The (palm) fruits, seeds, etc. are heated and digested in a closed vessel, fitted with an agitator, under the pressure of the steam generated from the natural moisture of the fruits etc. E. L.

**Derivatives of fatty acids.**—See III. **Measuring vitamin-A.**—See XXI.

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

**Durability of aluminium[–bronze] paints.** F. KOLKE (*Farbe u. Lack*, 1934, 473–474).—Exposure tests of a series of Al-bronze paints show that the composition of the binding medium does not affect the durability if two coats of the paint are uniformly applied, preferably by spraying. Media used for indoor protection gave satisfactory results after 2 years’ outside exposure. The effect is attributed to the formation in the film of overlapping pigment scales. S. M.

**Fungus growth on oil-paint films.** R. KEMPF and F. J. PETERS (*Farben-Ztg.*, 1934, 39, 1019–1021).—

White-Pb-, Ti-white-, and Al bronze-boiled linseed oil paints applied to glass plates and stored in aq.  $MgSO_4$  for 4 weeks develop black streaks of fungus of the *Torula* type, which are shown by photomicrographs to comprise fine threads, close-clustered at the point of development and thinning out down the "tail." In 4 months, especially on the Ti paint, the fungus growth has taken root in the film, involving its gradual destruction by enzyme action. Formation of this black fungus in "rocket" shape is also reported on normal exposure of Ti-white paint on wood, panels on ground level facing north being more affected than similar panels facing south, whilst panels exposed on the roof facing south developed no fungus. These results are discussed and illustrated. Films of the above-mentioned paints immersed in aq.  $K_4Fe(CN)_6$ , NaCl, and Na tartrate develop a white type of fungus as a slime on the film and as flocks suspended in the solutions. This type is, however, not so destructive as the *Torula* type.

S. S. W.

**The tonometer. Colour and brightness of white [paint or enamel] films.** L. A. WETLAUFER (Ind. Eng. Chem. [Anal.], 1934, 6, 376—379).—An instrument for studying the initial yellowness and brightness of the films, and the changes with exposure, storage, temp., etc., is described.

E. S. H.

**Behaviour of pigments in paints on plywood.** H. WAGNER and J. GOHM (Farben-Chem., 1934, 5, 365—368).—Exposure tests were carried out on paints made with a large no. of pigments ground into chlorinated rubber, using only one coat spread on a primer. The results after 1 year show (photomicrographs given) that the paint may (i) only partly fill the pores (I) to form an uneven surface, e.g., with ZnO; (ii) completely fill (I) and thus produce a smooth surface even after exposure, e.g., with white-Pb; (iii) immediately fill (I), but in consequence of deficient elasticity cracking develops along (I) during exposure, e.g., with Zn- and Cr-greens. Case (ii) gave the best resistance. Ideally the paint should possess sufficient flow to fill (I) and then become a plastic body.

S. M.

**Microchemical identification of ink in handwriting.** T. J. WARD (Analyst, 1934, 59, 621—622).—The ink-stroke is treated with a drop of reagent, usually 1% aq. AcOH, and the drop is removed and allowed to dry. The ink is identified by the form and arrangement of the crystals, when possible by the approx.  $n$ , and by the microchemical identification of Al, Cu, Cr,  $SO_4^{2-}$ , Cl<sup>-</sup>,  $H_2C_2O_4$ , and salicylic acid, and of glycerol.

E. C. S.

**Driers and the drying process at low temperatures.** H. WOLFF and G. ZEIDLER (Farben-Ztg., 1934, 39, 897—899, 921—923, 945—947, 967—968, 993—994).—There is an optimum Pb/Mn ratio in a mixed drier, dependent on the temp. of drying. With any ratio, the optimum mixed drier concn. corresponds with 0.1% Mn. Films dried at 5° are softer than those dried at higher temp. The accelerating effect of light and heat is less with linoleates (*L*) than with resins (*R*). At low R.H. (35%) the temp. coeff. of mixed *L* drier is independent of Pb/Mn ratio, whilst that of *R* falls with increasing Pb content. At higher R.H. the temp. coeffs.

are less, and at low temp. driers may even retard drying. The above results are for films 7.5  $\mu$  thick; thickness has some effect on the optimum conditions. Thicker films show higher sap. vals. than thin ones with *L*, but not with *R*. Graphs of wt. increase during drying show several distinct phases, particularly a period of slowing up ascribed to fission of the fatty acids, which is more marked with *L* than with *R*, and stress is laid on the magnitude of the effects due to the purely org. portion of driers.

G. H. C.

**Determination of tautening power of aeroplane dopes.** E. K. O. SCHMIDT (Farben-Ztg., 1935, 39, 1049—1050).—To replace the rough method of evaluating "doped" fabric by testing panels for "drum effect," an apparatus is described comprising a graduated plunger (*P*) passing vertically through a collar in the centre of a long, horizontal duralumin beam which rests on the frame on to which the fabric (*F*) is stretched. The curvature of *F* before and after doping is read off directly on the stem of *P*, which is suitably loaded. Regularity of quality of undoped *F* and effectiveness of various doping schemes may be measured by this device, and typical figures are given illustrating both of these uses. Considerable differences exist in the tautness of systems that may not show up in the simple "drum test."

S. S. W.

**[Manufacture of] cresylic acid resins, varnishes, and moulding powders.** W. McHUTCHISON (Ind. Chem., 1934, 10, 383—386).—The plant, labour, and materials necessary for manufacturing PhOH- $CH_2O$  resins, varnishes, and moulding powders are described. The production of a typical resin batch, using 2½ cwt. of cresylic acid, is explained and illustrated in detail, and the precautions to be observed in the running of the plant are outlined.

E. L. H.

**Shrinkage of phenolic resin plastics.** M. N. ALLEN (Brit. Plastics, 1934, 6, 206—208).—A relationship is traced between fillers, and moulding temp., and the shrinkage of PhOH- $CH_2O$  resin mouldings after ejection from the press, in cooling to room temp. Cooling of the mould before ejection, or predrying of the moulding powder, lowers the % change. Cracks, due to uneven shrinkage or expansion, are avoided by the use of fabric fillers instead of wood-flour fillers.

E. L. H.

**Constitution of artificial resins. Hardenability of resins. VI. Condensation products of aminoarylsulphonamides and aldehydes.** G. WALTER and H. POLLAK. **VII. Condensation products of naphthalene-mono- and -poly-sulphonamides and formaldehyde.** G. WALTER and H. ENGELBERG. **VIII. Resins from saligenin or phenols and hexamethylenetetramine.** G. WALTER and G. REIMER (Kolloid-Beih., 1934, 40, 1—28, 29—44, 45—54; cf. B., 1933, 639).—VI. Hardenability is not observed, even if reactive groups are present in no. sufficient for the purpose, if either one of the groups does not react with aldehyde or if further mol. association of the primary condensate is impeded at one of the two positions, thus rendering impossible the formation of ring-systems on which hardenability depends. *o*- $NH_2 \cdot C_6H_4 \cdot SO_2 \cdot NH_2$  and 37%  $CH_2O$  at 100° yield *o*-methyleneaminobenzene-sulphonmethyleneamide, m.p. 150—157° after softening

at 100—118°. The following compounds are prepared similarly: *m*-hydroxymethylaminobenzenesulphonmethyleneamide, decomp. 252°, converted by diazotisation and coupling with  $\beta$ -C<sub>10</sub>H<sub>7</sub>·OH into *m*-sulphonamidobenzeneazo- $\beta$ -naphthol, m.p. 240°; *m*-hydroxymethylaminobenzenesulphonmethyleneamide, decomp. 244°; compound [NHAc·C<sub>6</sub>H<sub>4</sub>·SO<sub>2</sub>·N(CH<sub>2</sub>·OH)]<sub>2</sub>CH<sub>2</sub>, m.p. 175—186° after softening at 100°, and the corresponding *o*-Me<sub>2</sub> derivative, m.p. 175—185° after softening at 105° (from 3-acetamido-*p*-toluenesulphonamide, m.p. 226°); 4-acetamido-1-methylbenzene-6-sulphonamidodi-(4'-acetamido-1'-hydroxymethylsulphonamido-4'-methyl-6'-N-methylenebenzene), m.p. 180—195° after softening at 115°; methylenedi-(4-amino-3-N-hydroxymethylsulphonamido-6-N-methylenesulphonamidotoluene), decomp. 245°, from *p*-toluidine-2:5-disulphonamide, which in dil. alkaline solution gives 2:5-di(sulphonhydroxymethylamido)-*p*-toluidine, m.p. 237° (decomp.); NH<sub>2</sub>·C<sub>6</sub>H<sub>2</sub>(SO<sub>2</sub>·NH<sub>2</sub>)<sub>3</sub> affords the resin C<sub>29</sub>H<sub>38</sub>O<sub>22</sub>N<sub>12</sub>S<sub>9</sub>, decomp. 277°, converted by dil. KOH into the substances C<sub>8</sub>H<sub>10</sub>O<sub>6</sub>N<sub>4</sub>S<sub>3</sub>, m.p. 288° (insol. in acid), and C<sub>8</sub>H<sub>10</sub>O<sub>6</sub>N<sub>4</sub>S<sub>3</sub>, m.p. 276° (sol. in acid), or, in dil. alkaline solution, the compound C<sub>8</sub>H<sub>10</sub>O<sub>6</sub>N<sub>4</sub>S<sub>3</sub>, m.p. 303°, transformed by CH<sub>2</sub>O into the substance C<sub>21</sub>H<sub>32</sub>O<sub>18</sub>N<sub>8</sub>S<sub>6</sub>, decomp. 255°; *o*-NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·SO<sub>2</sub>·NH<sub>2</sub> and MeCHO yield the compound C<sub>8</sub>H<sub>10</sub>O<sub>2</sub>N<sub>2</sub>S, m.p. 209°, whereas the *m*- and *p*-derivatives afford diethyldeneaniline-*m*-, decomp. 183°, and -*p*-, decomp. 185°, -sulphonamide; benzylideneaniline-*o*-, m.p. 151°, -*m*-, m.p. 158°, and -*p*-, m.p. 174°, -sulphonamide; benzylidene-*p*-toluidine-2:5-disulphonamide, m.p. 325° (decomp.); NH<sub>2</sub>·C<sub>6</sub>H<sub>2</sub>(SO<sub>2</sub>·NH<sub>2</sub>)<sub>3</sub>, *p*-NHAc·C<sub>6</sub>H<sub>4</sub>·SO<sub>2</sub>·NH<sub>2</sub> and *o*- and *p*-NHAc·C<sub>6</sub>H<sub>3</sub>Me·SO<sub>2</sub>·NH<sub>2</sub> do not condense with PhCHO; *o*-, *m*-, and *p*-NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·SO<sub>2</sub>·NH<sub>2</sub> with furfuraldehyde yield substances C<sub>32</sub>H<sub>30</sub>O<sub>11</sub>N<sub>4</sub>S<sub>2</sub>, decomp. 212°, decomp. 198°, and decomp. 215°, respectively, whilst NH<sub>2</sub>·C<sub>6</sub>H<sub>2</sub>Me(SO<sub>2</sub>·NH<sub>2</sub>)<sub>2</sub> and NH<sub>2</sub>·C<sub>6</sub>H<sub>2</sub>(SO<sub>2</sub>·NH<sub>2</sub>)<sub>3</sub> afford, respectively, compounds C<sub>34</sub>H<sub>36</sub>O<sub>15</sub>N<sub>6</sub>S<sub>4</sub>, decomp. 285°, and C<sub>11</sub>H<sub>12</sub>O<sub>7</sub>N<sub>4</sub>S<sub>3</sub>, decomp. 165°; with *p*-NHAc·C<sub>6</sub>H<sub>4</sub>·SO<sub>2</sub>·NH<sub>2</sub>, *o*-NHAc·C<sub>6</sub>H<sub>3</sub>Me·SO<sub>2</sub>·NH<sub>2</sub> and *p*-NHAc·C<sub>6</sub>H<sub>3</sub>Me·SO<sub>2</sub>·NH<sub>2</sub> the substances (·SO<sub>2</sub>·N·CH·C<sub>4</sub>H<sub>9</sub>O)<sub>n</sub>, m.p. 200° (decomp.), m.p. 140°, and m.p. 142°, are obtained.

VII. 1-C<sub>10</sub>H<sub>7</sub>·SO<sub>2</sub>Cl (prepared from C<sub>10</sub>H<sub>8</sub> and ClSO<sub>3</sub>H in CCl<sub>4</sub>) is transformed into 1-C<sub>10</sub>H<sub>7</sub>·SO<sub>2</sub>·NH<sub>2</sub>, which, when treated with CH<sub>2</sub>O and cold KOH, affords  $\alpha$ -naphthalenesulphonidihydroxymethylamide (or, possibly, C<sub>10</sub>H<sub>7</sub>·SO<sub>2</sub>·NH·CH<sub>2</sub>·O·CH<sub>2</sub>·OH), m.p. 120°, whereas when heated it gives the substance C<sub>10</sub>H<sub>7</sub>·SO<sub>2</sub>·N(CH<sub>2</sub>·OH)·CH<sub>2</sub>·NH·SO<sub>2</sub>·C<sub>10</sub>H<sub>7</sub>, which can be hardened. Naphthalene-1:5-disulphonamide, decomp. about 320°, from the 1:5-disulphonyl dichloride, m.p. 183° (improved prep. from C<sub>10</sub>H<sub>8</sub> and excess of cold ClSO<sub>3</sub>H), with cold CH<sub>2</sub>O yields the substance NH<sub>2</sub>·SO<sub>2</sub>·C<sub>10</sub>H<sub>6</sub>·SO<sub>2</sub>·NH·CH<sub>2</sub>·OH, decomp. 270—280° after softening at 245°, whilst with hot CH<sub>2</sub>O the compound C<sub>14</sub>H<sub>14</sub>O<sub>6</sub>N<sub>2</sub>S<sub>2</sub>, softens at 230°, is produced. 1:3:6-C<sub>10</sub>H<sub>5</sub>(SO<sub>2</sub>·NH<sub>2</sub>)<sub>3</sub> and CH<sub>2</sub>O at 118° afford the substance OH·CH<sub>2</sub>·NH·SO<sub>2</sub>·C<sub>10</sub>H<sub>5</sub>(SO<sub>2</sub>·N·CH<sub>2</sub>)<sub>2</sub> which hardens at 100—130°. All three naphthalenesulphonyl chlorides and (CH<sub>2</sub>)<sub>6</sub>N<sub>4</sub> give infusible resins.

VIII. Reaction between PhOH and its derivatives and (CH<sub>2</sub>)<sub>6</sub>N<sub>4</sub> (I) is followed by determination of NH<sub>3</sub>

evolved. All resins down to the ratio CH<sub>2</sub>:PhOH = 0.5—1 are infusible and even with a large excess of (I) the ratio 1CH<sub>2</sub>:1PhOH is not exceeded. The product with the smallest ratio consists exclusively therefore of isomeric dinuclear compounds and a product from isomeric trinuclear substances is infusible. The results are to some extent contradictory to those obtained with saligenin, which yields a poly-membered fusible resin and gives infusible resins with  $\geq$  11 units. For the customary PhOH·CH<sub>2</sub>O condensation it is provisionally assumed that the hardened products consist of mixtures of open chains and ring-systems, whereby in the former CH<sub>2</sub>O:PhOH is  $\geq$  1, whereas in the latter the figure 1.5 is attained. A similar conception explains the results of the condensation of CO(NH·CH<sub>2</sub>·OH)<sub>2</sub>, the products being assumed to consist of open and "ring" chains.

H. W.

Paint as an aid to lighting. ANON. (Nat. Master Painter, 1934, 5, 231—232). D. R. D.

See also A., Oct., 1067, Spreading of cellulose and its derivatives. 1082, Mineral colours. 1102, Action of OH·SO<sub>2</sub>Cl on C<sub>10</sub>H<sub>8</sub>.

## PATENTS.

Preparation of bituminous paint. S. W. SPARKS, ASSR. to R. T. LYTLE, L. W. STOFIEL, and P. S. MCPHERSON (U.S.P. 1,945,869, 6.2.34. Appl., 12.4.33).—Crushed bituminous rock of Kentucky type (8.5 pts.) is disintegrated with HNO<sub>3</sub> (1 pt.), H<sub>2</sub>SO<sub>4</sub> (*d* 1.42) (3 pts.) is stirred in, and before the reaction is complete collodion (1 pt.) is added; the product is a neutral liquid. S. M.

Protective paint or lacquer for food containers. A. WILLIAMS, ASSR. to SWIFT & Co. (U.S.P. 1,945,584, 6.2.34. Appl., 21.5.32. Argentina, 29.9.31).—EtOH, rosin, shellac, a dye, and castor oil are worked up in described manner. S. M.

