

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

MAR. 3 and 10, 1933.\*

### I.—GENERAL; PLANT; MACHINERY.

**Softening of feed water by phosphate before purification.** W. WESLY (Angew. Chem., 1933, 46, 19—20).—The residual hardness of  $H_2O$ , after treatment with NaOH, can be reduced by addition of  $PO_4'''$ . A max. effect is reached after 1 hr., further treatment having little more effect. The improvement brought about by  $PO_4'''$  is the more marked the less is the excess of NaOH added previously. A better result is obtained before than after purification of the  $H_2O$ . E. S. H.

**Control tests for treatment of feed and boiler water.** J. K. RUMMEL (J. Amer. Water Works Assoc., 1932, 24, 2004—2024).—Methods are described for the determination of NaOH,  $Na_2CO_3$ ,  $Na_2HPO_4$ , and  $SiO_2$  in boiler waters etc. When determining the alkalinity, titration with 0.05N- $H_2SO_4$  in the presence of a universal indicator ( $p_H$  4—11) is suggested, readings being taken at  $p_H$  8 and 5. Phosphate may be determined volumetrically or colorimetrically; in the latter case a method based on the reduction of phosphomolybdic acid by 1 : 2 : 4-aminonaphtholsulphonic acid is recommended. A colorimetric method for the determination of  $SiO_2$  depends on the production of a yellow colour by reaction with  $NH_4$  molybdate in an acid solution.

C. J.

**Portable hardness testing machine, with diamond pyramid indenter.** S. R. M. PORTER (J. Sci. Instr., 1933, 10, 18—20).—A steady load of 10 kg. is applied directly by hand through a spring to a diamond indenter for a definite time. The size of the impression is read by means of a portable microscope with micrometer attachment.

O. J. W.

**Industrial hydrogenation.** J. B. PHILLIPS (Chem. & Ind., 1933, 51—53).—A summary.

**Coal for steam-generator furnaces. Retort-bench vac. control.**—See II. **Refractories for boilers.**—See VIII. **Heat regenerators. Thermo-elements.**—See X.

### PATENTS.

**Regenerative furnace.** L. C. EDGAR (U.S.P. 1,853,409, 12.4.32. Appl., 8.7.30).—An open-hearth furnace is regenerative, but the flow of gases in the regenerators and furnace does not reverse; the former are parallel to each other and U-shaped in plan, and the air and gases are interchanged from one to the other by means of simple dampers.

B. M. V.

**Muffle furnaces.** GIBBONS BROS., LTD., and T. E. BRIDGFORD (B.P. 384,798, 10.12.31).—The heating gases are passed first through combustion passages, inside the muffle, which are structurally independent of the main

chamber, then through hollow bricks forming ribs on the floor, and finally through flues in the base of the setting in recuperative relation to flues for incoming air.

B. M. V.

**Rotary tube furnaces.** METALLGES. A.-G. (B.P. 384,931, 4.7.32. Ger., 6.7.31).—A rotary kiln is provided with fuel burners extending more or less radially through the cylindrical wall at various distances from the end. They are supplied with fuel and, if desired, also with air through pipes fastened to the outside of the kiln and brought to a manifold at one, preferably the discharge, end.

B. M. V.

**Heating of [horizontal] retorts.** KOPPERS COKE OVEN CO., LTD. From N. V. MAATS. VOOR KERAMISCHE EN CHEMISCHE INDUSTRIE "KERO-CHEMICA" (B.P. 384,934, 8.7.32).—The retorts are set in vertical rows with longitudinal fins on the centre line of each row completely preventing transverse passage of heating gases; there are also transverse partitions extending alternately all the way and not quite all the way from top to bottom, forming a no. of pairs of vertical flues in which the flow of gases is up one side and down the other of the shorter partitions. Jet passages at the bottom of the vertical flues are used alternately for admission of fuel and air and exhaust of products of combustion from and to reversing regenerators in the base of the setting.

B. M. V.

**[Steel] vessels for use in carrying out chemical processes [hydrogenation of coal and oil] under high pressures and temperatures.** F. KRUPP A.-G. (B.P. 385,781, 20.2.32. Ger., 27.4.31).—The vessels comprise two concentric cylindrical steel shells shrunk on to one another, the outer shell being made of a steel having a high strength at high temp., e.g., steel containing 1—5% Cr and  $\nabla$  3% Mo, with or without  $\nabla$  0.5% V or Ti, and the inner shell of a steel resistant to chemical action, e.g., one containing 5—20% Cr, 0—3% Mo, and, if desired, a small amount of Ni. Some or all of the Mo may be replaced by 3 times as much W or by an equal wt. of V.

A. R. P.

**Process and apparatus for catalytic gaseous reactions.** J. A. ALMQUIST, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,855,040, 19.4.32 Appl., 31.12.28).—A purifying catalyst (A) and a main catalyst (B) are contained in separate pressure-resisting chambers, and the gases pass first through an annular chamber surrounding B where they are preheated, then to A, and return in contact with B.

B. M. V.

**Process and apparatus for exothermic reactions.** L. LHEURE, Assr. to E. I. DU PONT DE NEMOURS & Co.

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

(U.S.P. 1,855,134, 19.4.32. Appl., 27.2.29. Fr., 2.3.28).—In a system having a similar object to that of the preceding abstract, a condenser for the removal of converted impurities may be inserted in the return flow from the purifier, and to obtain conveniently the additional heat exchange both catalysts are in one pressure-resisting vessel, the purifier innermost, and the annular space is subdivided by a thin heat-transmitting wall separating the flow of initial entry from that of re-entry.

B. M. V.

**Vacuum drying apparatus.** A. E. JONSSON (B.P. 385,670, 4.9.31. Swed., 25.9.30).—In a vac. drum provided with strainers for catching solid matter in the issuing stream of vapour, condensation on, and consequent choking of, the screens by wet solid matter is prevented by providing chambers, just beyond the screens, which are heated either by the heat provided for evaporation or a special supply.

B. M. V.

**Rotary drums, e.g., for rotary cooking boilers, drying apparatus, or the like.** A./S. KVAERNER BRUG (B.P. 384,696, 28.7.31. Norw., 29.7.30).—Bearings permitting the trunnions to run slightly out of line are described.

B. M. V.

**Grinding or crushing apparatus.** R. A. LISTER & Co., LTD., and W. G. MAW (B.P. 385,100, 8.12.31).—A no. of swing hammers are of increasing length to correspond with a sloping curved screen above which they operate.

B. M. V.

**Grinding or pulverising apparatus.** H. D. TOLLE-MACHE (B.P. 385,374, 1.10.31).—In apparatus, preferably of the ball-mill type, having the ground material borne away by an air current, the settled oversize material from the air separator is allowed to fall within range of a scoop attached to the outlet trunnion of the mill and is then conveyed back through that trunnion by means of a helical blade, the core of the helix being open to permit exit of the air-borne stream of separated ground material.

B. M. V.

**Making articles of bonded granular material [abrasive wheels].** NORTON Co. (B.P. 385,328, 19.6.31. U.S., 19.6.30).—Grains, selected as to material and size but not as regards shape, are mixed in calc. quantity with a calc. quantity of bond and pressed to a calc. vol. before baking, the change in vol. during baking being taken into account. The effects of the 3 variable proportions (of grains, binder, and voids) are discussed, and several series have been designed having one of these factors const. in the series.

B. M. V.

**Hardness-testing of materials.** G. REICHERTER (B.P. 384,885, 9.4.32. Ger., 23.12.31).—In an apparatus of the type effecting indentation by steady pressure, the sample is first clamped between the table and head of the machine by, e.g., springs with a load of the same order as the indenting pressure, which is additional, but localised.

B. M. V.

**Conveying muddy substances, e.g., thick cement sludge.** F. KRUPP GRUSONWERK A.-G. (B.P. 385,137, 26.2.32. Ger., 6.3.31).—Rakes are operated in the same manner as in a Dorr classifier, but are in a substantially horizontal trough; the link motion is also different.

B. M. V.

**Filtering apparatus for liquids.** R. POWLEY & SONS, LTD., and W. POWLEY (B.P. 385,365, 18.9.31).—The liquid, e.g., bottle-washing liquor, is passed through several (e.g., 3) screens of increasing fineness of mesh in series, the first screen being in the form of a moving band and the others trays between the upper and lower runs of the band. The labels etc. are washed off the band as it passes over the end pulley.

B. M. V.

**Filters [for saccharine or other solutions].** C. McNEIL (B.P. 384,749, 26.10.31).—Filter bags with a filling of, e.g., corrugated plate are attached to a framework of suction pipes and rotated in a bath of the prefill.