Production of titanium pigments. TITAN Co., INC. (B.P. 416,615, 20.10.33. U.S., 28.10.32. Cf. B.P. 405,340; B., 1934, 334).—Ilmenite or other Ti ore is calcined with (1—10% of) Pb or a Pb compound. S. M.

[Shellac] spirit varnishes and the like. N. DREY and I. FREEDLAND (B.P. 416,098, 16.3.33).—The shellac or resin mixture is dissolved in a mixture of substantially anhyd. EtOH and a petroleum distillate of b.p. 45—100°. S. M.

[Sprayable] coating composition. E. ROTHEIM (U.S.P. 1,945,998, 6.2.34. Appl., 20.4.32. Norw., 8.10.26. Cf. U.S.P. 1,800,156; B., 1931, 1126).—The paint or lacquer is thinned with sufficient of a liquefied hydrocarbon, *e.g.*, *iso*-C<sub>4</sub>H<sub>10</sub>, to produce several atm. pressure in the container and is then sprayed. For aq. media MeCl is used. S. M.

(A) [Vinyl ester] resinous compositions. (B) Articles made by use of solutions of vinyl ester resins. H. E. POTTS and (B) CANADIAN ELECTRO PRODUCTS Co., LTD. From SHAWINIGAN CHEMICALS, LTD. (B.P. 416,412—3, 8.2.33. [B] Addn. to B.P. 351,082; B., 1931, 853).—(B) Vinyl ester polymerides in which the acid group has been only partly replaced

by acetal formation (by hydrolysis and reaction with an aldehyde) are more sol. in common solvents and can be used, with or without other resins, plasticisers, and fillers, for impregnating paper etc. and for making films, adhesives, and moulding compositions. (A) The flexibility and H<sub>2</sub>O-resistance of the films are increased by incorporating a non- or semi-drying oil, e.g., castor oil.

S. M.

**Manufacture of artificial [resinous] compositions [from vinyl esters].** I. G. FARBENIND. A.-G. (B.P. 416,885, 2.12.33. Ger., 10.12.32).—Substances obtained by interpolymerisation of mixtures of vinyl and acrylic acid esters are hydrolysed and either treated with a strong acid, e.g., H<sub>2</sub>SO<sub>4</sub>, or, in the case of NH<sub>4</sub> salts, merely heated. The products are suitable for films, threads, etc.

S. M.

**Production of [vulcanised urea-aldehyde] artificial resins.** I. KREIDL (B.P. 416,661, 19.12.32. Austr., 19.12.31 and 9.12.32).—Urea or CS(NH<sub>2</sub>)<sub>2</sub> is heated in alkaline solution with an aldehyde in presence of a polysulphide, thiosulphate (I), polythionate (II), etc. containing intercombined S atoms. (I) and (II) may be formed *in situ*. Accelerators used in vulcanising rubber, e.g., K xanthate, may be added. Products containing > 1.5% of S can be hot-pressed.

S. M.

**Manufacture of polymerisation products from aldols.** DISTILLERS CO., LTD., J. V. EYRE, and H. LANGWELL (B.P. 416,734, 1.4.33).—Aldols are polymerised without formation of insol. resins when heated at > 70° with H<sub>2</sub>O in presence of either CaO, MgO, etc., or a more sol. alkali, e.g., NaOH, together with a salt of Ca, Sr, Ba, or Mg.

S. M.

**Production of oil-soluble resins [from phenols].** I. ROSENBAUM (B.P. 416,476, 9.12.32).—By condensing a phenol or cycloketone, e.g., cyclohexanone, with an aldehyde (I) in presence of  $\alpha$ -terpineol, with or without other terpene compounds, the resins formed possess improved solubility in oils and are permanently fusible. Comparatively large proportions of (I) may be used and may be further increased by using *p*-tert-butyl- and -amylphenol. Natural resins, rosin, rosin esters, and polyhydric alcohol-polybasic acid products and a catalyst, e.g., ZnO, Zn(OAc)<sub>2</sub>, may be present. 28 examples are given.

S. M.

**Manufacture of [phenol-formaldehyde] synthetic resin.** F. A. APGAR and A. RUNYAN, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,945,719, 6.2.34. Appl., 21.4.31).—A phenol and CH<sub>2</sub>O or other aldehyde are condensed in presence of hydrocarbon polymerides of b.p. > 300° obtained from the unsaturated components of cracked petroleum distillates of b.p. < 232°; a natural resin or drying oil may also be incorporated.

S. M.

**Production of [phenol-formaldehyde] synthetic resin.** F. S. GRANGER, AssT. to COMBUSTION UTILITIES CORP. (U.S.P. 1,946,459, 6.2.34. Appl., 30.4.30).—In the alkaline condensation of a phenol and CH<sub>2</sub>O, formation of MeOH and HCO<sub>2</sub>Na is reduced by conducting the initial reaction at about room temp. (< 60°). The product is resinified by heating at < 60° for several hr. and the resin pptd. by addition of acid.

S. M.

**Production of press-moulding materials by condensation of phenols with formaldehyde or sub-**

**stances that yield formaldehyde.** DR. F. RASCHIG, GES.M.B.H. (B.P. 416,847, 21.2.33. Ger., 27.2.32).—Phenol (1 mol.) is condensed with a :CH<sub>2</sub> compound (1½–2 mols.) to a fusible resin which is hardened at 50–70° until (i) a Vicat needle (area 2 sq. mm.; load 5 kg.) penetrates to 1 cm. deep in 5–20 min., or (ii) its "rising power" (Krahl), after removal of volatile matter, is 100–200 mm. under 300 kg./sq. cm. The product is pulverised and heated *in vacuo* at 80–100°.

S. M.

**[Apparatus for] mixing of plastic compositions and coating of pipes and the like therewith.** STAVELEY COAL & IRON CO., LTD., and R. FABRY (B.P. 416,967, 9.1.34).

**Ethylene glycol phthalate.**—See III. Reducing  $\eta$  of cellulose nitrate. Rosin size from paper.—See V. Seamless pipes.—See IX. Coatings for rubber.—See XIV.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Variations in the composition of latex from clone and seedling rubber.** J. L. WILTSHIRE (Rubber Res. Inst. Malaya, 1934, Bull. No. 5, 61 pp.).—Information on the variation of the composition of latex is collated and experimental results are recorded on the latex from 4 clones in Selangor which have been under investigation since 1931. Latex from high panels generally has higher  $\eta$  and higher contents of rubber, K, P, and total ash than that from low panels, but the % composition of the ash is practically the same in both cases. One clone gave latex of high rubber content and  $\eta$  and low ash, but the % composition of the ash was similar for all 4 clones. Variations in the latex were observed at different seasons. High latex yield tended to be associated with low rubber content and high ash. The relation between rubber content and  $\eta$  cannot be expressed by any single curve. All methods of raising the rubber content of latex from an area already in tapping necessitate alteration to a less drastic tapping system and, to obtain uniformity, it is important that this should involve no resting periods other than the tapping intervals. Uniformity in rubber content of latex, however, is no proof of uniformity in composition and properties, and large bulking capacity should be provided in order to average out seasonal and other variations as far as possible.

D. F. T.

**Influence of the sharpness of the punch-knife on the results of tensile tests with ring test-pieces of rubber.** D. J. VAN WIJK (Kautschuk, 1934, 10, 151–153).—The degree of sharpness of the knife has a considerable influence on the recorded tensile strength, especially for less highly compounded rubbers; the cutting edge throughout its length should be < 0.01 mm. thick.

D. F. T.

**Technical problems of rubber regeneration.** P. ALEXANDER (Kautschuk, 1934, 10, 133–139, 154–157).—A review.

D. F. T.

**Chatterton [compound]; its origin, physico-chemical nature, and application.** ANON. (Gummi-Ztg., 1934, 48, 984–985, 1009–1010).—The account includes methods of prep. and composition for various purposes.

D. F. T.

**Scientific investigation of rubber.** H. STAUDINGER (Kautschuk, 1934, 10, 157—159).—A brief survey of the relation between physical properties and mol. size and condition, with especial reference to rubber. D. F. T.

**Measurement of quality in rubber goods by physical tests.** A. W. CARPENTER (Ind. Eng. Chem. [Anal.], 1934, 6, 301—308).—The physical tests are classed in two groups, one group measuring fundamental properties, *e.g.*,  $d$  and tensile strength ( $T$ ), and the other determining service val. In the measurement of  $T$  and extensibility with the customary uniform rate of stretching of dumb-bell test-pieces, the rate of elongation of the actual length under observation is seriously influenced by the modulus of the sample. Also two different types of machine for measuring relative indentation hardness will give results at variance with one another, possibly because such hardness is the result of two factors, *viz.*, resistance to resilient and to plastic deformation. Several types of laboratory test are described for evaluating service. The val. of such performance tests is often lost because of incomplete analysis of the factors involved in the test and in the conditions of service. D. F. T.

**Ozone degradation of natural and sodium-butadiene rubber.** R. PUMMERER (Kautschuk, 1934, 10, 149—151).—Examination of the course of action of  $O_3$  on a solution of natural rubber in  $CHCl_3$  by titration of portions of the solution with Br shows that a primary ozonide (I) is first formed which reacts with Br; after several hr. (I) changes into a Br-stable isoozonide (II). Methylglyoxal can definitely be detected to the extent of approx. 1% in the hydrolytic products of (II) by means of semicarbazide. The formation of (II) from the reactive (I) of Na-butadiene rubber (III) occurs still more slowly. The fission products of (I) are more complex than those derived from natural rubber or from (III) polymerised by heat; this is probably because polymerisation has occurred both at the 1:2- and the 1:4-position, possibly even in the same rubber mol. This more complex structure presumably is accountable for the more involved physical and chemical behaviour of (III). D. F. T.

#### PATENTS.

**Treatment of [rubber] latex.** R. L. SIBLEY, Assr. to RUBBER SERVICE LABS. CO. (U.S.P. 1,946,003, 6.2.34. Appl., 23.3.33).—The wetting and penetrating properties of latex are increased by incorporating a small proportion of a sol. salt ( $Na, NH_4$ ) of a  $H_2SO_4$  derivative of the reaction product of an alcohol and a OH-substituted diaryl, *e.g.*, of a BuOH and  $C_6H_4Ph\cdot OH$ , the latter being used preferably in the form a mixture of the *o*- and *p*-isomerides (85:15).

D. F. T.

**Manufacture of materials of or provided with compositions of or containing rubber.** INTERNAT. LATEX PROCESSES, LTD., E. W. MADGE, and F. J. PAYNE (B.P. 416,077, 9.3.33).—An aq. dispersion of rubber etc. is introduced into one or more nips, formed, *e.g.*, by positioning one or more rotating rollers ( $R$ ) or two endless belts, and is coagulated between the moving surfaces, conveniently by a film of coagulant thereon. Concurrently, a length of fabric, or  $\leq 2$  lengths at a

desired angle, may be introduced between  $R$ . The formed material issues from the other side of the nip.

D. F. T.]

**Manufacture of rubber thread.** R. F. MCKAY. From INTERNAT. LATEX PROCESSES, LTD. (B.P. 416,144, 30.5.33).—A rubber-containing fluid, particularly an aq. dispersion, is applied to a travelling edge-surface, *e.g.*, of a rotating disc, so as to form thereon a solid rubber deposit which is then removed as a continuous length; if the strip so formed is U-shaped in cross-section it may be compacted to a substantially solid thread before vulcanisation.

D. F. T.]

**Manufacture of goods of rubber or similar material.** INTERNAT. LATEX PROCESSES, LTD., D. F. TWISS, and W. MCCOWAN (B.P. 416,499, 16.3.33).—Rubber articles of cellular structure are produced by freezing an aq. dispersion of rubber and subsequently vulcanising the solidified product under such conditions that evaporation of the liquid is prevented. Crushed ice or snow and also fragments of other easily liquefiable solids (gelatin) or of collapsible solids (silicic acid jelly) may be added to the dispersions before solidification, as also may a proportion of  $H_2O$ -sol. coagulant insufficient to effect coagulation either immediately or at room temp.

D. F. T.]

**Production of goods of or containing rubber.** INTERNAT. LATEX PROCESSES, LTD. (B.P. 416,904, 17.4.34. U.S., 21.6.33).—Perforate rubber films or sheets are produced by applying a rubber composition, particularly a latex composition, to a perforated deposition backing or form having holes as desired through which a gas is forced while the composition is undergoing partial or complete coagulation. Rubber articles may thus be produced with numerous perforations so minute as to be scarcely visible to the unaided eye.

D. F. T.]

**Production of soft rubber and soft rubber goods.** HANSEATISCHE MÜHLENWERKE A.-G. (B.P. 416,340, 21.2.34. Addn. to B.P. 389,480; B., 1933, 437).—The incorporation of 1—20% of phosphatides, together with active filling materials (C black,  $PbO$ ,  $ZnO$ ), if desired, instead of the earlier smaller % of lecithin, accelerates vulcanisation and has other beneficial effects.

D. F. T.]

**Non-permeable coatings [for rubber].** DUNLOP RUBBER CO., LTD., A. E. T. NEALE, E. W. B. OWEN, J. A. WILSON, and D. F. TWISS (B.P. 416,679, 21.3. and 4.5.33).—The gas-retaining qualities of heat-vulcanised rubber are improved by applying a composition comprising a synthetic resin ( $R$ ) of the polyhydric alcohol-polybasic acid type diluted with glycerol (which may be at least partly polymerised) or glycol, and gelatin. The additional polyhydroxy-compound may be introduced before formation of the  $R$ ; an adhesive may be incorporated to increase the tenacity of attachment to the rubber surface.

D. F. T.]

**Production of chlorinated rubber.** CHEM. FABR. BUCKAU (B.P. 416,252, 23.1.34. Ger., 20.2.33).—The degree of polymerisation of the product is influenced by irradiating the solution, preferably with ultra-violet light, during and/or after the chlorination process. [Stat. ref.]

D. F. T.]

**Production of solid chlorinated rubber products.**

J. P. BAXTER, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 416,056, 7.3.33).—A solution of chlorinated rubber (*C*) is emulsified in a liquid medium, such as  $H_2O$ , which is immiscible with the solvent (*S*) and is a non-solvent for the *C*, and the emulsion is heated to expel *S*. By agitating the mixture during the injection of the solution into the hot  $H_2O$ , a product can be obtained consisting of individual particles or small aggregates which can be easily dried. By heating the emulsion until *S* has evaporated, conveniently in suitable moulds and without the presence of plasticisers, blocks of cellular structure are produced which when dry have  $d$  0.06—0.13 and are suitable for thermal insulation.

D. F. T.

**Colouring of rubber surfaces.**

DUNLOP RUBBER CO., LTD., D. F. TWISS, F. A. JONES, and D. J. HADLEY (B.P. 416,154, 16.6.33).—The surfaces of articles of rubber, gutta-percha, etc. are stained with org. colouring matters after being treated with one or more halogens.

D. F. T.

**Treatment of rubber [for retardation of deterioration].**

W. P. TER HORST, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,945,576—8, 6.2.34. Appl., 10.12.32).—A preservative substance to be incorporated in rubber is prepared from the condensation product of an aliphatic ketone ( $CO_2Me_2$ ) and a *sec.*-arylamine ( $NHPh_2$ ) by conversion into (A) a  $HCO_2H$  derivative, (B) a  $NO$ -compound, or (C) a  $C_{10}H_7\cdot OH$  (preferably  $\beta$ ) reaction product.

D. F. T.

**Age-resisting vulcanised rubber product.**

R. L. SIBLEY, Assr. to RUBBER SERVICE LABS. CO. (U.S.P. 1,946,002, 6.2.34. Appl., 15.4.31).—Rubber is vulcanised in presence of an antioxidant obtained by condensing a primary arylamine ( $NH_2Ph$ , 2 mols.) with a reaction product of a  $C_{10}H_7\cdot OH$  (2 mols.), e.g.,  $\beta$ - $C_{10}H_7\cdot OH$ , and an aldehyde  $>C_3$  and  $<C_8$  (1 mol.), such as  $PrCHO$ , crotonaldehyde, or  $PhCHO$ .

D. F. T.

**Manufacture of rubber having age-resisting properties.**

E. I. DU PONT DE NEMOURS & CO. (B.P. 416,079, 9.3.33. U.S., 12.3.32).—The use as non-staining anti-oxidants of ( $Zn$ ,  $Na$ ,  $K$ ,  $NH_4$ , org. base) salts of *o*-diphenol [ $o$ - $C_6H_4(OH)_2$ , 1 : 3 : 4- $C_6H_3Me(OH)_2$ , 2 : 3- $C_{10}H_6(OH)_2$ ]- $H_3BO_3$  compounds is claimed.