B. M. V.

**Filtration and clarification of water.** FILTRATION & WATER SOFTENING PROPRIETARY, LTD. (B.P. 385,679, 28.9.31. Austral., 29.9.30).—The acid reaction produced by treatment with certain coagulants is corrected by contact with alkali or alkaline-earth silicates or Ca aluminate slags. Suitable apparatus is claimed.

C. J.

**Anti-rust solution for radiators.** A. DJIDICH (U.S.P. 1,853,341, 12.4.32. Appl., 15.1.30).—An 8:1 mixture of saturated aq.  $\text{Na}_2\text{CO}_3$  and saturated aq.  $\text{CuSO}_4$  is added in the proportion of 8 oz. to 5 gals. of radiator  $\text{H}_2\text{O}$ .

A. R. P.

**Centrifugal extractor.** H. KRANTZ (U.S.P. 1,853,821, 12.4.32. Appl., 20.5.29).—A centrifuge has practically all the moving parts supported on brackets uprising from the base, and the whole is surrounded by a dome-like cover bedded down on soft packing in the base.

B. M. V.

**Centrifugal machine.** J. B. KIRBY (U.S.P. 1,853,866, 12.4.32. Appl., 7.6.26).—A variable-speed friction drive is described.

B. M. V.

**Distillation of substances that are liquid at distillation temperature.** METALLGES. A.-G. (B.P. 385,866, 17.6.32. Ger., 25.6.31).—Substances boiling at a high temp. (e.g., mineral oil, b.p.  $300^\circ$ ) are distilled under high vac. by the application of heat and the injection of a substance boiling at a lower temp. (e.g., light oil, b.p.  $200^\circ$ ), this b.p. being still considerably above that of the available cooling  $\text{H}_2\text{O}$  so that a high vac. may be maintained. The desired fractions of heavy oil and the light oil are condensed in separate condensers at successively lower temp. and the light oil is re-used. A two-stage system comprising an ordinary still operating under moderate vac., the liquid in which is in direct connexion with the liquid in a second still, is described. Two compartments are provided in the latter at a difference in level corresponding to the increased vac. desired, the gas-lifting effect of the injected oil being used to circulate the heavy oil between the two compartments.

B. M. V.

**Evaporators for distilling or concentrating apparatus.** W. C. MASON and W. W. HUTCHESON (B.P. 384,741, 12.10.31).—A calandria is arranged in a sloping position and the rows of tubes (on the outside of which the steam is condensed) are staggered in such a way that the condensate never drips on to a lower tube, but falls on to inclined baffles which lead it to the centre-line, whence it flows over a single wide tube

forming a preheater and downtake for the circulating liquor. B. M. V.

**Fluid contact apparatus.** F. W. SPERR, JUN., Assr. to KOPPERS Co. (U.S.P. 1,854,492, 19.4.32. Appl., 15.7.26).—Gas is disseminated into a liquid by means of rotating cylinders of mesh-work partly submerged in the liquid, the gas being supplied to the interior of the screens by means of their hollow, perforated shafts. B. M. V.

**Regulating the viscosity of [liquid] materials.** A. OSBOURNE (B.P. 384,827, 14.1.32).—A paddle or other body is rotated in the liquid and the driving motor is pivotally mounted and counterpoised to resist any desired torque. On excessive tilting of the motor, electric switches are closed which start another motor to regulate a valve for addition of diluting liquid. B. M. V.

**Pneumatic separators.** ALLGEM. ELEKTRICITÄTS GES. (B.P. 385,014, 7.8.31. Ger., 9.8.30).—In a deflection-type separator, regulating dampers are crescent-shaped and pivoted about their centres. B. M. V.

**Air separator.** T. J. STURTEVANT, Assr. to STURTEVANT MILL Co. (U.S.P. 1,853,942, 12.4.32. Appl., 2.4.30).—Bearings for the bevel driving gear are described. B. M. V.

**Separation of suspended material from gases.** E. ANDERSON, Assr. to INTERNAT. PRECIPITATION Co. (U.S.P. 1,853,393, 12.4.32. Appl., 9.4.26).—The suspended material is agglomerated by passing through an electric field of high potential (which need not be rectified) and the agglomerated particles are arrested by a filter; agglomeration must be carried only to such a point that the filter remains permeable to solids. B. M. V.

**Removal of gases from materials containing many capillary spaces.** N.V. MAATS. TOT EXPLOIT. VAN "TEN BOSCH OCTROOIEN N.V." (B.P. 385,479, 8.3.32. Holl., 19.3.31).—The process described in B.P. 359,984 (B., 1932, 133) is extended to the removal of gas films, mechanical pressure as well as a difference of temp. being applied. The removal of gas assists greatly in the removal of any liquid which may be present. B. M. V.

**Means of conditioning air for dehydrating purposes, especially for withering tea leaf.** S. STROMGREN, and ALLMÄNNA SVENSKA ELEKTRISKA AKTIEB. (B.P. 385,180, 26.4.32).—The air is passed between cold and warm surfaces opposite to each other. Concentric tubes may be used, the outer surfaces being exposed to a warm atm. and the inner tubes containing refrigerating fluid which causes deposition of dew from the air in the annular spaces even though the mean temp. of the air is not much changed. B. M. V.

**Conditioning of gases.** W. W. TRIGGS. From INDUSTRIAL DRYER CORP. (B.P. 384,666, 4.6.31).—Two separate flows (*a*, *b*) of air or other gases are subjected *a* to heating and humidification, *b* to cooling by the latent heat of the moisture vaporised into *a*, to such an extent that part of its moisture may be pptd. An apparatus described comprises a no. of vertical tubes surrounded by tubular wicks kept moist by H<sub>2</sub>O contained on horizontal trays forming partitions for

zigzag flow of *a*, heaters being placed in the return bends. The gas *b* flows downwards through the tubes. B. M. V.

**Devices for allowing gases to bubble through a liquid.** ENGLISH ELECTRIC Co., LTD., and H. I. ANDREWS (B.P. 384,858, 29.2.32).—The application of the bird-bath feed principle, *e.g.*, to the lubricators of electric traction motors, is claimed, an object of the invention being to confine the surface of the liquid from which the gas is bubbled to as small an area as possible, whereby the effect of disturbances in the surface of the liquid on the level maintained is reduced to a min. B. M. V.

**Apparatus for thoroughly intermingling gases or vapours with molten substances.** P. WEFELSCHIED, Assr. to AMER. LURGI CORP. (U.S.P. 1,853,590, 12.4.32. Appl., 17.2.31. Ger., 18.3.30).—A centrifugal pump, preferably submerged in the molten substance, *e.g.*, metal, draws through separate inlet apertures both molten substance and gases and discharges the mixture below the surface of the bath. B. M. V.

**Apparatus for treating material with gases.** A. ANDREAS (B.P. 385,109, 18.12.31. Ger., 19.12.31. Addn. to B.P. 360,547; B., 1932, 87).—A no. of different forms of vertical permeable walls to confine the loose material (cf. the prior patent) are described. Examples are: chain-grate conveyors, rollers which may be hollow and have axial cooling fluids, cruciform rollers. B. M. V.

**Apparatus in which pulverulent material is conveyed in suspension in a gas stream.** ASHINGTON COAL Co., LTD., J. TAIT, and W. J. DRUMMOND (B.P. 385,428, 24.12.31).—In an air-borne pulverising system for solid fuel etc., choking due to excess feed causes the vac. in the outlet pipe to the fan (or pressure in the inlet pipe from the fan to the pulveriser) to rise; a small pipe transmits the vac. to H<sub>2</sub>O in a chamber in which is a float, operating a valve which transmits when necessary the same vac. (but through a different small pipe) to a control device on the feeder, *e.g.*, a spring-loaded diaphragm which will lift a pawl out of engagement with the feeder ratchet. B. M. V.

**Apparatus for spraying materials.** E. ROTHEIM (B.P. 385,380, 15.10.31. Norw., 16.12.30).—A spraying device for attachment to the stopper of a can containing a substance dissolved in a liquefied gas is described, the spraying being effected by the self-generated pressure of the gas. B. M. V.

**Rectification of vapours.** P. SUBKOW, Assr. to UNION OIL Co. OF CALIFORNIA (U.S.P. 1,854,002, 12.4.32. Appl., 6.9.27).—In a rectifying column operated under such a high vac. that the loss of head in the bubbling trays would, if summated, be prohibitive, the vapours are collected in a closed passage beneath each tray and injector devices are there provided so that the loss of head at each tray is neutralised on the spot. The ejectors may be operated by external fluid or by vapours pumped from the compartment above or below. B. M. V.

**Filling and drawing off liquefied gases.** L'AIR LIQUIDE SOC. ANON. POUR L'ETUDE ET L'EXPLOIT. DES

PROC. G. CLAUDE (B.P. 385,167, 8.4.32. Ger., 8.4.31).—A storage vessel is divided into two compartments, preferably by suspending a small vessel in it. Into one compartment newly manufactured liquid may be admitted under atm. pressure while from the other either liquid or gas may be delivered under self-generated pressure. B. M. V.

[Mercury-vapour] vacuum pumping systems. BRIT. THOMSON-HOUSTON CO., LTD., and J. C. READ (B.P. 385,376, 10.10.31).—In the conduit connecting a Hg-vapour pump (*A*) to a Hg arc rectifier (*B*), a collecting channel around the wall is situated at the desired level of Hg in *A* and connected thereto by a U-tube; Hg from *A* distils back towards *B* and is condensed on the wall of the conduit, which is H<sub>2</sub>O-cooled for the purpose. Any excess not required to maintain the level in *A* is allowed to flow back into *B*. B. M. V.

Separating materials of different sp. gr.—See II. Drying apparatus for pottery etc.—See VIII. Kneading machine.—See XIX.

## II.—FUEL; GAS; TAR; MINERAL OILS.

Manchurian coals. S. OKA (J. Fuel Soc. Japan, 1932, 11, 115—117).—The coal resources of Manchuria are estimated at 4800 million tons; the total production in 1930 was over 10 million tons. The types of coal found are briefly described; they include both anthracites and bituminous coals. A. B. M.

Comparison of processes for the cleaning of coal. W. R. CHAPMAN (J. Inst. Fuel, 1932, 6, 104—114).—The advantages of coal cleaning are discussed and typical cleaning processes are described and compared. A. B. M.

Action of water vapour on coal and coke [and graphite]. B. NEUMANN, C. KRÖGER, and E. FINGAS (Z. Elektrochem., 1932, 38, 936—938).—P. Dolch's views (B., 1932, 5) are criticised. The reactions  $C + H_2O = CO_2 + 2H_2$ ,  $2C + 2H_2O = CO_2 + CH_4$ , and  $C + CO_2 = 2CO$  probably occur in addition to the water-gas reactions. The action of steam on pure graphite and on graphite containing 8% of Fe<sub>2</sub>O<sub>3</sub>, CuO, Cr<sub>2</sub>O<sub>3</sub>, U<sub>3</sub>O<sub>8</sub>, Al<sub>2</sub>O<sub>3</sub>, and K<sub>2</sub>O has been investigated at various temp. CH<sub>4</sub> is formed and the water-gas equilibrium is not established. D. R. D.

Electrostatic precipitation in the carbonisation industries. C. H. S. TUPHOLME (Chem. & Ind., 1932, 918—920).—The operation of the Lodge-Cottrell, Simon-Carves, and Sturtevant electrostatic precipitators for de-tarring town gas is discussed. A tar extractor (spray-washer type) and a Lodge-Cottrell precipitator, working with settings of 5-ton continuous vertical retorts, gave liquor and tar yields (in gals. per ton of coal) of 18.5 and 15.4, and 23.3 and 17.6, respectively. The use of electrical de-tarrers leads to improved recovery and purity of by-products from coke-oven gas. H. E. B.

Modern trends in British by-product coking industry. G. E. FOXWELL (Chem. & Ind., 1933, 49—51).

Oxidation of semi-coke with nitric acid, nitric acid vapour, and metallic oxides. T. BAHR (Ges.

Abh. Kenntn. Kohle, 1932, 10, 116—124; Chem. Zentr., 1932, ii, 2264).—Semi-coke was nearly completely dissolved by 98% HNO<sub>3</sub>; mellitic acid was detected among the acids. Oxidation with HNO<sub>3</sub> vapour at 140—470° permits arrest at an intermediate stage. Heating with metallic oxides in vac. afforded H<sub>2</sub>O and CO<sub>2</sub> practically exclusively. A. A. E.

Active carbon from fuel ash. W. FUCHS (Ges. Abh. Kenntn. Kohle, 1932, 10, 144—151; Chem. Zentr., 1932, ii, 2265).—Of a lignite ash, > 50% C (active) was separated with HCl; further activation is effected by heating at 800°. A. A. E.

Hydrogenation of Fushun coal. T. OGAWA (J. Fuel Soc. Japan, 1932, 11, 118—119).—The coal was hydrogenated in a 2-litre rotating autoclave or in a 600-c.c. shaking autoclave. Coal tar was a better vehicle than were other heavy oils; the smaller the proportion of vehicle used the greater was the yield of liquid products from the coal. Addition of Fe<sub>2</sub>O<sub>3</sub> increased the oil yield and prevented coke formation. Other oxides and salts (not specified) also formed efficient catalysts. The optimum reaction temp. were 420—480° in the presence of tar and 425—450° with no vehicle. Increased pressure up to 300 atm. improved the results; pressures below 200 atm. were ineffective. The yields from 100 kg. of coal and 20 kg. of H<sub>2</sub> were: gasoline 17, heavy oil 24, pitch 20, unconverted coal and ash 16, H<sub>2</sub>O 14, and of gas (including 14 kg. of H<sub>2</sub>) 29 kg. A. B. M.

Thermal reaction and hydrogenation of coal. I. Thermal reaction of Fushun coal under a high-pressure nitrogen atmosphere. II. Berginisation of Fushun coal at 350—500°. R. ABE (J. Soc. Chem. Ind., Japan, 1932, 35, 503—507B).—I. Fushun coal was heated in an autoclave with paraffin oil under an initial pressure of 75 kg. per sq. cm. of N<sub>2</sub> and the results were compared with those obtained by the Bergius process carried out at the same pressure, Fe<sub>3</sub>O<sub>4</sub> being used as catalyst in each case. In the N<sub>2</sub> atm. the liquid product formed a very uniform paste at 350—450°, but changed completely to coke at 500°. The output of oil from 350° to 450° was independent of temp. and was approx. 17% of the pure coal. There was not much gaseous hydrocarbon produced at 450°, but 22.1% at 500°. At 350° 0.2 wt.-% of H<sub>2</sub> was formed, but at 350—450° there was none. More probably hydrogenation took place.

II. In a H<sub>2</sub> atm. coking took place at 450°. Very little gaseous hydrocarbon was formed and very little of the products were absorbed by the active C. The max. output of "berginised" oil was 35% of the coal, increasing very rapidly from 400° to 450°. The tendency of the paste to undergo fractionation at 350° was the same in both cases, indicating small change of coal at this temp. Hydrogenation took place above 499° and reached about 3% of the coal. At 350° it was negative although liquefaction at first increased. M. S. B.

Theory and practice of the gasification of coal in the furnaces of steam generators. H. L. PIRIE (J. Inst. Fuel, 1932, 6, 115—123).—The classification and evaluation of coal from the viewpoint of furnace

firing for steam generation, and the calculation of combustion data, furnace losses, etc. are discussed.

A. B. M.

**Theory and practice of the combustion of solid, liquid, and gaseous industrial fuels compared with coal for steam raising.** H. L. PIRIE (J. Inst. Fuel, 1932, 6, 124—128; cf. preceding abstract).—The discussion of the preceding paper is extended to cover gaseous, liquid, and some other solid fuels than coal, e.g., coke.

A. B. M.

**Control of retort-bench vacuum.** W. CAMPBELL (Gas World, 1933, 98, 31—34).—The design of the retort-house governor, the position of the offtakes and foul main, and the ability of the exhauster to maintain a const. pull are discussed in relation to this problem.

R. N. B.

**Drying and denaphthalising town gas.** ANON. (Gas World, 1933, 98, 37).—Gas is cooled by passing countercurrent to a spray of H<sub>2</sub>O chilled to 0° by an EtCl expansion refrigerator. The gas leaves the plant at 4° and rises to an average temp. of 15° in the mains, thus itself acting as a removal agent for H<sub>2</sub>O and C<sub>10</sub>H<sub>8</sub>. As the cooling H<sub>2</sub>O circulates in a closed system it becomes saturated with hydrocarbons and there is no subsequent loss of calorific val. The cost of the operation is claimed to be 0.8d./cu. ft.

R. N. B.

**Treatment of tar.** J. G. KING and M. A. MATTHEWS (J. Inst. Fuel, 1932, 6, 83—87).—A discussion on a paper previously abstracted (B., 1933, 6).

A. B. M.

**Determination of the creosote content of brown-coal tars. I. Effect of the acid constituents of brown-coal tar on the electrical conductivity of 10% sodium hydroxide solution.** W. FRANKE (Braunkohlenarch., 1930, No. 29, 39—58; Chem. Zentr., 1932, ii, 2269).—In order to provide data for the determination of creosote by measurement of the change in conductivity of a 10% NaOH extract, the effect of various phenols and mixtures thereof was measured. With 0.5N-phenols in 10% NaOH the conductivity falls in the order PhOH, cresols, xyenols, thymol. In an isomeric series the conductivity is the smaller the greater is the separation of Me from OH. The relation between mol. wt. and acidity holds only for monohydric phenols. The same quantity of the phenol, whether added to a dil. or a conc. phenolic solution, produces the same effect. A mixture of equal parts of PhOH, *o*-, *m*-, and *p*-cresol, *m*-4- and *p*-xylenol, thymol, and guaiacol (0.5N) gave a mean sp. conductivity scarcely differing from the calc. val. Omission or addition of phenols of moderate "acidity" did not markedly affect the conductivity, but substitution of eugenol for PhOH considerably diminished the conductivity. Small additions of AcOH have no disturbing effect, and vals. for PhOH-AcOH mixtures can be calc. from vals. for the pure substances.