H. A. P.

**Preservation of rubber.**

GOODYEAR TIRE & RUBBER CO. (B.P. 416,410 and 416,675, [A] 14.12.32, [B] 18.3.33. U.S., [A] 16.3.32, [B] 23.3.32).—(A) A *sec.*-mono-amine with  $\leq 2$  substituents consisting of 2 distinct, but directly connected, aromatic ring-structures, e.g., bisdiphenylamine, is an effective antioxidant without influence on vulcanisation. (B) Rubber is treated with a diamine having  $\leq 2$  substituents (*S*), each consisting of two distinct, but directly connected, aromatic ring-structures, *S* being directly attached to the *N* atoms; e.g., *NN'*-bis(diphenylene)benzidine or bis(dinaphthylene)phenylenediamine are suitable compounds. D. F. T.

Moulded blocks for roads etc.—See IX.

**XV.—LEATHER; GLUE.**

**Histological structure of rabbit skins and the changes produced by dressing.** A. SUBIN (Collegium, 1934, 437—450).—The thickness of the epidermis,

muscular and reticular layers, and the corium has been determined in the raw skin and during the various stages of dressing and for the different parts of the skin, respectively. D. W.

**Chemistry of the preparation of cowhide for tanning.** C. SCHIAPARELLI (Boll. Uff. Staz. Sperim. Ind. Pelli, 1934, 12, 217—270, 281—318, 329—374).—The knowledge acquired on this subject during the past decade is summarised and discussed. T. H. P.

**Bark of the Sardinian cork tree and its use in the leather industry.** F. BALDRACCO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1934, 12, 319—325).—Sardinian cork from young and old trees contained, respectively: tanning substances 10.71, 14.24; sol. non-tans 6.13, 7.26; insol. non-tans 72.01, 66.90;  $H_2O$  10.35, 11.6%. The tannin is readily extractable at 90° in Procter's extractor, and small-scale tests on sheepskin and sole leather give promising results. T. H. P.

**Determination of the  $p_H$  values of [vegetable] tanning extracts and liquors.** Report No. 4 of the British Section Committee [of the Society of Leather Trades Chemists]. D. BURTON (J. Soc. Leather Trades Chem., 1934, 18, 525—532; cf. B., 1933, 319).—The superiority of the glass electrode over the  $H_2$  and quinhydrone electrodes for such determinations has been confirmed. The max. degree of accuracy is about 0.02  $p_H$ . No evidence was obtained of any  $Na^+$  effect on the glass electrode in vegetable tan liquors, but the readings were affected by  $Na$  salts at the concns. present in chrome tanning liquors. D. W.

**Rapid tannage by the use of a formaldehyde pretannage.** M. L. HOUBEN (J. Soc. Leather Trades Chem., 1934, 18, 509—511).—Limed pelt can be treated with (a) 2%  $CH_2O$ , then delimed with  $NH_4Cl$ , and subsequently tanned with vegetable tanning extracts; or (b) a mixture of  $H_3BO_3$  (0.5%),  $PhOH$ , and  $CH_2O$  for 24 hr. and subsequently tanned with conc. extract liquors ( $d$  1.063—1.070) for 4 days. Leathers obtained by both methods have a good grain, but are light-weighting. D. W.

**Action of iron compounds on vegetable-tanned leather.** N. BERGMANN, A. MIEKELEY, and N. JAMBOR (Collegium, 1934, 451—454).—Vegetable-tanned leather (*L*) is strongly attacked by aq.  $FeSO_4$  but not to a greater extent than it is attacked by aq.  $H_2SO_4$  of the same  $p_H$ . The effect is hydrolytic and not due to oxidation, so that it is caused by the acid and not by the  $Fe$ . The elasticity of *L* was unaffected by aq.  $Fe(OAc)_2$  (10%) and slightly increased by aq.  $Fe(OAc)_2$  (15%), but the tensile strength of *L* was diminished by these solutions. Aq.  $AcOH$  of the above concns. had no effect on *L*. The presence of  $Fe$  in a damaged *L* is not necessarily the cause of the damage; the effect of acids present in *L* must be taken into account. D. W.

**Neutralisation of chrome[-tanned] leather.** D. McCANDLISH, W. R. ATKIN, and R. POULTER (J. Soc. Leather Trades Chem., 1934, 18, 516—525).—The  $p_H$  of 0.1M- $NaHCO_3$  is  $<$  that of 0.1M- $Na_2B_4O_7$ . Less  $NaHCO_3$  (I) is required to neutralise chrome leather than  $Na_2B_4O_7$  (II), and it is cheaper, so that neutralisation with (I) involves less risk of over-neutralisation. The

$p_H$  and quality of the finished leather were unaltered by the use of (I) instead of (II). D. W.

## PATENTS.

**Production of material for treatment [bating] of hides and skins.** E. LENK and F. LIPNER, Assr. to AMER. CYANAMID & CHEM. CORP. (U.S.P. 1,946,218, 6.2.34. Appl., 1.10.30. Austr., 30.6.28).—Hides and skins are treated with a pure culture of *B. coli communis* and *Staph. pyogenes albus* mixed with a neutral or acid org. salt. D. W.

**Manufacture of tanning substances.** J. R. GEIGY A.-G. (B.P. 416,191, 29.9.33. Ger., 29.9.32. Addn. to B.P. 375,160; B., 1932, 951).—An unsulphonated dihydroxydiarylsulphone is condensed with  $CH_2O$  in an alkaline medium and the product (after alkylation, if desired) further condensed with an arylsulphonic acid in acid solution. The products are in many cases faster to light than are those of the prior patent. Examples include the condensation of 4:4'-dihydroxy-*m*-tolylsulphone with aq.  $CH_2O$  and NaOH at 50°, and treatment of the 5- $CH_2-OH$  derivative produced with aq.  $C_{10}H_7SO_3H$  (I) at 106°; and the condensation of 4:4'-dihydroxy-5-hydroxymethyldiphenylsulphone with sulphonic acids of  $(-CH_2-OPh)_2$ ,  $HO-C_2H_4-OPh$ ,  $PhOMe$ , *o*-chlorophenyl  $\alpha$ -glyceryl ether, and, after alkylation with  $HO-CH_2-CH(OH)-CH_2Cl$ , with (I). H. A. P.

**Dyes for leather.**—See IV. **Dyeing leather.**—See VI. **Resinous compositions [as adhesives].**—See XIII.

## XVI.—AGRICULTURE.

**Reliability of mechanical analyses of soils.** R. LOEBE and R. KÖHLER (Mitt. Lab. preuss. geol. Landesanst., 1933, Nos. 18—48; Chem. Zentr., 1934, i, 2028).—Various methods are compared, and that of Kopecky is preferred. Widest variations occur in vals. for the fraction  $< 10 \mu$ . Prolonged heating with  $H_2O$  forms the most suitable pretreatment. Aq.  $NH_3$  produces satisfactory dispersion. A. G. P.

**Determination of nitrogen in soils. IV. Pretreatment with oxidising agents and its influence on the progress of acid digestion.** A. SREENIVASAN (Indian J. Agric. Sci., 1934, 4, 546—553; cf. B., 1934, 851).—Pretreatment with 1:1  $H_2SO_4$  followed by successive small additions of  $BaO_2$  facilitates the subsequent (Kjeldahl) digestion. HCl gives even better results. A. G. P.

**Comparison of methods for determining available phosphorus in alkaline calcareous soils.** R. D. HOCKENSMITH, R. GARDNER, and J. GOODWIN (Colorado Agric. Exp. Sta. Tech. Bull., 1933, No. 2, 24 pp.).—Results agreeing most satisfactorily with field trials were obtained by extraction with 1%  $K_2CO_3$  solution. In the Mo-blue method for determining P the acidity of the solution must be carefully adjusted. A. G. P.

**Winogradsky's spontaneous culture method for determining certain soil deficiencies.** A. W. YOUNG (Iowa Agric. Exp. Sta. Res. Bull., 1933, No. 157, 24 pp.).—The method was unsuitable for evaluating the P status of soils examined. A. G. P.

**Influence of  $p_H$  of citrate solutions on determination of assimilable phosphoric acid in fertilisers.** G. S. BLJACHER and M. L. TSCHEPPELEVETSKI (Zavod. Lab., 1934, 3, 593—597).—The solubility of superphosphates and of  $Ca_3(PO_4)_2$  in citrate solutions varies inversely with the  $p_H$  of the latter, whilst that of  $CaHPO_4$  and  $FePO_4$  is independent of  $p_H$ . The  $p_H$  of aq. extracts of fertilisers varies inversely with their sol. phosphate content. R. T.

**Proposed modified procedure for [determining] soil potash.** W. S. MARTIN and G. GRIFFITH (Chem. & Ind., 1934, 830).—K in the ignited residue from the usual soil extract is taken up in dil. HCl. Fe and Al are pptd. from the hot solution by addition of 0.1N-NaOH till bromothymol-blue is turned faintly blue. K is determined in the filtrate after concn. A. G. P.

**Determination of chlorides in soil.** J. FIALKOV and G. GALPERINA (Mem. Inst. Chem. All-Ukrain. Acad. Sci., 1934, 1, 71—79).—The aq. extract of 100—200 g. of soil is conc. to 50 c.c., made alkaline with NaOH, and heated at 100° with  $KMnO_4$  for 5 min., after which excess of  $KMnO_4$  is removed by adding  $H_2SO_4$  and  $H_2C_2O_4$ . The colourless solution is then titrated with 0.01N- $AgNO_3$  (NHPh<sub>2</sub>-blue indicator). Soils rich in humus should first be heated at 300—350°. R. T.

**Principles governing the reclamation of alkali soils.** W. P. KELLEY and S. M. BROWN (Hilgardia, 1934, 8, No. 5, 149—177).—Chemical factors concerned in reclamation by use of S,  $CaSO_4$ , and leaching are considered. The determination of exchangeable Ca and Mg in alkali soils presents serious difficulty, and a method for determining these bases collectively is described. A. G. P.

**Measurement of the electrical constants of soil by a Lecher-wire method at a wave-length of 1.5 m.** R. L. SMITH-ROSE and J. S. MCPETRIE (Proc. Physical Soc., 1934, 46, 649—658).—The dielectric const. and conductivity of typical samples of soil of varying moisture content have been measured by this method. For normal moisture content the vals. are 10—12 and  $(10-28) \times 10^8$  e.s.u., respectively. J. W. S.

**Soils in relation to fruit growing in New York. II. Significance of oxidation-reduction potentials in evaluating soils for orchard purposes.** R. BRADFIELD, L. P. BATJER, and J. OSKAMP (Cornell Univ. Agric. Exp. Sta. Bull., 1934, No. 592, 27 pp.).—Inferior yields of orchards, associated with poor subsoil drainage, are probably the result of depleted  $O_2$  supply. The  $E_h$  of soils was min. in early spring and max. in late summer. There is a considerable time lag between the lowering of the ground- $H_2O$  level and the establishment of max.  $E_h$ . Observations of  $E_h$  (preferably in 0.1N- $H_2SO_4$ ) may be utilised as an index of soil-drainage conditions and of the suitability of an orchard site. A. G. P.

**Availability of phosphatic fertilisers.** R. P. BARTHOLOMEW (Arkansas Agric. Exp. Sta. Bull., 1933, No. 289, 19 pp.).—Plants assimilated P from superphosphate (I) more readily than from  $Ca(H_2PO_4)_2$ , but less readily than from  $CaHPO_4$  or  $Ca_3(PO_4)_2$  in acid

soils. Use of liming materials in conjunction with sol. P fertilisers on acid soils did not appreciably affect availability. Ammoniation of (I) up to 6.3%  $\text{NH}_3$  did not lower its availability. The efficiency of rock phosphates was somewhat increased by supplements of  $\text{MgSO}_4$ . A. G. P.

**Relation of soil fertilisation with superphosphates and rock phosphate to fluorine content of plants and drainage waters.** E. B. HART, P. H. PHILLIPS, and G. BOHSTEDT (*Amer. J. Publ. Health*, 1934, **24**, 936—940).—Plant materials from plots fertilised with F-containing phosphates for 16—36 years showed no consistent increase in F content. Drainage  $\text{H}_2\text{O}$  from the single area examined showed high F content, one sample having 1.7 mg./litre. L. D. G.

**Official method of analysis of fertilisers.** ANON. (*Ann. Falsif.*, 1934, **27**, 392—426).—Official methods are described for the microscopical and chemical examination of fertilisers, replacing those issued in 1897. E. C. S.

**Wheat-manuring experiments in the South Island.** A. W. HUDSON and J. W. WOODCOCK (*New Zealand J. Agric.*, 1934, **48**, 321—330).—Comparative trials of various N and P fertilisers are recorded. A. G. P.

**After-effects on wheat of nitrogenous mineral fertilisers applied to beet.** M. LEMOIGNE and H. DUPIC (*Compt. rend. Acad. Agric. France*, 1934, **20**, 448—453).—On certain acid soils examined sufficient of the N applied to the preceding sugar-beet crop remained to satisfy the requirements of wheat. On more alkaline soils in which ammonification and nitrification are more rapid, the residual N is small. A. G. P.

**Development of spring cereals.** VINCENT, HERVIAUX, and SARAZIN (*Ann. Agron.*, 1934, **4**, 395—411).—Analytical data record the amount and distribution of mineral and N constituents in barley at varying stages of growth and the influence of fertilisers thereon. A. G. P.

**Rice nutrition.** L. C. KAPP (*Arkansas Agric. Exp. Sta. Bull.*, 1934, No. 302, 32 pp.).—Rice yields were influenced mainly by the supply of N in soil. The effect of N fertilisers under varying conditions and the influence of org. matter on N availability are examined. A. G. P.

**Effect of boron on plant growth.** K. SCHARRER and W. SCHROPP (*Landw. Jahrb.*, 1934, **79**, 977—999).—Addition of small amounts of  $\text{H}_3\text{BO}_3$  to complete fertilisers increased the yield and sugar content of sugar beet, and also the yield and starch content of potatoes on a no. of the acid soils examined. The decline in yield of both crops and the severity of attack of crown- and dry-rots of turnip induced by heavy liming were to a large extent corr. by application of  $\text{H}_3\text{BO}_3$ . A. G. P.

**Variations in the nitrogen, phosphorus, and potassium nutrition in a single species of plants in the same soil without the influence of fertilisers.** H. LAGATU and L. MAUME (*Compt. rend. Acad. Agric. France*, 1934, **20**, 443—448).—Vines grown in tilled soil assimilated more N, P, and K than those in undisturbed soil. The total amounts and relative proportions of mineral nutrients available to plants in any one soil are largely influenced by physical and climatic conditions. A. G. P.

**Effect of annual applications of the same fertiliser on the nitrogen, phosphorus, and potassium nutrition of a particular plant species during four successive years of growth in the same soil.** H. LAGATU and L. MAUME (*Compt. rend. Acad. Agric. France*, 1934, **20**, 549—563).—Applications of N fertilisers did not increase the intensity of total intake of N, P, and K, but definitely increased the ratio N/P and especially N/K. Fertiliser treatment tends to disturb the proportional intake of naturally occurring soil nutrients. A. G. P.

**Effect of green manuring.** E. A. MITSCHERLICH, W. SAUERLANDT, and A. KUHNKE (*Landw. Jahrb.*, 1934, **79**, 941—975).—The N content of green-manured soils one month after ploughing-in was  $\gt$  that of unmanured controls. The action of green lupins was  $\gt$  that of stubbles remaining from a harvested crop. Cattle manure had a more prolonged effect than green manures. A. G. P.

**Straw manuring.** FLIEG and GROSS (*Z. Pflanz. Düng.*, 1934, **B**, **13**, 380—384).—Reduction in crop yields resulting from ploughing-in straw may be corr. by additions of 0.5% of inorg. N. Heaviest crops were obtained when part of this supplementary N was applied late in the season (May). A. G. P.

**Influence of fallowing, green- and straw-manuring on the nitrogen supply in soil and its productivity.** GERLACH (*Z. Pflanz. Düng.*, 1934, **B**, **13**, 360—379).—Field trials are recorded in which the val. of systems of fallowing and straw-dressing, with and without the use of N fertilisers, and of green-manuring with lupins, are compared. A. G. P.

**Influence of cultivated plants on nitrogen fixation in soils.** O. LEMMERMANN and R. THEMLITZ (*Z. Pflanz. Düng.*, 1934, **B**, **13**, 353—356).—N fixation in soils following growth of mustard was somewhat  $\gt$  that following a no. of other crops. A. G. P.

**Effect of ammonium sulphate on the exchangeable-base status of a pasture soil.** H. O. ASKEW (*New Zealand J. Sci. Tech.*, 1934, **16**, 19—27).—Heavy applications of  $(\text{NH}_4)_2\text{SO}_4$  (I) increased the acidity of surface soils and lowered the degree of saturation with bases. The proportion of exchangeable Ca declined, but that of K increased. The total base capacity was unchanged. Losses of available  $\text{CaO} \propto$  the amounts of (I) applied, but were always  $< 70\%$  of the theoretical vals. A. G. P.

**Effect of fertilisers on composition of grasses.** A. J. TAYLOR (*J. S. African Chem. Inst.*, 1934, **17**, 48—53).—Fertilisers increased the mineral content of pastures to an extent which assisted materially in remedying the mineral deficiency of sour veldt grasses. A. G. P.

**Mineral content and feeding value of natural pastures in the Union of South Africa. II.** P. J. DU TOIT, A. I. MALAN, J. G. LOUW, C. R. HOLZAPFEL, and G. ROETS (*Onderstepoort J. Vet. Sci.*, 1934, **2**, 607—643; cf. B., 1933, 599).—Protein, P, Cl, Na, and K in 11 species of grasses diminished rapidly as growth advanced; the fall was less noticeable for Mg and Ca. NUTR. ABS.

**Weeds and [soil] acidity.** O. DE VRIES (*Z. Pflanz. Düng.*, 1934, **B**, **13**, 356—360).—Weed distribution is examined in relation to soil reaction. A. G. P.

**Influence of certain spray materials, herbicides, and other compounds on the desiccation of plant tissue.** H. A. RUNNELS and J. D. WILSON (Ohio Agric. Exp. Sta. Bimo. Bull., 1934, No. 168, 104—109).—The rate of drying-out of cut stems and leaves of plants was increased by dipping in Bordeaux mixture (I), the effect being greater in more conc. mixtures and showing a max. with a 4–6–50 prep. Neither  $\text{CuSO}_4$  nor  $\text{Ca}(\text{OH})_2$  was as effective as the mixture. Addition of  $\text{CuSO}_4$  to  $\text{Ca}(\text{OH})_2$  produced greater drying-out than did admixture in the reverse order. Addition of customary spreaders and adhesive agents to (I) increased its action in this respect. S sprays had little effect on, and oil emulsions retarded, desiccation.  $\text{CuCl}_2$  was more active than  $\text{CuSO}_4$ . Of common herbicidal substances  $\text{Na}_3\text{AsO}_3$  had the greatest and  $\text{FeSO}_4$  the least effect. A. G. P.

**Influence of Bordeaux mixture and an oil emulsion on the water requirement [of plants].** J. D. WILSON and H. A. RUNNELS (Ohio Agric. Exp. Sta. Bimo. Bull., 1934, No. 166, 21—28).—Bordeaux mixture (I) increased, and 1% oil emulsion decreased, the  $\text{H}_2\text{O}$  requirement of a no. of plants examined, the effects being nullified by use of a combined spray. Plants sprayed with (I) were somewhat smaller, but contained a higher % of dry matter, than oil-sprayed or untreated plants. A. G. P.

**Relative influence of calcium and magnesium in Bordeaux mixture on the transpiration rate [of plants]. I.** J. D. WILSON and H. A. RUNNELS (Ohio Agric. Exp. Sta. Bimo. Bull., 1934, No. 169, 158—163).—The increase in transpiration of potato and coleus plants induced by spraying with Bordeaux mixture varied inversely, though not proportionately, with the Mg:Ca ratio of the limestone used in its prep. When artificial mixtures of  $\text{Ca}(\text{OH})_2$  and  $\text{Mg}(\text{OH})_2$  were utilised the effect of  $\text{Mg}(\text{OH})_2$  became more apparent when the proportion was >50% of the total base. A. G. P.

**Lead and arsenic spray-residue removal from apples.** F. L. OVERLEY, J. L. ST. JOHN, E. L. OVERHULSER, and K. GROVES (Wash. Agric. Exp. Sta. Tech. Bull., 1933, No. 286, 83 pp.).—The ratio of Pb:As in residue after washing varied from 1:1 to 1:10. Effective removal resulted from the use of Na silicate (I) solutions (80 lb. per 100 gals.) containing sawdust or soap. Soda ash or  $\text{Na}_3\text{PO}_4$  (II), used alone or following a HCl wash, satisfactorily removed As but not Pb. (I) is more effective in foaming mixtures. Excessive foaming is reduced by additions of NaCl or paraffin. Use of fish oil in Pb arsenate sprays facilitates the removal of residue by (I) and (II). Fruit grown in light dry soils, or on trees infested with leaf-attacking insects, or injured during winter was less easily cleaned than that grown under more favourable conditions. A. G. P.

**Removal of lead and arsenic spray residues from New York apples.** W. T. PENTZER (Cornell Univ. Agric. Exp. Sta. Bull., 1934, No. 604, 32 pp.).—Washing with 1% HCl was effective unless late oil sprays had been applied, when 1.5% HCl at 25° with the use of wetting agents is necessary. Fruit should be treated immediately after harvesting; contact with acid should be for  $\nabla$  5 min., and washing with  $\text{H}_2\text{O}$  must be thorough to avoid acid and As injury. A. G. P.

**Effect of potassium on the seed yield of *Lolium italicum* (var. *Westerwoldicum*) under different lighting conditions.** R. SCHWARZ (Ernähr. Pflanze, 1934, 30, 293—299).—Heavy dressings of K fertilisers counteract the effect of unfavourable weather conditions, notably lack of sunshine. A. G. P.

**Influence of soil condition on early growth of [sugar] beet.** V. STEHLÍK (Z. Zuckerind. Czechoslov., 1934, 58, 437—444, 445—452, 453—455).—A general discussion on the influence of the physical condition of the soil on beet development. A correct proportion of air and  $\text{H}_2\text{O}$  is especially important, but the  $p_{\text{H}}$  and Ca content of the soil likewise exert a considerable effect, not only on growth, but on the incidence of disease. Seed variety is demonstrated by figures also to be a factor. J. P. O.

**Field experiments with sugar cane. III.** C. H. B. WILLIAMS, C. CAMERON, and R. R. FOLLETT-SMITH (Brit. Guiana Dept. Agric. Sugar Bull., 1934, No. 3, 144 pp.; cf. B., 1933, 884).—Effects of  $(\text{NH}_4)_2\text{SO}_4$ ,  $\text{NaNO}_3$ , and CaO on sugar yields are examined. A. G. P.

**Control of foot rot in wheat.** G. FRON (Compt. rend. Acad. Agric. France, 1934, 20, 644—650).—Use of 8-hydroxyquinoline sulphate (I) as a seed dip is suggested. Spores do not germinate in solutions containing 1 p.p.m. of (I). A. G. P.

**Copper sulphide in the control of fungus diseases.** J. DULAC (Compt. rend. Acad. Agric. France, 1934, 20, 650—652).— $\text{CuS}$  possesses fungicidal properties superior to those of Cu-CaO preps. The powdered material adheres well to foliage and causes no injury. Oxidation to  $\text{CuSO}_4$  increases with the temp. and R.H. of the atm. A. G. P.

**Inhibition of the growth of fungi by chemicals.** F. L. MUNRO and W. NEWTON (Sci. Agric., 1934, 14, 560—564).—The ratio of the max. concn. of fungicidal chemicals permitting growth of host plants (wheat) to the min. concn. inhibiting fungal growth was >1 for PhOH, cresol, chinosol, and malachite-green, approx. 1 for Cu salts, KCN,  $\text{Hg}_2\text{SO}_4$ ,  $\text{PbCl}_2$ , and  $\text{K}_2\text{Cr}_2\text{O}_7$ , and <1 for all other substances examined. A. G. P.

**Spraying for codling-moth control.** H. N. WORTHLEY (Penn. Agric. Exp. Sta. Bull., 1933, No. 285, 16 pp.).—In heavy infestations use of Pb arsenate (I) at < 3 lb. per 100 gals. was ineffective. Supplements of  $\text{Ca}(\text{OH})_2$  decreased, and of fish oil (1 quart per 100 gals.) improved, the efficiency of the spray. Neither nicotine tannate nor miscible oil satisfactorily replaced (I). A. G. P.

**Bacteriosis (blight) of the English walnut in California and its control.** B. A. RUDOLPH (Calif. Agric. Exp. Sta. Bull., 1933, No. 564, 88 pp.).—Use of Bordeaux mixture (8–4–50) as a pre- and post-blossom spray was more satisfactory than a no. of other materials examined. A. G. P.

**Protection of orchard and shade trees and ornamental shrubs from injury by Japanese beetle.** W. E. FLEMING, F. W. METZGER, and M. R. OSBURN (U.S. Dept. Agric. Circ., 1934, No. 317, 7 pp.).—Appropriate control measures are described. A. G. P.

**Potato flea beetles, *Epitrix cucumeris*, Harris, and *E. subcristata*, Leconte.** A. J. HANSON (Wash.

Agric. Exp. Sta. Bull., 1933, No. 280, 27 pp.).—The most effective control measure was to apply dusts containing Ca arsenate with CaO (1 : 4) or BaSiF<sub>6</sub> with diatomaceous SiO<sub>2</sub> (1 : 1).  
A. G. P.

**Sheep blowfly. Field tests of baits treated with sodium sulphide.** M. E. FULLER (J. Counc. Sci. Ind. Res. Australia, 1934, 7, 147—149).—The efficiency of baits was increased by Na<sub>2</sub>S (cf. B., 1932, 754).  
A. G. P.

**Use of zinc chloride for soil treatment in the control of termites.** G. F. HILL and F. G. HOLDAWAY (J. Counc. Sci. Ind. Res. Australia, 1934, 7, 169—172).—Application of solutions of ZnCl<sub>2</sub> to give 3½ oz. per sq. ft. of soil and a penetration of 1 in. repelled termites.  
A. G. P.

**Effect of anthracene oil [insecticide] on vegetation.** P. BOISCHOT (Comp. rend. Acad. Agric. France, 1934, 20, 410—413).—Anthracene oil accumulates in the surface (1—2 cm.) layers of soil and is not washed down by H<sub>2</sub>O. Crops grown beneath sprayed fruit trees are seriously injured and germination of seeds is delayed or inhibited even if the oil is incorporated with soil.  
A. G. P.

**Penetration of insecticidal oils into porous solids.** W. M. HOSKINS (Hilgardia, 1933, 8, No. 2, 49—82).—Penetrability (I) of various oils into bark, filter paper, etc. is favoured by low *n* and volatility and a high degree of refinement, but is decreased by presence of moisture. Effects of dissolved substances on (I) are examined.  
A. G. P.

**Sulphuric acid as a penetrating agent in arsenical sprays for weed control.** A. S. CRAFTS (Hilgardia, 1933, 8, No. 4, 125—147).—The killing action of acid As sprays is directly related to the [H<sub>2</sub>SO<sub>4</sub>] used. For practical purposes the optimum proportion of H<sub>2</sub>SO<sub>4</sub> is approx. 1·0*N*. The presence of As eliminates corrosion of Fe equipment.  
A. G. P.

**Vine scorching by arsenic [insecticides].** K. MÜLLER (Weinbau u. Kellerwirts., 1933, 12, 213—215; Chem. Zentr., 1934, i, 1866).—Schweinfurt-green preps. containing excessive amounts of Ca(OH)<sub>2</sub> may cause scorching as a result of the formation of Ca(HCO<sub>3</sub>)<sub>2</sub> and subsequent dissolution of As. Also, in presence of CO<sub>2</sub>, Cu arsenite is partly transformed into basic Cu carbonate and some free As<sub>2</sub>O<sub>3</sub>. The fatty layer of the vine leaf is attacked by Ca(OH)<sub>2</sub> and penetration of As is facilitated.  
A. G. P.

**Toxicity of arsenical insecticides.** A. CHAPPELLER and M. RAUCOURT (Compt. rend. Acad. Agric. France, 1934, 20, 598—603).—Toxicity to several types of animals of common arsenicals was in the order Paris-green > Ca<sub>3</sub>(AsO<sub>4</sub>)<sub>2</sub>, AlAsO<sub>4</sub> > PbHASO<sub>4</sub>.  
A. G. P.

**Superphosphate. Determining P<sub>2</sub>O<sub>5</sub> in technical H<sub>3</sub>PO<sub>4</sub> etc.**—See VII.

See also A., Oct., 1066, Solubility of the H<sub>3</sub>PO<sub>4</sub> of different phosphates.

#### PATENTS.

**NH<sub>3</sub> [as fertiliser] from gases.**—See II. BaSiF<sub>6</sub>.—See VII.

### XVII.—SUGARS; STARCHES; GUMS.

**Working quality of different varieties of sugar beets.** II. O. SPENGLER, S. BÖTTGER, and G. LINDNER

(Z. Ver. deut. Zucker-Ind., 1934, 84, 365—399).—A similar investigation to that carried out last season (B., 1934, 116) and leading to similar conclusions. Rate of filtration after the first carbonation and colour of purified juice depend far more on methods of purification than on the type of beets worked. The converse applies to the contents of ash and melassigenic N (I). Raw juice contains about 44—54% and purified juice about 34—44% of the total ash in sliced beets; this total ash is a pronounced varietal character, but strongly influenced by soil conditions. More valuable as a criterion of working quality is the content of (I) in beets, which is a more const. varietal character than the ash content; 97% of the (I) in beets is found in the juice, even after purification.  
J. H. L.

**Ash constituents of the cane and raw juice.** P. HONIG (Arch. Suikerind. Nederl.-Indië, 1934, II, Meded. No. 10, 435—524).—Figures are given for the extreme variations which may be observed for the K<sub>2</sub>O, CaO, MgO, and P<sub>2</sub>O<sub>5</sub> present in the plant and in the juice. It is emphasised that these figures vary much under climatic conditions from year to year, besides depending on the cane variety and the location of the estate.  
J. P. O.

**Defecation method of clarification [of sugar juice].** J. F. BOGTSTRA (Arch. Suikerind. Nederl.-Indië, 1934, II, Meded. No. 15, 587—596).—Factory experiments were conducted comparatively with (A) hot liming and (B) cold saccharate liming, using the same amount of Ca throughout. B was found to have the more thorough clarifying effect, removing the wax much more completely than A, and it also gave the clearer clarified juice. It, however, produced 21% of mud as compared with 12·8% obtained in B, and a slower rate of filtration. In practice this would mean a filter-press area 3—4 times > at present is used in hot-liming.  
J. P. O.

**Determination of the polarisation of very small quantities of [sugar] juice.** K. D. DEKKER and H. J. MONSANTO (Arch. Suikerind. Nederl.-Indië, 1934, II, Meded. No. 12, 531—537).—Satisfactory results for the polarisation and purity of cane juice for indicating its ripeness can be obtained by clarifying 2 c.c. of the juice with 30 mg. of dry basic Pb acetate, filtering, and polarising in a 50-mm. tube the internal diam. of which has been reduced to 3 mm., the *n* of the unclarified juice being taken at the same time.  
J. P. O.

**Apparatus for the continuous titration of alkaline sugar juices of beet-sugar factories.** L. P. TOGO (L'Ind. Chimica, 1934, 9, 1170—1172).—Apparatus for the rapid titration of samples of sugar juices with H<sub>2</sub>SO<sub>4</sub> so that a definite CaO content may be maintained is described.  
T. H. P.

**Sulphitation process [for raw sugar juice].** J. F. BOGTSTRA (Arch. Suikerind. Nederl.-Indië, 1934, II, Meded. No. 15, 597—608).—A comparison was made between (A) heavily sulphuring the raw juice before liming, and (B) simultaneous sulphuring and liming, the same amount of CaO being used in both processes. In general, little difference was observed in the results provided conditions were adjusted so as to restrict glucose decomp. to the min. possible. In A a good result was obtained

by sulphuring to  $p_H$  4.3—3.7, liming to raise the alkalinity to 8.0, and finally sulphuring to  $p_H$  7.0. In *B* a better result was obtained with the juice at 58° than at 77°; it should not rise above 70°. A good circulation in the tanks was important. J. P. O.

**Washing or affining pre-dried white sugars.** J. F. BOGTSTRA (Arch. Suikerind. Nederl.-Indië, 1934, II, Meded. No. 8, 265—278).—Washing is best done with a saturated solution of sugar of the highest possible purity. Pugging with evaporator syrup is, therefore, inadvisable. Further, the solution should be saturated, since if unsaturated it will dissolve the crystal; also the temp. should not be raised above that at which the clear liquor leaves the after-dryers. In the case of stale sugars, *i.e.*, those which have been in storage for a rather long time, a very spacious pugging trough is required. A table is given showing the effect of increasing quantities of saturated wash-liquors on colour, ash (by electrical conductivity), and reducing sugars in the cases of two sugars, pugging being continued in both cases for 48 min. J. P. O.

**Heat economy in the [sugar] refinery.** K. ŽERT (Z. Zuckerind. Czechoslov., 1934, 58, 455—459).—Calculations and results are given, based on the author's experience, showing how considerably the steam consumption may vary according to the scheme adopted in the case of the particular product. J. P. O.