A. A. E.

**Determination of the creosote content of brown-coal tars. II. Quantitative extraction of creosote with 10% sodium hydroxide solution.** W. FRANKE (Braunkohlenarch., 1930, No. 31, 15—28; Chem. Zentr., 1932, ii, 2269—2270).—The decrease in the sp. conductivity of 10% NaOH solution caused by dissolved hydrocarbons is very small; C<sub>5</sub>H<sub>5</sub>N has a marked, and

$\alpha$ -picoline a smaller, effect, whilst quinoline has practically no influence. The distribution of phenols between solvents and NaOH solution has been determined. Corrections for the presence of C<sub>6</sub>H<sub>6</sub> and H<sub>2</sub>O in the determination of creosote by conductivity measurements are proposed.

A. A. E.

**Determination of paraffin in brown-coal tars.** B. G. SIMEK (Teer u. Bitumen, 1932, 30, 305—309; Chem. Zentr., 1932, ii, 2270).—A vac.-distillation apparatus is described. Butanone is recommended as precipitant.

A. A. E.

**Paraffin in petroleum bitumen.** J. B. LONGINUS (Erdöl u. Teer, 1932, 8, 125—127; Chem. Zentr., 1932, ii, 2400—2401).—The val. of the product is closely associated with its paraffin and S contents.

A. A. E.

**Petroleum bitumens.** G. BANDTE (Erdöl u. Teer, 1932, 8, 380—383; Chem. Zentr., 1932, ii, 2401).—Polemic (cf. preceding abstract).

A. A. E.

**Physico-chemical properties of bitumen emulsions.** E. VELLINGER and R. FLAVIGNY (Ann. Off. Nat. Combust. liq., 1932, 7, 217—224; Chem. Zentr., 1932, ii, 2401).—The emulsions (soap and 50% bitumen) are most stable at  $p_H$  9 and 13. The active acids are much more active in the dissociated than in the undissociated condition. The surface activity of the bitumens depends also on their origin and treatment.

A. A. E.

**Separation of the original tar from road-tar emulsions.** H. WAGNER (Mitt. Materialprüf., 1932, 220).—Removal of the H<sub>2</sub>O by distillation is a lengthy operation and is accompanied by much foaming and by the danger of change in composition of the tar. Pptn. of the tar from the emulsion by treatment with EtOH, acids, bases, or salts is fraught with similar difficulties, as is also extraction with C<sub>6</sub>H<sub>6</sub>. The best method appears to be filtration through a series of porous clay plates which absorb the H<sub>2</sub>O and the emulsifier without removing the free C from the tar or altering its composition in any material respect.

A. R. P.

**Colloid chemistry of asphalts.** C. MACK (J. Physical Chem., 1932, 36, 2901—2914).—Asphalts are considered as sols of asphaltenes in a mixture of asphaltic resins and oily constituents. Whilst the relative viscosity ( $\eta$ ) of asphalts at lower temp. is high, at 120° the liquids behave as ideal solutions. Structure viscosity was not observed. The mol. wt.,  $M$ , calc. from the equation  $\log \eta = CkM$  (where  $C$  is the concn. and  $k$  a const.), is 1800 at 120°. Mol. wt. determinations and adsorption measurements of asphaltic resins on asphaltenes appear to indicate that high viscosity is brought about by association rather than by solvation.

E. S. H.

**Cost of producing solid and liquid hydrocarbons from bituminous sand.** S. C. ELLS (Canada Dept. Mines, Mineral Resources Invest., 1932, No. 127, 140—145).—Estimated costs are analysed, and a flow sheet is given.

C. W. G.

**Fractionation of hydrocarbons by distillation with steam.** C. AB-DER-HALDEN (Rev. Pétrol., 1932, 905—907; Chem. Zentr., 1932, ii, 2400).—Large-scale

experiments illustrate the advantage of rectification with the aid of superheated steam. A. A. E.

**Determination of sulphur in petroleum.** J. GIVAUDON (Rev. Pétrol., 1932, 892—896; Chem. Zentr., 1932, ii, 2270).—Grote's method is described. The lamp method has been improved.  $H_2S$  in petroleum is removed with  $N_2$  and passed into  $Zn(OAc)_2$  solution, the  $ZnS$  being determined iodometrically. A. A. E.

**Determination of the organic bases in a Bergius-process oil.** J. M. PERTIERRA (Anal. Fis. Quím., 1932, 30, 792—793).—By Hinsberg's method the composition of the basic fraction is found to be: primary bases 3.8, secondary 27.6, *tert.* 68.6%. The high proportion of *tert.* bases is a factor in the colloidal dispersion of the coal. R. K. C.

**Production of light motor fuels.** N. MAYER (Chem.-Ztg., 1933, 57, 81—83).—A review.

**Nitrotoluenes from gasoline.**—See III. **Bitumen paints.**—See XIII. **Brown coal and plant nutrition.**—See XVI. **Refuse disposal.**—See XXIII.

#### PATENTS.

**Apparatus for washing coal.** F. H. BLATCH (B.P. 385,372, 25.9.31).—In an apparatus in which a downward stream of raw coal meets an upward current of  $H_2O$ , the rush of coal causes the  $H_2O$  level in the refuse elevator chamber (*A*) to rise; a float is situated in *A* to regulate the feed of coal. B. M. V.

**Separation of materials of different sp. gr. [e.g., coal from refuse].** T. M. CHANCE (U.S.P. 1,854,107, 12.4.32. Appl., 30.9.27).—In a system for the cleaning of coal by flotation in a sand pulp, fine coal is separated from the return sand by using the sand-collecting sump as a classifier and overflowing the fine coal with the return  $H_2O$ ; no definite attempt to separate coal from the main stream of  $H_2O$  is made, but a proportion of that stream is withdrawn, e.g., by siphon, from the bottom of the inlet sump of the return- $H_2O$  pump, and treated in an external apparatus for recovery of the fine coal. The bulk of the fine coal is removed by a dry screen previously to the wet treatment and the proportion of  $H_2O$  withdrawn from the system is automatically adjusted by the siphon to equal that added in the form of various clean sprays. B. M. V.

**Blending of materials in the making of briquettes.** C. S. WOLF, Assr. to AMER. BRIQUET Co. (U.S.P. 1,851,689, 29.3.32. Appl., 21.8.29).—Finely-pulverised coking coal is mixed with a binder, which consists of an emulsion of asphalt or pitch in a paste of starch or other carbohydrate, and the product is mixed with pulverised non-coking fuel, e.g., anthracite culm, in such proportion as to give a ratio of non-coking to coking fuel of about 93:7 in the final mixture, which is then briquetted. The viscosity of the mixture of binder and coking coal may be decreased by heating it to about 150° under pressure (3 atm.); the subsequent mixing is thereby facilitated. A. B. M.

**Carbonisation of solid combustibles by internal heating.** D. DE NAGY (B.P. 384,092, 15.12.31).—Separate closed containers, sector-shaped in plan, are arranged around a vertical shaft with which they are

connected by valved offtake pipes leading from both the top and the bottom of each container. The containers are provided with lids through which they can be charged with the material (coal, peat, straw, or grass) to be carbonised, and with doors which permit ignition of the material, the heat required for carbonisation being derived from the combustion of part of the charge. The outer wall of each container is perforated by a no. of small apertures, some or all of which may be closed by plugs, whereby the entry of air into the container during carbonisation is controlled. The gaseous products of carbonisation are withdrawn by suction through the central shaft. A. B. M.

**Distillation of coal at a low temperature.** C. FRANÇOIS (B.P. 384,171—2, 23.4.32).—(A) The coal is compressed into a series of small compartments (about 250 c.c. capacity) arranged side by side to form a pocketed plate, a no. of such plates are superimposed so that each compartment is closed, though not in a gastight manner, by the bottom of the corresponding compartment in the superimposed plate (the compartments of the top plate being provided with special closing elements), and the whole is heated to 550—850°. The compartments are so shaped that passages are formed between them to allow access of heat and free liberation of the products of distillation. (B) Carbonisation is effected by passing the pile of charged plates through an externally-heated tunnel or muffle oven, suitable forms of which are described. A. B. M.