**Sucrose from cornstalks.** C. BODEA (Act. Chim. et Ind., 1934, 31, 182—184  $\tau$ ).—Corn stalks contain 10—12% more sucrose (I) at the time the grain begins to ripen, up to 17% if the ear is removed just after silking, but large quantities of glucose (II) (25% of the total sugars) are also present. The author proposes to ferment the (II) with a yeast (*Torula pulcherrima*) which does not attack (I). J. P. O.

**Determination of reducing sugars in glucose (corn syrup).** B. SEGAL (J. S. African Chem. Inst., 1934, 17, 54—57).—Ratios of maltose, dextrin, and glucose in syrup are examined and methods of calculating from the analytical vals. are indicated. A. G. P.

**Action of hot alkaline solutions, with and without copper salts, on sucrose.** O. SPENGLER and F. TÖDT (Z. Ver. deut. Zucker-Ind., 1934, 84, 400—401).—The decomp. of sucrose (I) on heating with dil. alkali, as reported by Herzfeld and by the authors (B., 1930, 259), will not account for 10% of the reducing action of (I) on Fehling's solution (II). Possibly the Cu salts or the high alkali content of (II) increases the decomp. The very high reducing action of (I) on mixtures of (II) with glycol (III) and extra alkali (B., 1934, 694) should not be attributed to the (III). J. H. L.

**Clarification previous to the determination of reducing sugars.** K. D. DEKKER and H. W. GOSLINGS (Arch. Suikerind. Nederl.-Indië, 1934, II, Meded. No. 11, 527—530).—As the result of the examination of 50 samples of molasses sugars, it is recommended that clarification with neutral Pb acetate, as prescribed in the official "Handboek," should be retained, though it can be dispensed with for sugars of a higher quality than molasses sugars. J. P. O.

**Determination of glucose syrup.** R. INTONTI and A. VERCILLO (Annali Chim. Appl., 1934, 24, 327—347).— $H_2O$  in commercial glucose syrup (I) is best determined by adding a little  $H_2O$  to 5—6 g. of (I) and quartz sand in a tared dish, heating on a  $H_2O$ -bath with frequent mixing until most of the  $H_2O$  is expelled, and drying at 80—85° in vac. for 4—5 hr. Glucose and dextrin are determined by means of Fehling's solution. For determining (I) in caramel, use is made of Juckenack and Pasternak's method (Z. Unters. Nahr. u. Genussm., 1904, 2, 19); a new table showing the relation between the  $[\alpha]_D^{20}$  of the inverted aq. extract and the % of anhyd. starch syrup is given. The applicability of the procedure described to the analysis of (I)-containing fruit juices or pulps, EtOH solutions of essential oils, etc. is shown. T. H. P.

**Determination of total reducing sugars, glucose, and fructose in raw cane sugars.** F. W. ZERBAN and M. H. WILEY (Ind. Eng. Chem. [Anal.], 1934, 6, 354—356).—A combination of Lane and Eynon's method (J.S.C.I., 1923, 42, 32  $\tau$ ) (corr. for the presence of sucrose) for total reducing sugars and of Jackson and Mathews' modification (A., 1932, 835) of Nyns' method for fructose is suitable. R. S. C.

**Filtration with kieselguhr.** Höppler viscosimeter.—See I.  $p_H$  of activated carbons.—See II. Sugar cane and beet.—See XVI. Citric fermentation.—See XVIII. Potatoes [for starch manufacture].—See XIX.

See also A., Oct. 1069, Physical chemistry of starch. 1104, Properties of adipoin.

#### PATENTS.

**Fermentation of fructose.**—See XVIII. Molasses feed.—See XIX.

### XVIII.—FERMENTATION INDUSTRIES.

**Influence of the liquor on the composition of the wort and ash of the beer.** H. SIEGFRIED (Schweiz. Brauerei-Rundsch., 1934, 45, 1—5; Chem. Zentr., 1934, i, 2052).—The Mg content of the ash declines with increasing  $Ca(HCO_3)_2$  in the liquor (I), and the total ash decreases with rising proportions of  $Ca(HCO_3)_2$  or  $Mg(HCO_3)_2$  in (I). Ash- $PO_4'''$  falls with increasing  $CaSO_4$  in (I), but ash-Mg and total ash are not appreciably affected. Relations between the  $HCO_3'$  and  $CaSO_4$  contents of (I) and the amount, colour, and acidity of the extract are examined. A. G. P.

**Brewing of consistent draught beers.** W. DUNCAN (J. Inst. Brew., 1934, 40, 368—377).—The importance of a knowledge of  $p_H$  at various stages of the brewing process is shown, and the significance of total and permanently sol. N of malt discussed. Present methods of malt valuation are criticised, and advantage is claimed for a process of re-drying all malts. Liquor treatments and wort-boiling methods are discussed, and recent advances shown in cooling systems and in methods of yeast storage. I. A. P.

**Control of after-fermentation.** G. W. A. BRISCHKE (Woch. Brau., 1934, 51, 297—301).—Apparent extract in beer passing to store-tanks is found hydrometrically, and the result is corr. for the EtOH present to give

the true extract. The observations, together with temp. readings, are repeated at intervals during storage, and the fermented and unfermentable extract calc. at the end of each control interval, as also is the % of available extract (*A*) fermented during each period. From the figures is calc. for each interval the "after-fermentation activity" (I) of the yeast, defined as amount of *A* fermented per 100 hectolitres per day. A beer with a regular temp. fall gave a satisfactory after-fermentation (II); a slight temp. rise in a second beer accompanied an incomplete fermentation, giving a product of poor quality; and in a third case rapid fall in temp. at the beginning of storage gave unsatisfactory (II), but, after decreasing, (I) later increased. In each case, the approx. heat loss was  $10^4$  kg.-cal. per kg. of extract fermented. I. A. P.

**Oxidation-reduction potential in beer.** F. MENDLIK (Woch. Brau., 1934, 51, 305—307).—In carrying out electrometric measurement (I) of  $r_H$  the danger of polarisation of the electrode is great, whilst traces of air or  $O_2$  introduce considerable errors. The apparatus and process of (I) are described. In general, the results are in good agreement with those obtained by the methylene-blue (II) method. Using a  $N_2$  cell, an  $r_H$  of approx. 17 is usually obtained after some hr., the initially higher val. being due to unavoidable aëration. When it was possible to avoid aëration the  $r_H$  level was lower, and addition of (II) caused a slight rise. A small quantity of  $Na_2S_2O_4$  depressed the  $r_H$ ; when a const.  $r_H$  was reached in the mixture, titration with  $K_3Fe(CN)_6$  showed that between  $r_H$  10 and 18 the beer is very sensitive to oxidation, but above 21 it is more stable. The  $p_H$  of the beer was unchanged during the titration. The views of de Clerck (B., 1934, 777) on the connexion between  $r_H$  and yeast turbidity are confirmed in general. I. A. P.

**Physical and physico-chemical data of wine.** A. AMATI and G. CONFORTO (Atti Congr. naz. Chim., 1933, 4, 614—623; Chem. Zentr., 1934, i, 2052).—The f.p. of wine is controlled by the sum of the EtOH, dry matter, and acid contents, EtOH having the greatest influence. The b.p. is a function of the EtOH content. The electrical conductivity is exclusively influenced by the ash constituents. A. G. P.

**Copper in musts and wines.** L. BENVENIGNI and E. CAPT (Mitt. Lebensm. Hyg., 1934, 25, 125—138).—The Cu content (I) of completely fermented wines is extremely small and independent of that (derived from sprays) present in the grapes at the time of pressing or in the must, owing partly to retention in the marc, although 90% of the remainder is eliminated in the lees during fermentation. During cellar-storage from December to June, (I) fell from 3 to 0.5 mg. per litre. (I) may be higher in cases of defective fermentation or contamination by plant, and for commercial, non-alcoholic, raisin juices (e.g., > 10 mg. per litre). Methods of determination of (I) are discussed. J. G.

**Determination of benzoic, salicylic, and cinnamic acids, saccharin, and hydroxybenzoic esters in wine.** R. FISCHER and F. STAUDER (Ind. Ital. Conserve aliment., 1933, 8, 292—301; Chem. Zentr., 1934, i, 1899).—The sample (10—20 c.c.) is made alkaline, conc.

to  $\frac{1}{3}$  vol., acidified (HCl), and extracted with a (1:1) mixture of  $Et_2O$  and petroleum. Extracts are dried ( $CaCl_2$ ), conc., and re-extracted as before. The evaporated extract is sublimed and examined microscopically. A. G. P.

**Determination of isopropyl alcohol in alcoholic beverages.** E. M. ALESSANDRINI (Atti Congr. naz. Chim., 1933, 4, 611—614; Chem. Zentr., 1934, i, 2052—2053).—The sample is distilled and the distillate (I) tested for  $Pr^sOH$  by oxidation with  $CrO_3$  and application of the nitroprusside test for  $COMe_2$ . Quantitatively, the  $COMe_2$  obtained by oxidation is distilled into  $NH_2OH.HCl$  and the free HCl determined volumetrically.  $C_5H_5N$ , if present, is removed from (I) by distilling with  $MgO$  and treating the distillate with  $CdCl_2$  in EtOH. The filtered liquid is then oxidised as above. A. G. P.

**Determination of the adsorption capacity of carbon used for the treatment of wine.** T. VON FELENNBERG (Mitt. Lebensm. Hyg., 1934, 25, 65—81).—The % of solid methylene-blue (I) adsorbed from a 0.1% solution of its hydrochloride when the colour/C ratio is equiv. to the adsorption of half the colour has been determined for various wines and C products. Tannin, NaCl, NaI, vanillin, and  $Bu^sOH$  were also adsorbed normally, but  $H_2S$  was removed in exceptionally large quantities, probably owing to fixation by heavy metals. The "kunstrosé" method (determination of the C required to decolorise a wine after it has been diluted to the colour of a standard  $K_2Cr_2O_7-KMnO_4$  mixture with 1% tartaric acid) is less satisfactory as a testing method, but affords useful information as to the nature of the colouring matters present. C suitable for wine treatment should have (I) adsorption (determined as above)  $\leq 10\%$ , HCl-sol. ash  $< 1\%$ , and should contain no heavy metals and only traces of  $Cl^-$  and  $SO_4^{2-}$ . J. G.

**Dehydration of crude spirits by the "Drawinol" process.** E. LÜHDER (Z. Spiritusind., 1934, 57, 252).—The apparatus and process are described. Using  $C_2HCl_3$  in the prep. of the ternary mixture the process is continuous, and apparatus designed for earlier processes may be employed. It is asserted that sulphite-spirit may be dehydrated without difficulty by this process. I. A. P.

**Distinction of wine vinegar from other types of vinegar.** J. PRITZKER (Mitt. Lebensm. Hyg., 1934, 25, 101—106).—The methods of Patzauer (B., 1933, 985) and Pralongo (B., 1933, 281) are unsatisfactory (cf. B., 1934, 118, 165), and detection of  $CHAcMe.OH$  (I) by conversion into dimethylglyoxime and thence into the Ni salt (cf. B., 1933, 995) is preferred; 5 commercial wine vinegars yielded (per litre) 0.049—0.122 g. of (I) and 2.2—4.4 g. of glycerol. The factor 0.25 gives (I) from the wt. of  $Cu_2O$  obtained on mixing equal vols. of cold Fehling's solution and the distillate from the EtOH determination, and filtering after 18 hr.  $Ac_2$  or (I) is not detectable in authentic and properly fermented spirit vinegars. J. G.

**Citric fermentation.** A. QUILICO and A. DI CAPUA (Annali Chim. Appl., 1934, 24, 355—363).—In the production of citric acid (I) by growing *Aspergillus niger*

on beet molasses solutions containing 15% of sucrose, good yields are obtained only if the phosphate nutrition is controlled within narrow limits (0.1—0.2 g. of  $\text{KH}_2\text{PO}_4$  per litre). The yield of (I) depends on the amounts of P and N and on the nature of the latter, abundant supplies of both nutrients resulting in enormous growth of mycelium and complete combustion of the sugar. T. H. P.

**Filtration with kieselguhr.**—See I. **Flour-amylases.** Diastatic powers of flours.—See XIX.

See also A., Oct., 1136, Carboxylase [in powder form]. Amylase from sweet potato. 1137, Purification of invertase.

#### PATENT.

**Propionic acid fermentation of fructose-containing mashes.** H. R. STILES (U.S.P. 1,946,447, 6.2.34. Appl., 29.6.32).—A mash containing fructose (I) (e.g., containing invert sugar, molasses, etc.) is fermented by the combined action of mannitol-forming bacteria (*M*) and  $\text{EtCO}_2\text{H}$  bacteria (*P*), the order of inoculation with *M* and *P* being immaterial owing to the rapid growth of *M*. In addition, accelerating organisms of the *proteus*, *alcaligines*, or lactic groups may also be used. The carbohydrate mixture used should be capable of yielding at least as much mannitol as could be obtained if 25% of the sol. carbohydrate of the mash consisted of (I). Suitable nutrients may be added, and the  $p_{\text{H}}$  regulated by addition of  $\text{CaCO}_3$ . I. A. P.

#### XIX.—FOODS.

**Carotene content and other quality characters in a series of hybrid wheats.** C. H. GOULDEN, W. F. GEDDES, and A. G. O. WHITESIDE (Cereal Chem., 1934, 11, 557—566).—Statistical studies of carotene (I) determinations, made on the flour and on the whole wheat from a series of 139 miscellaneous and new hybrid strains of wheat, indicated that tests for (I) in the whole wheat might be of considerable val. for predicting the (I) content of the flour in breeding work if applied in the early generations and continued in the progeny for 2 or 3 years. E. A. F.

**Vitality of wheat. II. Influence of moisture in wheat seeds on imbibition and speed of germination.** R. WHYMPER and A. BRADLEY (Cereal Chem., 1934, 11, 546—550; cf. B., 1934, 857).—Reduction of the  $\text{H}_2\text{O}$  content much below 5% by artificial drying in the cold over  $\text{CaCl}_2$ , while considerably stimulating the speed of germination in the case of fresh wheat, is less effective than drying to about 5%  $\text{H}_2\text{O}$  if the wheat is to be stored for some years and still retain its vitality; this is probably not true of wheat stored in the dark. The course taken by the  $\text{H}_2\text{O}$  absorbed was followed by immersing the whole grains in dil. I solution. E. A. F.

**Flour extract as standard in the colorimetric determination of flour colour.** C. G. FERRARI and A. B. CROZE (Cereal Chem., 1934, 11, 511—514).—A flour extract prepared by using 93% of cleaner's naphtha + 7% of abs. EtOH as solvent and stored in a brown, glass-stoppered bottle forms a satisfactory standard for the determination, using a Duboscq colorimeter or, better, Nessler tubes. E. A. F.

**Mercury-vapour lamp for accelerating the bleaching action of benzoyl peroxide [on flour]. II. Quartz electrode.** C. G. FERRARI and A. B. CROZE (Cereal Chem., 1934, 11, 505—510; cf. B., 1932, 1052).—The accelerating effect of radiant energy from a quartz Hg-vapour arc on the bleaching of flours (*F*) by  $\text{Bz}_2\text{O}_2$  is much > that from a Uviol-glass electrode; the former but not the latter bleached untreated *F*. The Pekar test is unsatisfactory for determining the extent of colour removal when the bleaching accelerator is used. E. A. F.

**Amylases of normal and germinated wheat flours.** J. S. ANDREWS and C. H. BAILEY (Cereal Chem., 1934, 11, 551—556).—The diastatic activity (*D*) of sound normal wheat is primarily due to  $\beta$ -amylase (*B*);  $\alpha$ -amylase (*A*) develops during germination. Methods suitable for isolating *A* and *B* are indicated. The max. activity of *A* is exhibited at  $p_{\text{H}}$  5.6—5.8, that of *B* at 4.5—5.1. The increase in rate of maltose (*M*) production effected by adding 1% of germinated wheat flour (*N*) was positively correlated with the *M*-producing ability of the *N*, indicating that *D* is more a function of the condition of the starchy substrate than of the actual amylase content. This is confirmed by the fact that aq. flour extracts possessing widely different diastatic activities gave nearly equal quantities of *M* when acting on a sol. substrate. E. A. F.

**Factors affecting the diastatic activity of wheat flour.** M. C. MARKLEY and C. H. BAILEY (Cereal Chem., 1934, 11, 515—522).—The condition of the surface of the flour (starch) particles as affected by the R.H. during the grinding and bolting of middlings is of greater influence than granulation on diastatic activity (*D*). Other factors which affected *D* were: the method of tempering or conditioning the wheat in experimental milling; environmental factors during growth; varietal factors. A fairly accurate prediction could be made of the relative *D* of flours milled from different wheat varieties from *D* determinations, if sufficient replicates of these varieties were grown under different environmental conditions. E. A. F.

**Correlation between diastatic activity and gassing power in commercial flours.** C. F. DAVIS and D. F. WORLEY (Cereal Chem., 1934, 11, 536—545).—In a random group of commercial flours, the correlation between diastatic activity by Blish's ferricyanide method (B., 1933, 602) (I) and the Blish *et al.* (B., 1932, 860) manometric suspension test (II), using a correction factor for yeast variability, was  $+0.979 \pm 0.0025$ ; between (I) and the total gas-production (*P*) test after Heald (B., 1933, 168) (III) was  $+0.9271 \pm 0.0086$ ; and between (II) and (III) was  $+0.9327 \pm 0.0087$ . With sound flours of high or low non-reducing sugar content (II) is probably a better measure of *P* than is (I). (II) possesses the advantage over (III) that the reading can be taken at the end of 1 hr. and that the method can be modified to indicate the original sugar content of the flour. (III) possesses the disadvantage of the length of time required to complete the test; its advantage lies in the fact that short-interval readings (an automatic recorder may be used) supply information as to the *P* and the time at which the "crit. point" is

reached, as well as the effect on *P* of various dough-batch ingredients. Yeast variability can be decreased by (III) and the accuracy of the method is high.

E. A. F.

**Diastatic activity in [flour] suspensions and doughs.** R. M. SANDSTEDT (Cereal Chem., 1934, 11, 532—535; cf. B., 1934, 472).—Correlation coeffs. for collaborative diastatic activity (*A*) determinations show that the Blish-Sandstedt method gives concordant results between different collaborators. Comparative determinations on suspensions and doughs indicate that the latter give vals. of the same order of magnitude and variation as the corresponding suspensions. With flours of high *A* there was a tendency for the doughs to give comparatively lower vals. than the suspensions, and this tendency increased with the *A* of the flour, with increasing time of diastasis (*B*), and with increasing absorption in the doughs. The satisfactory agreement between the results for the total fermentable sugars (*C*) obtained by the dough-fermentation method and for the total *C* (maltose + sucrose) present in the suspensions after 4-hr. *B* showed that the Rumsey suspension type of procedure reflects *B* as it occurs in doughs containing yeast.

E. A. F.

**Diastatic activity of whole wheat and some other cereal grains as determined by the Blish-Sandstedt method.** D. A. COLEMAN, S. R. SNIDER, and H. B. DIXON (Cereal Chem., 1934, 11, 523—531).—The technique developed for determining the diastatic activity (*D*) of whole wheat, using the Blish-Sandstedt method (B., 1933, 602), possesses the following essential features: initial grain-H<sub>2</sub>O content  $\geq 13\%$  in the case of hard, and  $\geq 12.5\%$  in the case of soft, wheats; grinding temp.  $\geq 43^\circ$ ; gradual reduction of the grain. In the case of sprouted wheats or wheats of high *D* it is necessary to reduce the amount of K<sub>3</sub>Fe(CN)<sub>6</sub> and of diastatic extract. On the basis of averages it was found that the spring-grown (hard red spring and durum) wheats were highest in *D*, whilst winter (hard red and soft red winter) wheats were considerably lower in *D*. The variability in *D* within a given class, however, was not great. Different varieties, grown on the same soil and under the same climatic conditions, do not differ in *D* by  $> 10\%$ . A brief account is also given of the application of the method to rye, barley, and maize.

E. A. F.

**Diastatic powers of native [Italian] flours.** R. STRATTA (L'Ind. Chimica, 1934, 9, 1189—1193).—Stress is laid on the importance of mixing flours for bread-making in accordance with their diastatic powers, and the results of fermentation tests made on a no. of Italian flours by Joergensen's method (B., 1932, 860) are given. Addition of (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub> or NaBrO<sub>3</sub> in small proportions has virtually no effect on the fermentation curves.

T. H. P.

**Determination of the water-absorbing capacity of flour by means of the supercentrifuge.** E. B. WORKING (Cereal Chem., 1934, 11, 567—568).—The correct amount of H<sub>2</sub>O to use for experimental baking is determined by suspending flour in H<sub>2</sub>O, allowing it to digest for 30 min., and then removing most of the H<sub>2</sub>O by means of a supercentrifuge.

E. A. F.

**Modified proofing cabinet.** H. W. PUTNAM (Cereal Chem., 1934, 11, 569—570).—Modifications are described which were made in an existing 2-shelf-type cabinet so as to give perfect temp. control within a range  $\pm 0.5^\circ$ , and the desired humidity conditions in either summer or winter on the whole surface of the upper shelf (use of the lower shelf was abandoned).

E. A. F.

**Experimental baking tests. I. Effects of variation in baking formulæ on gas production and loaf volume. II. Application of a high-yeast-sugar formula in evaluating flour strength.** R. K. LARMOUR and S. F. BROCKINGTON (Cereal Chem., 1934, 11, 451—470, 470—486).—I. Gas production (*P*) and gas retention (*R*) measurements were made, using an apparatus similar to that of Bailey and Johnson (B., 1925, 111), on typical commercial and experimental Western Canadian wheat flours. Punching increased *R* in strong flours, had no effect on a weak flour, and little effect on the rate of *P* in doughs made up by either the basic or the bromate formula. Bromate increases *R* in the case of strong, and decreases it in weak, flours when *P* is adequate. Loaf vol. is closely related to *P* during the proofing, as is also response to bromate except with low-protein flours of poor quality, which generally show a negative response. Gas-rate curves and loaf vol. show that various estimations of baking quality would be obtained unless limiting conditions of fermentable sugar during proofing are eliminated, particularly with experimentally milled flours of high protein content.

II. The "fermentation (*F*) tolerance" procedure, in which the behaviour of doughs fermented for varying times is observed, is of little val. for estimating the quality of experimentally milled, Canadian hard red spring wheat flours. The 5% yeast, 6% sugar, 0.001% KBrO<sub>3</sub>, 3-hr. *F* procedure provides the best differentiation between high- and low-protein flours of this type.

E. A. F.

**Behaviour of strong flours of widely varying protein content when subjected to normal and severe baking procedures.** T. R. AITKEN and W. F. GEDDES (Cereal Chem., 1934, 11, 487—504).—With all 24 procedures investigated the loaf vol. (*V*) generally increased with protein (*P*) content, the relation being approx. linear over the entire range (11.4—15.7% *P*). The abs. *V* and the max. differentiation between samples was greatest with the formula containing 0.3 g. of high-diastatic malt, 0.1 g. of NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub>, and 0.001 g. of KBrO<sub>3</sub>, and using a 3-hr. fermentation. By imposing severe baking conditions (increasing increments of KBrO<sub>3</sub>, prolonged fermentation, extended mixing, and progressive dilution with a low-*P* flour), the *V* of the higher-*P* flours (*W*) were reduced to a greater extent than those of *W* of lower *P* content, thus decreasing the differentiation between the *W*. The addition of KBrO<sub>3</sub> and overmixing the dough appeared to exert the same general effect on gluten development (as reflected in *V*). Abs. *V* under corresponding baking conditions must be used as the criterion of strength in studies of this nature: the use of severe baking conditions is of little val., as it decreases the differentiation between the *W*.

E. A. F.

**Composition of zwieback jelly [rusk paste].** H. KÜHL (Pharm. Zentr., 1934, 75, 485—487).—German

and Dutch samples consisted of malt extract (I), soap (II), fat (III), and wax (IV). In the Dutch sample, (I) was mixed with 9% of a 60% (II), 3.0% of palm oil, 3.15% of lard-like (III), and 10% approx. of (IV). E. C. S.

**Examination of sheep's milk.** R. MARTIN (Ann. Falsif., 1934, 27, 426—430).—The vals. given previously (B., 1928, 872) for morning and evening milking should be interchanged. Variations in composition of the milk (*M*) of a flock of 170 ewes during 40 days are given. Morning *M* is usually poorer in fat than evening *M*. The means of detecting adulteration are discussed. E. C. S.

**Technique of the f.-p. test for milk.** G. D. ELSDON and J. R. STUBBS (Analyst, 1934, 59, 585—593; cf. B., 1934, 424).—Results of comparative determinations with the Hortvet (I) and Monier-Williams (II) (B., 1933, 569) cyroscopes are given. 8.5 and 9.0% aq. sucrose are used for checking thermometers at the approx. f.p. of normal milk. A modification of (I) is described which embodies certain advantages of (II). E. C. S.

**Acid content of abnormal milk.** R. VUILLAUME (Lait, 1934, 14, 12—25; Chem. Zentr., 1934, i, 2054).—In 84 tests on milk from old or emaciated cows, 47 samples were alkaline ( $pH$  6.83—7.36). H. J. E.

**Determination of the degree of heating of milk.** C. ZÄCH (Mitt. Lebensm. Hyg., 1934, 25, 87—100).—Existing methods are described, criticised, and compared for a no. of raw and dairy- and laboratory-heated milks. The max. temp. (I) of heating at which the reactions are strongly positive are as follows for milks heated for 30 min. and momentarily, respectively: peroxide test (Rothenfusser) 75°, 79°; catalase 56°, 65°; Schardinger 65°, 75°; diastase 52°, 63°; lactalbumin 58°, 72°; creaming test 60°, 72°; Schern-Gorli 60°, 72°. In general, the lower is (I) the sharper is the reaction; 30 min. at 63° is approx. equiv. to momentary heating at 80°. J. G.

**Fat losses in skim milk, and methods of determination of fat in skim milk.** F. H. McDOWALL (New Zealand Dairyman, Reprint, Oct., 1933, 8 pp.).—The vals. obtained by the Gerber and Babcock skim-milk tests are no reliable measure of the efficiency of a separator, although a val. of 0.01 or 0.02 by these methods indicates an actual % of fat not much > 0.10. The BuOH method gives fairly accurate results, usually somewhat low compared with the standard Röse-Gottlieb val. There are occasional incorrect results the causes of which are unknown. The interior of the tester must be kept hot, otherwise low results are obtained. E. C. S.

**Detection of milk pasteurisation by the Schern and Gorli ring-reaction.** R. SCARPITTI and B. ROSSI (Atti Congr. naz. Chim., 1933, 4, 818—821; Chem. Zentr., 1934, i, 2056).—The blood used as indicator in the ring reaction is replaced by a 1%  $Cr_2O_3$  suspension. A lactic acid concn. > 0.17% makes the reaction uncertain. H. J. E.

**Effect of pasteurisation on the vitamin-C content of milk.** C. G. KING and W. A. WAUGH (J. Dairy Sci., 1934, 17, 489—496).—Electrical conductivity and "flash contact" methods caused no appreciable loss of vitamin-C (I). Direct vat-heating (62° for 30 min.) resulted in significant destruction of (I). A. G. P.

**Influence of homogenisation on the curd tension of milk.** D. R. THEOPHILUS, H. C. HANSEN, and M. B. SPENCER (J. Dairy Sci., 1934, 17, 519—524).—Reduction of curd tension by homogenisation increases with the pressure used. A. G. P.

**Loss of vitality in dairy starters.** H. R. WHITEHEAD and L. WARDS (New Zealand Dept. Sci. Ind. Res., Dairy Res. Inst. Bull., 1933, No. 38, 6 pp.).—When milk, which is peculiar in a certain unknown respect, is pasteurised and cooled by the ordinary procedure, a medium is produced which has an adverse effect within 2—3 generations on certain starter bacteria. This is prevented by pasteurising and cooling in small closed vessels without stirring. E. C. S.

**Effect of heat and chemical sterilisation on the rubber parts of milking machines.** J. L. HENDERSON, C. L. ROADHOUSE, and A. FOLGER (J. Dairy Sci., 1934, 17, 475—482).—Sterilisation with aq.  $Cl_2$  (220 p.p.m.) or NaOH (0.3—0.5%) controlled thermophilic bacteria, although the total no. of surviving organisms was somewhat > when direct-heating methods were used. A. G. P.

**Effect of salt on quality of cheddar cheese.** W. RIDDET, G. M. VALENTINE, F. H. McDOWALL, and L. A. WHELAN (New Zealand Dept. Sci. Ind. Res., Dairy Res. Inst. Bull., 1933, No. 37, 19 pp.).—The effect of under- and over-salting on the texture of the rind and the main portion of the cheese (I) is described. With a normal curd (II), salting at the rate of 2¼% of the wt. of (II) at milling gave (I) less open in texture and with better body than at the rates of 3¼ and 4¼%. (I) from (II) salted at full acidity was better than (I) from (II) salted immediately after, or 20 min. after, milling. With an over-moist (II), 3¼% of salt was better than 2¼ or 4¼%, but in no case was the (I) of satisfactory quality. The % of salt of normal-bodied (II) was 1.45—1.85 (mean 1.66); that in the  $H_2O$  of normal-bodied (I) was 4.15—5.45% (mean 4.82%). The rate of salting should  $\propto$  the % of fat. E. C. S.

**Slow development of acidity in cheese manufacture. Investigation of a typical case of "non-acid" milk.** H. R. WHITEHEAD and W. RIDDET (New Zealand J. Agric., Reprint, April, 1933, 7 pp.).—In a typical "non-acid" milk the effect was due to an inhibitory substance, not destroyed by heating at 100° for 30 min., produced by a streptococcus (*S*) indistinguishable in appearance from an ordinary lactic *S*. The phenomenon could be prevented only by cooling the infected milk to such a low temp. that growth of the *S* could not take place. E. C. S.

**Slow development of acidity in cheese manufacture.** H. R. WHITEHEAD (New Zealand J. Agric., Reprint, Dec., 1933, 4 pp.; cf. preceding abstract).—The streptococcus (*S*) responsible for the phenomenon is a lactic *S*, indistinguishable in most respects from the normal organism. The inhibitory substance acts only on the lactic acid bacteria; it has no effect on *B. coli* or *B. subtilis*. E. C. S.

**Determination of lactic acid in cheese.** H. HOSTETTLER (Mitt. Lebensm. Hyg., 1934, 25, 107—124).—Methods are compared, and oxidation to  $MeCHO$ , distillation into  $NaHSO_3$ , and titration of the excess of

this with 0.005*N*-I is preferred; the end-point is sharpened by then decomposing the  $\text{MeCHO}\cdot\text{NaHSO}_3$  with  $\text{NaHCO}_3$  and completing the titration, and 0.1 mg. per 20 c.c. may be determined with an error  $\pm 2\%$ . If proteoses or  $\text{NH}_2$ -acids are present the sample is emulsified with 0.25*N*- $\text{NaOH}$ , the proteins are pptd. by saturation with  $(\text{NH}_4)_2\text{SO}_4$ , and the filtrate is extracted with  $\text{Et}_2\text{O}$ .  $\text{ZnCO}_3$  then ppts.  $\text{Zn}$  lactate, which is determined: as above; gravimetrically after washing and drying at 110–117°; or by ignition to  $\text{ZnO}$ .  $[\alpha]_D^{20}$  for the  $\text{Zn}$  salts of *d*- and *l*-lactic acid are  $-6.06^\circ$  and  $+8.6^\circ$ , respectively. Amounts found vary from  $> 1\%$  (hard cheeses) to 0.07% (white cheeses); it is unlikely that these arise from protein decomp. or sources other than lactose. J. G.

**Progressive changes in composition of edible shell eggs during storage.** L. C. MITCHELL (J. Assoc. Off. Agric. Chem., 1934, 17, 506–511; cf. B., 1933, 40, 522).—The change in wt., in the relative proportions of yolk and white, and in  $\text{Cl}$ ,  $\text{P}_2\text{O}_5$ ,  $\text{N}$ , fat, and glucose of hens' eggs stored for 60 weeks in cold storage, and for 5 weeks at room temp., are recorded. E. C. S.

**Determination of ammoniacal nitrogen in eggs.** A. W. THOMAS and M. A. V. HAUWAERT (Ind. Eng. Chem. [Anal.], 1934, 6, 338–342).—Aeration (1200 litres during 5 hr.) of egg magma at  $p_{\text{H}}$  9.5–10.7 and absorption in 0.02*N*- $\text{H}_2\text{SO}_4$  permits determination of loosely bound  $\text{NH}_3$  within  $\pm 2\%$ . At  $p_{\text{H}} > 10.7$  decomp. of proteins occurs. R. S. C.

**Swelling in canned chopped hams.** L. B. JENSEN, I. H. WOOD, and C. E. JANSEN (Ind. Eng. Chem., 1934, 26, 1118–1120).—The cultural characters of a no. of strains of a bacillus responsible for the rapid production of  $\text{CO}_2$  swells in canned spiced hams (I) are described. Gas is produced only in presence of both  $\text{NO}_3^-$  and sugar. The absence of swelling in cans incubated at 37° or 49° is no indication of the sterility or wholesomeness of (I). E. C. S.