**Gas producers.** J. GOHN (B.P. 383,980, 23.7.31).—The apparatus comprises a chamber (*A*) for the instantaneous gasification of the fuel, surmounted by a fuel reservoir and connected thereto by means of a conduit, a valve in which controls the rate of flow of fuel to *A*. The latter is provided with one or more tuyères (*B*), arranged axially or laterally therein, through which compressed air is injected into *A*. Both *A* and *B* may be water-jacketed. The supply of air is automatically regulated in accordance with the pressure in the gas offtake. The ash may be removed periodically from the bottom of *A*, or, by the provision of an extra tuyère, may be melted and removed continuously in the liquid form. The gases are filtered by being passed laterally through columns of sand or gravel supported between suitably spaced, inclined surfaces. A. B. M.

**Manufacture of gas.** H. ROSENTHAL, Assr. to COLUMBIA ENG. & MANAGEMENT CORP. (U.S.P. 1,853,084, 12.4.32. Appl., 1.3.28).—In a carburetted water-gas plant part only of the blast gases is burned in the carburettor and superheater, the remainder, after passing through the wash-box, being enriched by the addition of sufficient  $C_3H_8$  or  $C_4H_{10}$  to raise its calorific val. to equal that of the carburetted gas produced during the run, with which it is then mixed. A. B. M.

**Purification of gas.** W. J. HUFF (U.S.P. 1,851,312, 29.3.32. Appl., 24.5.29).— $SO_2$  and  $H_2S$  are removed simultaneously from gases by passing them over hydrated  $Fe_2O_3$ , or oxides of other metals of groups V—VIII, at temp. below 100°. Gases containing  $H_2S$  may be freed therefrom by oxidising part of the  $H_2S$  to  $SO_2$  by known means and then treating as above. A. B. M.

**Gas purification.** D. L. JACOBSON, Assr. to KOPPERS Co. (U.S.P. 1,852,014, 5.4.32. Appl., 23.2.29).—Gases are purified from  $H_2S$  etc. by washing with an aq. solution containing the equiv. of about 1% of  $As_2O_3$  in the form of  $NH_4$  arsenite and/or thioarsenite, the composition of the solution being such that it has substantially no v.p. with respect to  $NH_3$ . The fouled liquid is regenerated, with separation of free S, by aëration. A. B. M.

**Gas purification.** J. C. WOODHOUSE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,852,497, 5.4.32. Appl., 5.11.30).—Gases are freed from  $CO_2$  at elevated temp., e.g.,  $< 100^\circ$ , by treatment with  $FeO$ , preferably under pressure ( $> 5$  atm.). The process is facilitated by adding Al or an alkaline-earth halide to the  $FeO$ . In the catalytic production of mixtures of  $H_2$  and  $CO_2$ , e.g., from steam and  $CH_4$ , the  $FeO$  may be added to the catalyst in order to remove the  $CO_2$  as it is formed. A. B. M.

**Freeing coke-oven gas from nitrogen oxides and/or converting the latter into ammonia.** H. TROPSCH and R. KASSLER (B.P. 384,532, 16.4.32. Czechoslov., 22.4.31).—The gas is passed over a catalyst consisting of the sulphides of metals of group VI, e.g., Mo or W, at  $200$ – $230^\circ$ , under normal or increased pressure. A suitable catalyst may be prepared by impregnating pumice stone with  $NH_4$  molybdate and then subjecting it to the action of  $H_2S$  at  $210^\circ$ . A. B. M.

**Production of ammonium sulphate from coke-oven gas and other gases obtained from the distillation of coal.** CARGO FLEET IRON Co., LTD., and A. J. CADDICK (B.P. 384,392, 22.6.32).—The gases, when they have arrived at substantially the normal temp., are passed into an aq. suspension of alkaline-earth sulphate, and the liquor produced, after separation of the pptd. carbonate, is evaporated to crystallisation. A. B. M.

**Treatment of gases arising in the distillation or carbonisation of coal and like carboniferous material.** C. COOPER, D. M. HENSHAW and W. C. HOLMES & Co., LTD. (B.P. 384,388, 25.9.31).—The  $H_2O$ -vapour content of the gases is reduced by treating them with a solution of  $NH_4$  salts as described in B.P. 304,333 (B., 1929, 232), the evaporation of the  $H_2O$  taken up being effected by bringing the diluted solution into intimate contact with a stream of heated gas from an external source, e.g., flue gas. A. B. M.

**Compositions comprising asphalt emulsions and products made therefrom.** K. D. SVENSSON (B.P. 384,138, 18.2.32).—A composition for road coatings, for the insulation of concrete basements, etc. is made by mixing an asphalt emulsion, of a min. concn. of 40%, with a colloidal solution of caoutchouc, gutta-percha, or balata, also of a min. concn. of 40%. Substances for increasing the viscosity of the emulsion, e.g., leico gum or water-glass, protective colloids, e.g., casein, and/or fillers, e.g., gravel, may be added. A. B. M.

**Treatment of natural gas.** J. M. WADSWORTH (U.S.P. 1,851,743, 29.3.32. Appl., 1.4.26).—The gas is scrubbed with oil for the recovery of gasoline and the enriched oil is fractionated under pressure in a rectifying column, the distillate from which is further fractionated, also under pressure, in a second column to remove

the normally gaseous hydrocarbons from the gasoline. The pressure used is developed by making use of the v.p. of the enriched oil itself, and is adjusted to be sufficiently high to give the required sharp fractionation and to permit condensation of the gasoline, which, if desired, may contain all or most of the  $C_4H_{10}$  present, to be effected with ordinary cooling means. A. B. M.

**Inhibition of gum formation in low-boiling hydrocarbon oils and products obtained thereby.** STANDARD OIL DEVELOPMENT Co., Asses. of C. L. GUTZEIT (B.P. 385,066, 6.11.31. U.S., 21.11.30).—Indophenols, oxazines, indamines, eurhodines, safranines, aposafranines, and aminoazo compounds (except Spirit-yellow R), free from  $SO_3H$  and  $S-CH_2Ph$  groups, are added as bases to petrols, free from  $Fe(CO)_5$ , as gum inhibitors, and may be renewed as the colour fades. C. H.

**[Stabilisation of] mineral oil [or grease] compositions.** P. I. MURRILL, Assr. to R. T. VANDERBILT Co., INC. (U.S.P. 1,852,788, 5.4.32. Appl., 4.3.30).—Salts of 2- or 4-hydroxydiphenyl with org. stabilising bases, e.g., alkylenediaryldiamines, are added as stabilisers. C. H.

**Treatment of carbonaceous articles.** M. J. MARCIN and D. MACKENZIE (B.P. 386,022, 14.8.31. U.S., 29.8.30).—See U.S.P. 1,804,361; B., 1932, 827.

**Heating of retorts. Vessels for hydrogenations. Distillation [of, e.g., oils]. Conveying pulverulent material. Rectification of vapours.**—See I. Purification of S.—See VII. Protecting metal surfaces.—See X. Disinfecting etc. agents.—See XX.

### III.—ORGANIC INTERMEDIATES.

**Determination of methyl alcohol in liquids containing ethyl alcohol.** H. JEGLINSKI (Pharm. Ztg., 1933, 78, 77).—The Patzsch method (B., 1933, 43) is criticised. The EtOH content of the standard solution in the colorimetric determination of MeOH by Denigès' method should be approx. the same as that of the test solution. The reaction is of max. sensitiveness in solutions containing 20% EtOH. A. A. L.

**Behaviour towards hard water of condensation products of high-molecular aliphatic alcohols with sulphonation media.** WELWART (Seifensieder-Ztg., 1932, 59, 427; Chem. Zentr., 1932, ii, 2257).—Phenols or cresols diminish the salting-out of the condensation products by Ca. Mg has no salting-out action. A. A. E.

**Preparation of mono- or di-nitrotoluene from the aromatic gasoline of the Shukkōkō crude oils by direct nitration.** M. MIZUTA (J. Soc. Chem. Ind., Japan, 1932, 35, 519B).—Mononitrotoluene (mainly the *o*-compound) and dinitrotoluene were recovered from mixtures of PhMe and the aromatic-free toluene fraction (b.p.  $97$ – $120^\circ$ ) of Shukkōkō crude oils, in as high yield, and of as good quality, as from pure PhMe. E. L.

**Creosote.**—See II. New emulsifier.—See XII.

### PATENTS

**Forming compounds from acetylene [and organic compounds].** W. W. TRIGGS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 384,332, 30.5.31).—For

condensations of  $C_2H_2$  with alcohols, phenols, arylamines, hydrocarbons, ethers, or esters in presence of a dry Hg catalyst, an anhyd. fluoro-acid (fluoboric or fluosilicic) is used in place of the usual  $H_2SO_4$ ; the acid may be in the form of Hg salt, e.g., from  $BF_3$  and HgO. Products from  $C_2H_2$  and glycerol, glycol Et ether, Et lactate,  $NH_2Ph$ , and  $PhOH$  are described. C. H.