**Spontaneous changes in meat and methods for their examination.** G. LABRANCA and F. PISTELLI (Ann. Igiene, 1932, 42, 677–682; Chem. Zentr., 1934, i, 1900).—Current methods of analysis fail to indicate incipient putrefaction of flesh of mammals or fish, since variations in analytical vals. involved are within the range observed in fresh products. Limits of variation in protein substances are narrow in mammal but wide in fish meats. A. G. P.

**Change in properties of meat proteins stored under antiseptic conditions.** V. S. SADIKOV and A. F. SCHOSCHIN (Compt. rend. Acad. Sci. U.R.S.S., 1934, 3, 258–262).—During storage of rabbit meat under  $\text{PhMe}$  the content of sol. proteins falls by approx. 15% after 2 days at 17°, and by  $> 50\%$  after 1 day at 37°, owing to conversion of globulins into insol. globulans, but partly also to peptone production from albumins. Similar results are obtained for meat stored in  $\text{CS}_2$  vapour. R. T.

**Possibility of distinguishing by physical-colloid-chemical means between sound potatoes and those suffering from "breakdown."** E. WIEGEL (Z. Spiritusind., 1934, 57, 245).—The electrometric (A.,

1933, 103),  $\text{Cu}$  (B., 1934, 252), and luminescence tests for "breakdown" in potato tubers are described. Their importance in starch manufacture is discussed. E. C. S.

**Estimation of the dry substance in tomato conserves from the determination of water by distillation.** P. GUARNIERI (Atti Congr. naz. Chim., 1933, 4, 729–731; Chem. Zentr., 1934, i, 2055).—10 g. of the material are distilled with 50–60 c.c. of a 9:1 mixture of petroleum and  $\text{C}_6\text{H}_6$ . The  $\text{H}_2\text{O}$  in the first half of the distillate is measured, and the dry substance obtained by difference. H. J. E.

**Preparation of citric acid from whortleberries.** P. GRIGOREV and G. CHOLMER (Zavod. Lab., 1934, 3, 661–662).—Citric acid is pptd. as the  $\text{Ca}$  salt from the juice. R. T.

**Preservation of lemon juice and vitamin-C.** R. DE FAZI and F. PIRRONE (Atti Congr. naz. Chim., 1933, 4, 720–724; Chem. Zentr., 1934, i, 2054).—Numerous fungi and bacteria can thrive in lemon juice, and may change its chemical composition and vitamin-C content. Sterilisation is not effected by addition of sugar or by ultra-violet light. Filtration through bacteria filters is probably effective. H. J. E.

**Acid constituents of food products, with special reference to citric, malic, and tartaric acids.** B. G. HARTMANN and F. HILLIG (J. Assoc. Off. Agric. Chem., 1934, 17, 522–531).—The citric (C) and malic acid contents of a no. of vegetables (V), and the C content of a no. of miscellaneous foods, are given. The % of tartaric acid in fruits and V is  $> 0.014$  except in the black raspberry, quince, artichoke, avocado, and grape fruit. The methods of determining  $\text{H}_2\text{C}_2\text{O}_4$ ,  $\text{BzOH}$ , isocitric, and lactic acids are discussed. E. C. S.

**Determination of lecithin in chocolate.** C. MEURICE (Bull. Off. int. Fabr. Choc., 1934, 4, 3–8; Chem. Zentr., 1934, i, 2055).—The material is extracted with  $\text{EtOH}$ , the extract treated with  $\text{H}_2\text{SO}_4$ - $\text{HNO}_3$ - $\text{HClO}_4$ , and P in the residue pptd. as molybdate and titrated with  $\text{NaOH}$ . H. J. E.

**Transport and storage of food.** ANON. (Nature, 1934, 134, 480–482). L. S. T.

**Nutrient losses during ensilage of green fodder with chemical and natural supplements.** W. KIRSCH, K. E. FEEDER, and J. LUKACZEWICZ (Bied. Zentr. [Tierernähr.], 1934, B, 6, 149–158).—Of various materials added to fodder prior to ensilage, 0.5% of sugar and "Defu" proved the most satisfactory. Use of whey resulted in considerable losses of digestible nutrients. Acidification with "Alfasil" gave irregular results and lowered the digestibility of the N-free extract. A. G. P.

**Vitamin-A and -E content of field-cured and artificially cured lucerne hay.** I. L. HATHAWAY and H. P. DAVIS (Nebraska Agric. Exp. Sta. Res. Bull., 1933, No. 62, 15 pp.).—Artificial curing conserved the vitamin-A and -E contents to a greater degree than did field-curing. A. G. P.

**Changes in vitamin content of lucerne hay.** E. DOUGLASS, J. W. TOBISKA, and C. E. VAIL (Colorado Agric. Exp. Sta. Tech. Bull., 1933, No. 4, 68 pp.).—Highest vitamin-A contents occurred in hay cut at the early blooming stage. Samples cured in diffused light

gave higher vals. for -A and -B than those cured in direct sunlight. -B<sub>1</sub> tended to decrease with advancing age of lucerne beyond the early bud stage, and also to decrease in successive cuttings in the same growing season. Loss of -B<sub>2</sub> during curing was less marked than that of other vitamins. Lucerne hay contained no -C. A. G. P.

**Determination of husk in sunflower cake.** R. HEUBLUM (Seifensieder-Ztg., 1934, 61, 34—35, 53—54; Chem. Zentr., 1934, i, 1902—1903).—Decomp. with alkali yields irregular results. Determination of the residue after peptic digestion gives reproducible vals. A. G. P.

**Extraction of gossypol from cottonseed meal. Effect of moisture and repeated extraction with ether by different procedures.** J. O. HALVERSON and F. H. SMITH (Ind. Eng. Chem. [Anal.], 1934, 6, 356—357; cf. B., 1933, 1082).—The amount of gossypol (I) extracted by Et<sub>2</sub>O is increased by addition of H<sub>2</sub>O to the meal and/or solvent. There is no definite limit between Et<sub>2</sub>O-sol. and bound (I). R. S. C.

**Rice by-products in the poultry ration for egg production and growth.** R. M. SMITH (Arkansas Agric. Exp. Sta. Bull., 1934, No. 304, 20 pp.).—Rice products supplemented with a vitamin-A concentrate may replace the customary grain ration without adverse influence on growth of hens, or the yield, hatchability, or storage properties of eggs. A. G. P.

**Rice by-products for feeding swine and effects on quality of pork.** E. MARTIN (Arkansas Agric. Exp. Sta. Bull., 1934, No. 303, 36 pp.).—Uses of rice bran, polish, etc. in mixed rations are examined. The products may be used in pig-fattening provided the animals are finished on hard fat-producing foods. A. G. P.

**Grinding of feeding-stuffs in a power mill.** F. J. ELLIOTT (Analyst, 1934, 59, 606—609).—In the milling of feeding-stuff samples, dust is lost which is richer in N than the bulk of the material. E. C. S.

**Odour and taste of metals.**—See X. **Passion fruit seed oil.**—See XII. **Determining glucose syrup.**—See XVII. **White-lupin flour.**—See XIX.

See also A., Oct., 1069, **Changes in starch and staling of bread.** 1084, **Determination [of fat in chocolate] by extraction.** 1126 and 1145—6, **Vitamins.** 1130, **Nutritive val. of suet and sunflower oil,** and (1132) of **green immature soya beans.**

#### PATENTS.

**[Cereal] food product and its preparation.** F. B. DEHN. From MEAD JOHNSON & Co. (B.P. 416,149, 12.6.33).—A cereal prep. is made by cooking 100 lb. of the following mixture with 35 gals. of H<sub>2</sub>O and drying the product on a heated drum: wheatmeal 52.5, oatmeal 18.0, cornmeal 10.0, wheat germ 15.0, NaCl 0.5, lucerne 1.0, dried brewers' yeast 1.0, edible bone-meal 2.0%. E. B. H.

**Production of improved cheese products.** A. K. EPSTEIN and B. R. HARRIS (U.S.P. 1,945,345, 30.1.34. Appl., 15.8.30).—Sterilised or partly sterilised cheeses are flavoured with  $\alpha$ -diketones having 4—8 C (Ac<sub>2</sub>). H. A. P.

**Treatment of an Irish potato:** (A) to remove the corky layer, (B) having the skin removed, with fumes of burning sulphur to bleach, preserve, and

prevent discoloration. (A) S. A. PATTERSON, (B) C. D. DRAPER, Assrs. to A. P. GROMMESCH (U.S.P. 1,948,884 and 1,948,877, 27.2.34. Appl., [A] 8.8.31, [B] 12.9.31).—(A) Removal of the corky layer with preservation of the natural eye marks etc. is effected by a severe heat shock at  $\leq 260^\circ$  (600—1100°) for 7—25 sec., followed by cooling and scrubbing in H<sub>2</sub>O sprays. (B) The potatoes are then subjected for 15—20 min. to an atm. containing 0.25—0.50 oz. of S per cu. yd. B. M. V.

**Treatment of fruit juice.** CONSERVENFABR. LENZBURG VORM. HENCKELL & ROTH, and J. SCHLÖR (B.P. 416,632, 2.2.34).—Jellification of conc. fruit juice is prevented by subjecting the juice (before concn.) to a pressure of 4—6 atm., in an atm. of CO<sub>2</sub>, for 60 hr. E. B. H.

**Treatment of earth-nut kernels.** P. AMMANN (B.P. 416,818, 17.10.33. Fr., 21.10.32).—The decorticated kernels (K) are heated and dried (to 1—4% H<sub>2</sub>O) at 80—90° without previous grinding, and the whole K pressed to remove 60—70% of the oil. The skins are removed from the flattened K, and the latter then treated by steam and finally heated or roasted. The product has the vol. and shape of the original K and is suitable for human food. E. B. H.

**Manufacture of molasses feed.** A. H. SCHMIDT (U.S.P. 1,945,918, 6.2.34. Appl., 27.7.31).—A process and apparatus are described for mixing the molasses with cereal or roughage and drying and disintegrating the product. E. B. H.

**Manufacture of [laminated, readily-soluble, table] jellies.** CHIVERS & SONS, LTD., and T. RENDLE (B.P. 416,777, 23.12.32).

**Heat-transfer apparatus [for cream].**—See I. **Treating oleaginous seeds etc.**—See XII. **Photometric apparatus.**—See XXI.

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

**Microchemistry of atophan.** M. WAGENAAR (Pharm. Weekblad, 1934, 71, 1100—1103).—Characteristic dichroic crystals (herapatites) are obtained when atophan is treated in aq.-CO<sub>2</sub> solution with KI-I solution (I) and H<sub>2</sub>SO<sub>4</sub> or H<sub>2</sub>SeO<sub>4</sub> (sensitivity 0.05 mg., 1 : 200). Intense blue crystals are obtained with (I) and HCl in aq. CCl<sub>3</sub>-CH(OH)<sub>2</sub>. The reaction with Br is sensitive but not characteristic. Phenylecinchonic acid is pptd. by HCl, HNO<sub>3</sub>, NH<sub>4</sub>NO<sub>3</sub>, or (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> (sensitivity 0.05 mg., 1 : 200). S. C.

**Comparison of methods for evaluation of pyrethrum.** C. B. GNADINGER and C. S. CORL (Soap, 1934, 10, No. 9, 89).—The Seil acid method (B., 1934, 603) gives slightly higher vals. for pyrethrin content than does the Gnadinger-Corl Cu reduction method (B., 1929, 996). L. D. G.

**Identification of superol.** G. H. WAGENAAR (Pharm. Weekblad, 1934, 71, 1122—1124).—The following microchemical reactions for superol [8-hydroxyquinoline (I) sulphate] are described, the sensitivity being given in parentheses: emerald-green colour with FeCl<sub>3</sub> (II); SO<sub>4</sub><sup>''</sup> with BaCl<sub>2</sub>; picrate (III) (1 : 5000); dark brown ferrocyanide (1 : 300); (I) with NaHCO<sub>3</sub>, Na<sub>2</sub>CO<sub>3</sub>, or NH<sub>3</sub> sol. in NaOH (very sensitive); cryst. ppts. with PtCl<sub>4</sub>, HgCl<sub>2</sub>, AgNO<sub>3</sub>; (II) and (III) are most characteristic. S. C.

**Stability of aqueous dispersions of lecithin.** G. MALQUORI (Atti. Congr. naz. Chim., 1933, 4, 752—753; Chem. Zentr., 1934, i, 2004).—Small proportions of Ca salts flocculate, larger concns. peptise, and still higher concns. flocculate the dispersions. Stability ranges for a no. of org. salts of Ca are given. A. G. P.

**Norwegian tobaccos.** A. HEIDUSCHKA and E. NIER (Pharm. Zentr., 1934, 75, 646—647).—Analyses of various brands are given. The nicotine contents varied from 2.13 to 4.01% (1.13% for cigarettes) and 44—53.8% was recovered from the smoke (10.27% in case of cigarettes). S. C.

**Paraffin in cassia oil.** SALAMON & SEABER (Perf. Ess. Oil Rec., 1934, 25, 277).—The *n* of the portion unabsorbed in NaHSO<sub>3</sub>, especially after treatment with H<sub>2</sub>SO<sub>4</sub>, is the best indication (cf. B., 1934, 701). E. H. S.

**Sassafras wood and bark.** F. W. FREISE (Pharm. Zentr., 1934, 75, 627—628).—The following types are found as adulterants of Brazilian *Sassafras officinale*, Nees: *Miscanthea*, the essential oil of which contains 10% of apiol (I); *Ocotea*, containing less safrole (II) and 4.5—8.5% of (I); *Siparuma erythrocarpa*, D. Cand., which on steam-distillation gave 1.15—1.65% of oil (*d*<sub>20</sub> 1.0195—1.0225, *n*<sub>20</sub> 1.4855, dextrorotatory, sol. in 70% EtOH) containing 45% of (II). S. C.

**Determination of aldehydes in essential oils.** M. A. SCHWARZ (Annali Chim. Appl., 1934, 24, 352—355).—Higher aliphatic aldehydes, salicyl- and anisaldehydes may be determined by applying Ripper's iodometric method (A., 1901, ii, 205) to 0.5—0.06% solutions of the aldehydes in aq. EtOH. Anisaldehyde in anise oil and cinnamaldehyde in cannella oil may be determined by van Eck's benzidine titration method (A., 1928, 313). T. H. P.

**Determination of volatile oils in herbs, spices, and drugs.** C. E. SAGE and H. R. FLECK (Analyst, 1934, 59, 614—618).—Apparatus and procedure for the determination of volatile oil (*V*) are described. The % of *V* in numerous samples of mint, thyme, and marjoram, with suggested "useful limits," and in certain other herbs, drugs, and spices, are given, and compared with the volatile Et<sub>2</sub>O extract as usually determined. E. C. S.

**Determination of admixtures in crystalline perfumes with the quartz analytical lamp.** E. EKMAN and A. SAMYCHLAYEWA (Riechstoffind. u. Kosmetik, 1933, 8, 221—222; Chem. Zentr., 1934, i, 2049).—Fluorescence (I) phenomena are described for numerous perfumes. Changes in (I) may be used to detect adulterants. H. J. E.

**Effect of oils, fats, and waxes on antiseptic action.** L. D. GALLOWAY (Manuig. Chem., 1934, 5, 233—234).—The unexpectedly low toxicity to moulds and bacteria shown by many antiseptics when added to emulsions is due to their greater solubility in the non-aq. phase of the emulsions. L. D. G.

**Dielectric constants.**—See XI. Determining glucose syrup.—See XVII.

See also A., Oct., 1079, Na tartrate oxidation and diabetes. 1099, Prep. of salts of ergosteryl sul-

phate. 1109, Bacteriostatic azo compounds. 1111, Antimalarials from aminoquinolines. 1112, Optically active barbituric acids. 1115, Bactericidal thiazole-azo dyes. Thiomorpoline derivatives. 1120, HgI<sub>2</sub>-KI reagent for alkaloids. 1126 and 1145—6, Vitamins. 1140, Fermentation of tobacco. 1141, Purified tuberculin and vaccinia virus. 1143, Assay of insulin.