**Acetylene reactions. Polymerisation products of [acetylene and of] non-benzenoid acetylene polymericides, and composition containing the same.** W. W. TRIGGS. From E. I. DU PONT DE NEMOURS & Co. (B.P. 384,654—5, 27.5.31).—(A)  $C_2H_2$  is polymerised by contact at 20—40° with a catalyst prepared from a Cu salt and an  $NH_4$  or *tert.* amine salt in  $H_2O$ , the main products being divinylacetylene, b.p. 80—85°, and monovinylacetylene, b.p. 6—7°. (B) These non-benzenoid polymericides are further polymerised by heat or by treatment with air or other gas containing  $O_2$  below 113°. Formation of explosive products may be prevented by polymerising at 84—87° in a non-oxidising atm. in presence of 0.2% of phenols or amines as anti-oxidants. The degree of polymerisation may be regulated so as to give liquid products. The polymericides are applied in coating compositions, moulding powders, etc. C. H.

**Manufacture of aliphatic halogenated alcohols.** I. G. FARBENIND. A.-G. (B.P. 384,156, 19.3.32. Ger., 21.3.31).—Saturated halogenated aliphatic aldehydes are treated with magnesyl alkoxides, e.g.,  $BrMgOEt$ ,  $BrMgOPr\beta$ , or the product from  $BrMgEt$  and hydrobenzoin, and then with  $H_2O$ . The reaction is:  $BrMgO \cdot CH_2R + \cdot CHO \rightarrow X \cdot CH(O \cdot CH_2R)(OMgBr) \rightarrow X \cdot CH_2 \cdot OH + RCHO + MgBrOH$ . In the examples X is  $CBR_3$  (alcohol, m.p. 79—80°, b.p. 92—93°/10 mm.),  $CCl_3$  (b.p. 55—56°/11 mm.),  $CH \cdot CMe \cdot CCl_2$  (m.p. 61°),  $CHBr_2$  (b.p. 70—72°/10 mm.), and  $CH_2Cl$  (b.p. 128°). C. H.

**Manufacture of sulphuric acid esters of alcohols.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 382,942, 17.7.31).—Alcohols, especially alcohols above  $C_{10}$ , are sulphated by heating with a sulphamic acid or a salt of  $NH(SO_3H)_2$ . Examples are H sulphates from: stearyl alcohol with  $NH_2 \cdot SO_3H$ , *p*- $C_6H_4Me \cdot NH \cdot SO_3H$ , or  $NH_2Bz$  salt of  $NHBz \cdot SO_3H$ ; alcohols from oxidised paraffin wax with  $NH_2 \cdot SO_3H$  or  $NH(SO_3NH_4)_2$ ; lauric  $\beta$ -hydroxyethylamide with  $NH_2 \cdot SO_3H$  and  $C_5H_5N$ ;  $BuOH$  with  $NH(SO_3NH_4)_2$ . C. H.

**Catalytic production of amines from alcohols and ammonia.** E. I. DU PONT DE NEMOURS & Co. (B.P. 384,714, 27.8.31. U.S., 27.8.30).—An aliphatic alcohol vapour is passed with  $NH_3$  at 250—500° over a porous rigid (e.g.,  $SiO_2$ ) gel impregnated with a dehydrating oxide catalyst, e.g.,  $Al_2O_3$  or  $B_2O_3$ . C. H.

**Manufacture of condensation products [from polyhydric alcohols and cyclic ketones].** I. G. FARBENIND. A.-G. (B.P. 385,139, 3.3.32. Ger., 4.3.31. Addn. to B.P. 383,764; B., 1933, 199).—Other polyhydric alcohols above  $C_5$  are used in place of polyvinyl alcohol for condensation with cyclic ketones; the alcohols may be produced *in situ* from esters. Acetal-like products, stable up to 200°, are obtained from cyclohexanone and glucose (m.p. 134°), mannitol (m.p. 89°),

fructose (m.p. 145°); 2-methylcyclohexanone and sucrose, or sorbitol (liquid at 100°); 4-chlorocyclohexanone and trihydroxydimethoxyhexane (reduced cellulose Me ether). C. H.

**Production of carbonyl compounds [ketones and aldehydes].** H. T. BÖHME A.-G. (B.P. 385,551, 17.3.32. Ger., 14.11.31. Addn. to B.P. 382,929; B., 1933, 139).—Aldehydes are obtained from  $(HCO_2)_2Ca$  and salts of fatty acids above  $C_1$  with other metals, and ketones from mixtures of fatty acid salts of metals, by heating at 300° or higher in a solvent under pressure. Examples are: Na stearate and  $(HCO_2)_2Ca$  in cyclohexane; Ca salts of coconut oil acids and  $Ca(OAc)_2$  in  $C_6H_6$ . C. H.

**Production of alcohols.** H. T. BÖHME A.-G. (B.P. 385,488, 17.3.32. Ger., 14.11.31. Addn. to B.P. 381,476; B., 1933, 11).—The process of B.P. 385,551 (preceding abstract) is combined with a simultaneous hydrogenation in presence of Cu catalyst. Octadecyl alcohol is obtained in 97% yield from Na stearate  $(HCO_2)_2Ca$ ,  $CuCO_3$  on kieselguhr, and  $H_2$  at 300°/200 atm. C. H.

**Production of polyvinyl halides of high mol. wt.** I. G. FARBENIND. A.-G. (B.P. 385,004, 22.6.31. Ger., 8.8.30).—Polymerisation by peroxides in absence of diluents is interrupted before complete, e.g., after 50% conversion, and the unpolymerised product is distilled off, leaving a uniform polymericide. C. H.

**Manufacture and recovery of glutamic acid and its compounds.** LAROWE-SUZUKI Co. (B.P. 385,054, 15.10.31. U.S., 29.4.31).—The starting material (vegetable proteins, gluten, casein, beet-molasses residues, etc.) is hydrolysed with 3—7% NaOH, preferably at 85—95°, and cooled rapidly to prevent racemisation. The liquor is made acid (3.5% HCl) with conc. HCl, and evaporated to *d* 1.48 at 65°. HCl is again added (to  $p_H$  2.1) and, after cooling, the cryst. inorg. salts are removed. NaOH is then added (to  $p_H$  3.2), whereupon *d*-glutamic acid crystallises out. C. H.

**Manufacture of amides of higher fatty acids.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 384,665, 4.6.31).—Natural or synthetic glycerides of higher fatty acids are treated with  $NH_3$  or a primary or *sec.* amine (except purely aromatic amines) in absence of  $H_2O$  at 100—200°. An org. solvent may be present provided no substantial amount of unchanged amine remains dissolved therein after reaction. Examples are amides from  $NH_3$  and olive oil, beef tallow, castor oil, or coconut oil; cyclohexylamine and castor oil;  $\beta$ -hydroxyethylamine and olive or coconut oil. Application in the paper, textile, soap, leather, and perfume industries etc. is indicated. C. H.

**Manufacture of sulphonated nitrogenous derivatives of higher paraffin hydrocarbons [emulsifying agents].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 383,736, 3.11.31).—The nitrogenous derivatives obtained according to B.P. 339,962 (B., 1931, 194) are treated with a sulphonating agent (100%  $H_2SO_4$  or  $ClSO_3H$ ) in presence or absence of diluent ( $C_2HCl_3$ ). C. H.

**Manufacture of readily soluble derivatives of the pyridine series.** I. G. FARBENIND. A.-G. (B.P.



385,214, 10.6.32. Ger., 15.10.31).—Bromo- or iodo-hydroxy-pyridines or -pyridones carrying salt-forming groups are converted into their amine salts. Examples are  $\text{NH}_4\text{Et}_2$  salts of 3:5-dibromo- or 3:5-di-iodo-2(or 4)-pyridone-1-acetic acid, and 3:5-di-iodochelidamic acid. The salts are suitable as contrast media in X-ray examinations. C. H.

**Production of mesityl oxide and its homologues from acetone and its homologues.** BRIT. INDUSTRIAL SOLVENTS, LTD. From DEUTS. GOLD- U. SILBERSCHEIDENSTALT VORM. ROESSLER (B.P. 383,474, 21.1.32).—A mixture of  $\text{COMe}_2$  and diacetone alcohol, or of corresponding homologues, is heated with dil. acid. Thus,  $\text{COMe}_2$  or  $\text{COMeEt}$  is first condensed in presence of soda-lime to a mixture of ketone and about 10% ketone-alcohol, very dil.  $\text{H}_2\text{SO}_4$  is then added, and the whole charged into 33% aq.  $\text{H}_2\text{C}_2\text{O}_4$  at  $120^\circ$ , the ketone vapours from the reaction vessel being returned to the soda-lime tubes; mesityl oxide or  $\gamma$ -methyl- $\Delta^7$ -hepten- $\alpha$ -one is separated in 85–95% yield. C. H.

**Manufacture of organic sulpho[nyl] chlorides.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 384,722, 14.9.31).—Salts of org. sulphonic acids are heated with a benzotrichloride at  $150$ – $200^\circ$ , with or without org. solvent. Sulphonyl chlorides are thus obtained from Na 2-naphthalene- (m.p.  $66^\circ$ ), *p*-toluene- (m.p.  $69^\circ$ ), methane- (b.p.  $161$ – $166^\circ$ ), cyclohexane- (b.p.  $125^\circ/13$  mm.), pyridine-2-, and *m*-nitrobenzene-sulphonates. C. H.

**Sterilisation of organic substances.** E. SCHULZE and PHARMACEUT. CORP., LTD. (B.P. 385,323, 12.6.31).—Org. substances are sterilised at  $15^\circ$  or lower by addition of the homologues of quinatoxines (cf. B.P. 339,602; B., 1931, 366) or of hydrocupreine, in presence of aromatic acids other than  $\text{BzOH}$ , e.g., *p*-hydroxybenzoic, salicylic, or cinnamic acid, the proportions being 1 pt. of the quinatoxine etc., 0.2–0.05% of acid, and 5000–80,000 pts. of substance to be sterilised. C. H.

**[Manufacture of] phthalic anhydride.** W. S. CALCOTT and N. C. SOMERS, ASSTS. to E. I. DU PONT DE NEMOURS & Co. (B.P. 1,854,882, 19.4.32. Appl., 11.5.29).—Decarboxylation and other side reactions in the catalytic oxidation of  $\text{C}_{10}\text{H}_8$  are avoided by excluding from the catalyst chamber metals which oxidise and/or form phthalates. The chamber may be constructed of Ni, Al, Cr, Ag, Ni-Cr-Fe, Cr-Fe, Fe-Si, or stainless steels. C. H.

**Manufacture of aromatic [hydr]oxycarboxylic acids.** Dr. A. WACKER GES. F. ELEKTROCHEM. IND., G.M.B.H. (B.P. 384,619, 18.8.32. Ger., 9.9.31).—Carboxylation is effected in presence of a solvent, preferably the free phenol. C. H.

**Purification and decolorisation of aromatic hydroxycarboxylic acids.** MONSANTO CHEM. WORKS, LTD., and S. SMITH (B.P. 384,558, 17.5.32).—Coloured impurities are removed from salicylic and cresotic acids etc. by adding a pptd. oxide, hydroxide, or carbonate of Zn or Al (which may be produced *in situ*) to an aq. solution of a sol. salt of the crude acid. C. H.

**Production of arylmercapto-compounds [hydroxythiophenols].** IMPERIAL CHEM. INDUSTRIES,

LTD., and K. W. PALMER (B.P. 383,284, 31.7.31).— $\text{PhONa}$  or  $\alpha$ - or  $\beta$ - $\text{C}_{10}\text{H}_7\cdot\text{ONa}$  or homologues are heated with  $\text{Na}_2\text{S}_2$ , e.g., at  $170$ – $180^\circ$ , with or without  $\text{Na}_2\text{S}$ , whereby  $\cdot\text{SNa}$  enters *ortho* to the  $\cdot\text{ONa}$  group. 2-Hydroxythiophenol, 2-hydroxythionaphthol, m.p.  $56$ – $60^\circ$ , the 1:2-isomeride, m.p.  $15^\circ$  (disulphide, m.p.  $147$ – $148^\circ$ ), and a dithiophenol are described. C. H.

**Manufacture of 2-chloroarylothiazoles.** GOODYEAR TIRE & RUBBER Co., Assees. of A. M. CLIFFORD (B.P. 382,940, 30.7.31. U.S., 19.11.30).—A 2-thiolarlylothiazole is converted into the 2-Cl-compound by  $\text{S}_2\text{Cl}_2$  at about  $150^\circ$  in absence of solvents. 2-Chlorobenzthiazole has b.p.  $160$ – $165^\circ/65$ – $70$  mm. (Cf. B.P. 277,338; B., 1928, 361.) C. H.

**[Manufacture of] intermediate products and azo dyes therefrom [ice colours].** IMPERIAL CHEM. INDUSTRIES, LTD., and R. F. GOLDSTEIN (B.P. 384,317, 29.5.31).—Bisacetoacetyl derivatives of 4:4'-diamino- and 2:2'-dichloro-4:4'-diamino-stilbenes are prepared ( $\text{Cl}_2$ -compound, m.p.  $187^\circ$ ), and are coupled on the fibre with diazo compounds of the  $\text{C}_6\text{H}_6$  series free from azo,  $\text{SO}_3\text{H}$ ,  $\text{CO}_2\text{H}$ ,  $\text{CN}$ , and  $\text{NO}_2$  groups (e.g., diazotised 6-chloro-*o*-toluidine, *o*-chloroaniline, 4-chloro-*o*-anisidine, 2:5-dichloroaniline) to give yellow shades fast to kier-boiling. C. H.

**Manufacture of chloro- and bromo-derivatives of 2-aminonaphthalene-1-sulphonic [Tobias] acid.** I. G. FARBENIND. A.-G. (B.P. 385,219, 15.6.32. Ger., 15.6.31).— $\beta$ -Naphthol-1-sulphonic acid carrying Cl or Br in 5-, 6-, 7-, or 8-position is aminated, e.g., with 25% aq.  $\text{NH}_3$ , at  $130$ – $170^\circ$  in presence of sulphite. 6-Cl-, 7-Cl-, and 7-Br-derivatives are described. C. H.

**Isolation of 1:2- and 2:3-diaminoanthraquinone sulphates.** I. GUBELMANN and J. M. TINKER (B.P. 383,625, 6.5.31).—The 2:3-diamine, m.p.  $368^\circ$ , separates from cold 83%  $\text{H}_2\text{SO}_4$ , the 1:2-diamine, m.p.  $298^\circ$ , on further dilution to 67%. C. H.

**Manufacture of anthraquinone derivatives.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 383,737, 6.11.31).—Chlorocyananthraquinones are obtained by treating cyananthraquinonesulphonic acids or their salts with  $\text{Cl}_2$  in aq. medium above  $80^\circ$ , preferably in presence of acid. Examples are the prep. of 2-chloro-1-cyano- (m.p.  $284$ – $285^\circ$ ), 2-chloro-6-cyano- (m.p.  $304$ – $306^\circ$ ), 2-chloro-3-cyano- (m.p.  $284^\circ$ ), and 2:6-dichloro-1:5-dicyano-anthraquinones. C. H.

**Benanthrone derivatives.**—See IV.

#### IV.—DYESTUFFS.

$\text{Al}_2\text{O}_3$  lakes.—See XIII.

##### PATENTS.

**Manufacture of cyanine dyes and their application in photography. Sensitising of photographic emulsions.** KODAK, LTD., Assees. of L. G. S. BROOKER (B.P. 385,320 and 385,332, 11.6.31. U.S., 11.6.30).—(A) Quaternary salts of 2-methylthiazoline are condensed with quaternary salts of quinoline or of a 2-halogenoquinoline, or with an alkyl orthoformate or other ortho-ester, or with a dialkylaminobenzaldehyde. Examples include the condensation of 2-methylthiazoline meth- and eth-iodides with meth- or eth-iodides of

quinoline, 6- or 8-methyl-, or 2-iodo-quinoline; or the metho-*p*-toluenesulphonate with  $\text{CH}(\text{OEt})_3$ ,  $\text{CMe}(\text{OMe})_3$ ,  $\text{CEt}(\text{OMe})_3$ ; and the methiodide with *p*-dimethylamino-benzaldehyde. (b) The products sensitise in the region 360—520  $\mu$ . C. H.

**Manufacture of yellow mordant dyes.** W. W. GROVES. From DURAND & HUGUENIN A.-G. (B.P. 384,901, 12.5.32).—4- or 3-amino-1:8-naphthalic acid or anhydride (preferably sulphonated) is condensed with an aminosalicyclic acid; the dyes resist discharge with hyposulphite. Examples are: 4-amino-6-sulphonaphthalic acid with 3- or 5-aminosalicyclic or 6-amino-*m*-cresotic acid; 4-aminonaphthalic acid with 5-amino-3-sulphosalicylic acid. A nitronephthalic acid may be used and the product reduced. C. H.