## PATENTS.

**Manufacture of therapeutic media [halogenopolyhydroxyalkylbenzenes].** I. G. FARBENIND. A. G. (B.P. 415,715, 28.2.33. Ger., 29.2.32).—Polyhydroxyalkylbenzenes having a side-chain of 6—12 C are halogenated (Cl<sub>2</sub>, ICl, and a metal catalyst, e.g., Fe, SO<sub>2</sub>Cl<sub>2</sub>), or a halogenated polyhydric phenol is condensed with a higher aliphatic alcohol, olefine, or acid (ZnCl<sub>2</sub>, H<sub>2</sub>SO<sub>4</sub>), and the product in the last-named case reduced (Clemmensen), or, finally, a suitably substituted compound is converted into a OH-compound by nuclear replacement (e.g., Br → OH). The products are in most cases sol. in aq. Na<sub>2</sub>CO<sub>3</sub>. Examples include the prep. of 5-chloro-, b.p. 155°/0.5 mm., and 3:5-dichloro-2:4-dihydroxy-*n*-octylbenzene, b.p. 170°/0.5 mm., 3:5-dichloro-2:4-dihydroxy-*n*-heptyl-, b.p. 165—170°/1 mm., and -*n*-hexylbenzene, b.p. 145°/0.5 mm., 3:5-dibromo-2:4-dihydroxy-*n*-decylbenzene, 5-chloro-2:4-dihydroxy-*n*-octyl- and -*n*-dodecylbenzene (direct halogenation); 2-chloro-4:5-dihydroxy-*n*-octylbenzene, b.p. 180—185°/4 mm. (acid), 5-chloro-2:4-dihydroxy-*tert*-heptyl-, b.p. 158—164°/6 mm. (olefine), and 5-chloro-2:3-hydroxyisooctylbenzene, b.p. 148—152°/4.5 mm. (3-Br-compound and 15—20% NaOH at 180°). H. A. P.

**Manufacture of anæsthetic preparations.** H. A. METZ LABORATORIES, INC., Assees. of A. WINGLER (B.P. 416,193, 3.10.33. U.S., 3.10.32).—An anæsthetic (*p*-NH<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·CO<sub>2</sub>·C<sub>2</sub>H<sub>4</sub>·NET<sub>3</sub>·HCl), a vaso-constrictor (adrenaline), a reducing agent (Na<sub>2</sub>SO<sub>3</sub>, Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>, NaHSO<sub>3</sub>), and a buffer to give a solution isotonic with blood (NaHCO<sub>3</sub>, Na<sub>2</sub>HPO<sub>4</sub>) are thoroughly dried, mixed, and pressed into tablets. H. A. P.

**Manufacture of [barbituric acid] derivatives of [hydr]oxy- and amino-pyridines.** CHEM. FABR. VON HEYDEN A. G. (B.P. 416,273, 7.3.34. Ger., 31.3.33).—Prep. of cryst. additive compounds is claimed. Examples are diethylbarbituric acid (I) (1 mol.) + 5-nitro-4-hydroxy- (1 mol.), m.p. 245°, 5-acetamido-2-ethoxy- (2 mols.), m.p. 112—115°, and 2-amino-pyridine (2 mols.), m.p. 82—85°, phenylethylbarbituric acid + 2-hydroxy-1-methylpyridine (II) (equimols.), m.p. 122°, and (I) + (II) (equimols.), m.p. 120°. H. A. P.

**Production of concentrated solutions of follicle hormones and their esters.** SCHERING-KAHLBAUM A. G. (B.P. 416,256, 30.1.34. Ger., 31.1.33).—Approx. 0.5—0.7% solutions in vegetable oils (olive, sesame) are producible in presence of phenolic esters (0.5—1% of *m*-C<sub>6</sub>H<sub>4</sub>Me·OAc or *p*-C<sub>6</sub>H<sub>4</sub>Me·OBz). H. A. P.

**Photometric apparatus.**—See XXI.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Newspaper records.—See V.

See also A., Oct., 1079, **Formation of AgBr emulsion. Theory of development.** 1114, Phthalocyanines. 1115, Oxacyanines.

## PATENTS.

**Sensitisation of photographic silver halide emulsions.** I. G. FARBENIND. A.-G. (B.P. 415,949, 26.1.34. Ger., 26.1.33).—The use of *s*-naphthoxocarboyanines derived from 2-methyl- $\alpha$ - or - $\beta$ -naphthoxazole and a carboxylic acid  $> \text{HCO}_2\text{H}$  (cf. B.P. 412,309; B., 1934, 753) is claimed. H. A. P.

**Production of photographic dyestuff images.** B. GASPÁR (B.P. 416,666, 14.2.33. Ger., 15.2.32).—In processes in which a Ag-dye image is treated so that the dye is destroyed at either the image or non-image portions (cf. B.P. 395,718, and 397,159; B., 1933, 813, 941), the method is characterised by having a vigorous Ag image such that the destruction of the dye takes place in presence of an amount of Ag  $>$  that required for the dye removal, the remaining Ag being removed in a subsequent reducing bath. Suitable dye bleaching baths are made with  $\text{CuSO}_4$  or  $\text{CuCl}_2$ , with  $\text{HNO}_3$ . J. L.

**Production of coloured photographic materials.** B. GASPÁR (B.P. 415,756, 2.12.32. Ger., 2.12.31).—A  $\text{H}_2\text{O}$ -insol. salt of an acid or substantive dye (I) with an org. base (II) (guanidine, 4-amino-2-phenylquinoline), prepared by pptn. of (I) with a  $\text{H}_2\text{O}$ -sol. salt of (II), is used to colour a photographic gelatin layer. H. A. P.

**Photographic film having an antihalation layer.** I. G. FARBENIND. A.-G. (B.P. 416,198, 16.10.33. Ger., 15.10.32).—Lenticular films are protected from halation by applying a layer of dye (cf. B.P. 383,799 and 399,387; B., 1933, 93, 1085) or pigment, decolorisable in the usual developing or fixing baths, incorporated in gelatin, to the smooth surface of the film, the emulsion being then coated on top of the pigment layer. Suitable pigments are finely-divided Ag, or glassmaker's soap. The concn. of the layer must be such that the layer absorbs about 20% of the light passing through the lenticular elements (cf. B.P. 312,992; B., 1930, 841). The dye or pigment should be so mixed as to produce a grey (non-selective) colour. J. L.

**Manufacture of (A) protected, (B) coated, motion-picture film.** L. L. STEELE, ASSR. to ESSEM LABS., INC. (U.S.P. 1,946,004—5, 6.2.34. Appl., 28.2.29).—(A) The film is treated with a polyglycol, preferably diethylene glycol (I). The best results are obtained with a 2:1:1 mixture of  $\text{H}_2\text{O}$ , EtOH, and (I); the treatment may be applied after development, before drying, or to the finished film. The dried film may be coated with a layer of a cellulose ester composition to give a hard, resistant surface. (B) The film may be coated with a mixture of the cellulose ester [e.g., nitrocellulose (II)] and (I). A satisfactory mixture comprises 100 pts. of (II), 40 pts. of (I), 200 pts. of  $\text{BuOAc}$ , 150 pts. of  $\text{BuOH}$ , 1250 pts. of denatured EtOH, and 1250 pts. of toluol. Flexibility of the film is increased, and a highly resistant surface provided. J. L.

**Photometric apparatus for measuring substances [vitamin-A] which absorb radiation.** A. HILGER, LTD., F. TWYMAN, and D. H. FOLLETT (B.P. 416,423, 10.3. and 23.5.33, 5.1. and 17.1.34).—Apparatus is

described in which light of (approx.) 3280 Å., produced by filtering the light from an Fe or Cu arc with a thinly silvered quartz plate, is divided into two beams, one passing through a quartz cell containing the oil under examination, and the other through a variable aperture. By equalising the images thrown on a fluorescent screen the absorption of the oil may be measured; this figure may be compared with that of the oil after it has been treated (blown with  $\text{O}_2$ , or irradiated) to destroy the vitamin-A. Various optical devices which may be used are described. The apparatus may be adapted to measure absorptions at other wave-lengths, for the measurement of, e.g., vitamin-C or -D content. J. L.

**Production of positives from two-colour screen negatives.** G. S. WHITFIELD (B.P. 416,114, 6.4.33).—The two positives are printed, preferably by contact printing, simultaneously on to two emulsions (E) coated on opposite sides of a single support, the E being selectively sensitised to the two colours; white light or a mixture of two appropriate coloured lights is used. The E nearest the negative may contain a filter dye. The second E is exposed through the first E and the support. The layers are appropriately dyed after development. The two-colour positive obtained may be combined with a third colour record. J. L.

**Ferropussiate printing. [Development of blue-prints.]** H. D. MURRAY, D. A. SPENCER, and NORTON & GREGORY, LTD. (B.P. 416,489, 10.3. and 28.6.33).—Instead of fixing blue-prints by washing in  $\text{H}_2\text{O}$ , the unchanged ferricyanide is converted into an insol. compound (I) which cannot react to give a blue colour. In order of preference, the print may be treated, by a roller machine, with a solution of a salt of Ni, Zn, Mn, Cd, or Co; the unexposed parts of the print will be coloured yellow, pale yellow, pink, yellow, and brick-red, respectively. Preferably oxidising agents, e.g.,  $(\text{NH}_4)_2\text{S}_2\text{O}_8$  together with  $\text{NaClO}_3$ , are added. To prevent bleaching of the Prussian-blue (by alkali) or dissolution of the (I) by too much acid, the solution should be kept just acid by buffering with, e.g.,  $(\text{NH}_4)_2\text{HPO}_4$  and  $\text{NH}_4\text{H}_2\text{PO}_4$ . The printing paper for this process preferably contains an excess of ferricyanide over  $\text{Fe}^{\text{III}}$  salt; the comparatively small depth of blue colour thus obtained may be compensated by colouring the surface with tartrazine, giving yellow lines on a dark green background. Wetting agents may be added to the fixing solution. J. L.

**Colour photography. [Means for taking and projecting colour-record photographs on lenticular films.]** C. NORDMANN (B.P. 416,602, 24.5.33. Fr., 28.6.32).

**Film base from seaweed.**—See V. **Artificial compositions.**—See XIII.

## XXII.—EXPLOSIVES; MATCHES.

**Condition of the mixture of camphor and diphenylamine in the powder.** L. G. SVETLOV (Kriegschem., 1933, No. 2, 3—6; Chem. Zentr., 1934, i, 1922).—Camphor (I) and  $\text{NHPh}_2$  form a eutectic [f.p. 18—22°, 54% of (I)]. Vaporisation is minimised by using a concn. of (I)  $<$  that for the eutectic.

H. J. E.

**Defence against gas attack.** G. F. JAUBERT (Mém. Soc. Ing. Civ. France, 1934, 87, 277—281).—The use of gases in warfare is outlined; these are classified as asphyxiating, asphyxiating and toxic, lachrymatory, sternutatory, and blistering. W. J. W.

**Filters for use in gas warfare.** L. BERGÉ (Mém. Soc. Ing. Civ. France, 1934, 87, 287—304).—Various types of gas mask are described and details of their manufacture given. W. J. W.

**Shelters against gas attack.**—See XXIII.

See also A., Oct., 1080, **Explosion temp. of complex Cu<sup>II</sup> salts with C<sub>2</sub>H<sub>4</sub>(NH<sub>2</sub>)<sub>2</sub>.**

## PATENT.

**Manufacture of safety explosives.** J. S. B. FLEMING, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 416,586, 14.3.33).—A safety sheath for blasting explosives consists of fibrous material (I) (cellulose pulp), impregnated with cooling salts, e.g., NaHCO<sub>3</sub>, NaCl, or mixtures of these, the amount of (I) being 4—10% of the total dry constituents. A non-volatile, hygroscopic substance may be added to prevent loss of flexibility through evaporation. W. J. W.

## XXIII.—SANITATION; WATER PURIFICATION.

**Disinfection of gas masks.** H. STOLTZENBERG, M. Y. LIU, and W. STOLTZENBERG (Chem.-Ztg., 1934, 58, 791—792).—Fumigation with CH<sub>2</sub>O (I) is recommended. Where a chemical method of vaporising the (I) is desirable, BaO<sub>2</sub> is superior to KMnO<sub>4</sub>, CaOCl<sub>2</sub>, Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, CaO, or NaClO<sub>3</sub>. For a room of 30 cu. m. capacity, 690 g. of BaO<sub>2</sub> and 600 c.c. of 40% (I) are required. L. D. G.

**Isolators and shelters [against gas attack],** G. F. JAUBERT (Mém. Soc. Ing. Civ. France, 1934, 87, 305—372).—Isolators may be portable devices or adapted to extract toxic gases from the air entering enclosed spaces. The Oxylythe-Lemoine apparatus and the construction of shelters are described. W. J. W.

**Evaluation of the germicidal potency of chlorine compounds. I. Hypochlorites.** C. K. JOHNS (Sci. Agric., 1934, 14, 585—607).—Germicidal efficiency in Cl compounds depends on their available Cl content and on *p*<sub>H</sub>. Discrepancies in comparative vals. at varying dilution are attributable to differences in buffer capacity of the preps. A. G. P.

**Coagulants used in water purification.** L. L. HEDGEPEETH (J. Amer. Water Works Assoc., 1934, 26, 1222—1233).—The composition, advantages, and disadvantages of the materials commonly used for this purpose are described. Chlorinated coppers and aq. FeCl<sub>3</sub> (in 8000-gal. tank-car lots) show a substantial saving in cost, on the basis of the oxides present, over Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, provided the railway facilities are available. C. J.

**"Henry" process for clarification of polluted water.** ANON. (Engineering, 1934, 138, 213—215, 293—295).—Coal-washing slurry or sewage sludge is made alkaline (*p*<sub>H</sub> 11.0) with Ca(OH)<sub>2</sub> and treated with a small amount of a mixture of frozen potato-starch and NaOH. The suspended particles coalesce and settle very readily; the supernatant liquor is suitable for re-use in the coal-washing plant or for dis-

charge into the river. The densified sludge can be treated on a vac. filter, to produce a cake with 15—20% H<sub>2</sub>O which may be burned or buried. The addition of a little clay or coal dust is recommended as an aid in clarifying sewage. C. J.

**Distillation apparatus for phenols in water.** E. A. MEANES and E. L. NEWMAN (Ind. Eng. Chem. [Anal.], 1934, 6, 375).—A single-unit apparatus is described. E. S. H.

**Taste control of water supplies.** R. A. IRWIN (J. Amer. Water Works Assoc., 1934, 26, 1202—1213).—The most commonly adopted treatments for taste and odour removal are prechlorination, activated C, and NH<sub>3</sub>-Cl<sub>2</sub>. C. J.

**Determination of ammonia [in water] in the field.** W. S. DAVIS and C. B. KELLY (J. Amer. Water Works Assoc., 1934, 26, 1196—1201).—Direct nesslerisation of the sample in square 2-oz. bottles is recommended; the comparison is made through the sides of the bottle with permanent standards of K<sub>2</sub>PtCl<sub>6</sub> and CoCl<sub>2</sub> modified for the reduced depth of liquid used. C. J.

**Effect of iron in the determination of residual chlorine [in water].** R. D. SCOTT (J. Amer. Water Works Assoc., 1934, 26, 1234—1237).—In distilled H<sub>2</sub>O, Fe<sup>+++</sup> in amounts < 1.0 p.p.m. gives false results with the *o*-tolidine (I) test on prolonged keeping; with a natural (river) H<sub>2</sub>O the effect is much accentuated and a tap-H<sub>2</sub>O gave intermediate results. Rise of temp. to 30° produced an increased effect, but light had no action. The use of a modified (I) reagent is recommended, 2 c.c. of which contain the normal amount of (I) but 4 times the amount of HCl present in 1 c.c. of standard reagent. C. J.

**Photometric micro-analysis of drinking and service water. XII. Determination of iron.** C. URBACH (Mikrochem., 1934, 15, 207—226; cf. B., 1934, 654).—Total Fe in 100 c.c. is determined by acidifying (Congo-red), adding 2 c.c. of 2*N*-NH<sub>4</sub>Cl and 2 c.c. of 20% sulphosalicylic acid, then making alkaline with 2 c.c. of 10% aq. NH<sub>3</sub>. The yellow colour produced is photometered by monochromatic light. Fe<sup>+++</sup> is similarly determined in a 100-c.c. sample, acidified by addition of 0.5 c.c. of 2*N*-HCl after neutralising against Congo-red. In presence of Mn, NO<sub>2</sub>, or org. matter, the sample is boiled with 1—2 c.c. of 0.1*N*-KMnO<sub>4</sub>, excess KMnO<sub>4</sub> destroyed with H<sub>2</sub>O<sub>2</sub>, and the solution neutralised. Total Fe only is then determined in acid solution. J. S. A.

**C for gas masks.**—See II. **Corrosion [in mains].**—See X. **Soil fertilisation and drainage H<sub>2</sub>O.**—See XVI. **Antiseptic action.**—See XX.

See also A., Oct., 1084, **Determining SO<sub>4</sub><sup>''</sup> in waters and mineral waters.** 1142, **Oligodynamic action of metals.**

## PATENT.

**Regeneration cartridges for breathing apparatus.** DEGEA A.-G. (AUERGES.), Assees. of DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (B.P. 413,979, 29.1.34. Ger., 27.1.33).—The filling substance (KOH or NaOH) is used in the form of spheres or ellipsoids of an average diam. of 4 mm. A. R. P.