**Manufacture of dyes of the isorosinduline and rosinduline series.** E. I. DU PONT DE NEMOURS & Co. (B.P. 384,709, 17.8.31, U.S., 15.8.30).—*p*-Nitrosophenylmorpholine is condensed with an alkyl- or aryl- $\beta$ -naphthylamine, which may carry a 6- or 7-monoalkyl- or monoaryl-amino-group. The resulting isorosindulines may be converted into rosindulines by oxidation in presence of  $\text{NH}_3$  or a primary or *sec.* amine, and may subsequently be sulphonated. In the examples, phenyl-, *p*-tolyl-, ethyl-, and *p*-phenetyl- $\beta$ -naphthylamines and 2:7-dianilinonaphthalene are used, and for the rosindulines 2:4-tolylendiamine, *p*-amino-diphenylamine and -dimethylaniline. C. H.

**Manufacture of [thio]indigoid vat dyes.** I. G. FARBENIND. A.-G. (B.P. 383,662, 4.8.31. Ger., 1.8.30).—A 7-alkoxy- $\alpha$ -naphthathioindoxyl is condensed with a thioisatin chloride (etc.), or a 7-alkoxy- $\alpha$ -naphthathioisatin chloride (etc.) with a thioindoxyl. Examples are: 7-methoxy- $\alpha$ -naphthathioindoxyl, m.p. 173°, with thioisatin *p*-dimethylaminoanil (red-brown) or with  $\beta$ -naphthathioisatin naphthylimide (olive-brown), or with monobrominated  $\beta$ -naphthathioisatin chloride (olive). C. H.

**Production of dyes and intermediates of the benzanthrone series.** D. C. R. JONES, R. F. THOMSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 384,999, 16.6.31).—The alkali-sol. products obtainable from *o*-halogenobenzoic acids and benzanthrone (see B.P. 358,426; B., 1932, 175) are extracted with  $\text{AcOH}$ ,  $\text{C}_6\text{H}_6$ , or other solvent for benzanthrone, and the filtrate is diluted with a suitable non-solvent to ppt. the benzanthrone derivative. Extraction with  $\text{AcOH}$  leaves a violet residue and the filtrate gives on dilution a green ppt., of a benzanthranylbenzoic acid which is converted by condensing agents into a dibenzpyrenequinone. C. H.

**Manufacture of dyes of the anthraquinone series.** I. G. FARBENIND. A.-G. (B.P. 385,202, 20.5.32. Ger., 20.5.31).—A 1-amino- or -alkylamino-4-aminoanilinoanthraquinone is condensed with an olefine halohydrin in presence of an acid-binding agent and preferably in a solvent. Examples are: ethylene chlorohydrin with 1-amino-4-*p*-aminoanilinoanthraquinone (steel-blue), the 2-Me derivative (grey-blue), and the 1-NHMe compound (green). C. H.

**Manufacture of acid dyes of the anthraquinone series.** I. G. FARBENIND. A.-G. (B.P. 383,871, 18.4.32. Ger., 16.4.31).—A 4-halogeno-1-aminoanthraquinone-2-

sulphonic acid is condensed with an aminobenzoic amide or derivative thereof, especially  $\text{NH}_2 \cdot \text{C}_6\text{H}_4 \cdot \text{CO} \cdot \text{NRR}'$  in which R is H, alkyl, hydroxyalkyl, or cyclohexyl and R' is H, or R and R' are both alkyl. In the examples the  $\text{NH}_2$  is *o*-, *m*-, or *p*-, and R and R' are H and H, H and cyclohexyl, Me and Me, H and Me, or H and  $\text{C}_2\text{H}_4\text{OH}$ . The products give blue to violet-blue shades on wool. C. H.

**Production of dyes of the anthraquinone [dibenzanthrone] series.** IMPERIAL CHEM. INDUSTRIES, LTD., C. SHAW, and R. F. THOMSON (B.P. 383,624, 16.4.31).—A hydroxy-, alkoxy-, or acyloxy-derivative of dibenzanthrone or isodibenzanthrone, which may also contain halogen, is heated above 270° with an *o*-dicarboxylic anhydride. Examples are: 2:2'-dimethoxydibenzanthrone with phthalic anhydride (blue-green), 3:4-dichlorophthalic anhydride (blue) or naphthalene-2:3-dicarboxylic anhydride; 2:2'-dihydroxydibenzanthrone with tetrachlorophthalic anhydride (blue-grey); chlorinated in phthalic anhydride at 180°, (grey); oxidised isodibenzanthrone (violet) or 2:2'-diacetoxy- (navy-blue) or brominated 2:2'-dimethoxy- (blue) dibenzanthrone with tetrachlorophthalic anhydride. C. H.

**Manufacture of vat dyes of the pyranthone series.** I. G. FARBENIND. A.-G. (B.P. 382,877, 18.4.32. Ger., 17.4.31).—Pyrene is condensed with an aromatic monocarboxylic acid chloride or anhydride having a free *ortho*-position in presence of  $\text{AlCl}_3$  and a dehydrogenating agent (air,  $\text{O}_2$ ,  $\text{MnO}_2$ ), and preferably a fluxing agent. Direct conversion into pyranthones occurs with 50—60% yield. Condensations with  $\text{BzCl}$  or  $\text{Bz}_2\text{O}$  (orange), *m*- $\text{C}_6\text{H}_4\text{Cl} \cdot \text{COCl}$  (orange), *p*- $\text{C}_6\text{H}_4\text{Me} \cdot \text{COCl}$  (orange), and  $\alpha$ - $\text{C}_{10}\text{H}_7 \cdot \text{COCl}$  (brown-orange) are described. C. H.

**Manufacture of products [vat dyes] of the anthrapyrimidine series.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 385,295, 18.5. and 7.12.31, 17.3.32).—Various methods for the production of acyl-aminoanthrapyrimidines are described; the products may be halogenated. Examples include: 1-amino-4-benzamidoanthraquinone condensed with  $\text{HCO} \cdot \text{NH}_2$  (green-yellow on wool from ammoniacal vat); 6-aminoanthrapyrimidine with 1-aminoanthraquinone-2-carboxylic chloride (red on cotton) or 1-(*p*-chlorocarbonylanilino)anthraquinone (red-orange); 8-methylaminoanthrapyrimidine with *p*-chlorobenzoyl chloride (corinth); 4-amino-2-phenylanthrapyrimidine with anthraquinone-2-carboxyl chloride (green-yellow); monochlorinated 6-aminoanthrapyrimidine with  $\text{NH}_2\text{Bz}$  (yellow); 6-chloro-2-phenyl-4-methylanthrapyrimidine with *p*-toluenesulphonamide (yellow); 4:4'-dianthrapyrimidyl, mononitrated, reduced, benzoylated (yellow); chlorinated 8-chloroanthrapyrimidine, converted into diamine with  $\text{NH}_3$ , dibenzoylated (orange); 6-benzamidoanthrapyrimidine with  $\text{PCl}_5$  to give the 2-Cl-compound (green-yellow); 8-benzamidoanthrapyrimidine, chlorinated (gold-orange). C. H.

**Manufacture of vat dyes [of the anthraquinone-acridone and -thioxanthone series].** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 384,311, 29.4.31).—An anthraquinone-acridone (or -thioxanthone)-carboxylic

acid is condensed with an *o*-amino-phenol or -thiophenol or an *o*-chloro- or *o*-nitro-arylamine to give cyclic compounds. Examples include: anthraquinone- $\beta$ -thioxanthone-2- or -3-carboxyl chloride with 2-amino-1-thiolanthraquinone (green-yellow); the 1-carboxyl chloride with 2-amino-1-hydroxyanthraquinone (yellow); anthraquinone-1 : 2 : 2' : 1'-naphthacridone-3'-carboxyl chloride with 2-amino-1-hydroxyanthraquinone (red-orange). C. H.

**Dyes and dyeing.** D. A. W. FAIRWEATHER, R. F. THOMSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 385,307, 19.6.31. Cf. B.P. 333,506; B., 1930, 1024).—1-Aryldiazosulphonates of 2 : 3-hydroxynaphthoic arylamides,  $R \cdot SO_2 \cdot N \cdot NAr'$ , are obtained by treating a 2-hydroxy-1-sulpho-3-naphthoic arylamide with a diazo compound in acid or neutral solution. These are converted by alkali ( $Na_2CO_3$ ) into 1-arylo-1-sulpho-2-keto-1 : 2-dihydro-3-naphthoic arylamides (I), which by treatment on the fibre with dil. acid yield the normal azo dyes by elimination of the  $SO_3Na$ . The products (I) may be dyed in conjunction with the compounds of B.P. 333,506 or with sulphuric esters of leuco-vat dyes. C. H.

**Production of azo dyes [for wool, cotton, leather, acetate silk].** A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 384,800, 10.12.31).—4-Nitro-*m*-phenylenediamine (I) gives a diazo compound which is coupled with suitable components. Examples are: (I)  $\rightarrow NH_2Ph$  (orange on acetate silk),  $NEt_2Ph$  (red), *p*-cresol (yellow-brown),  $\alpha-C_{10}H_7 \cdot NH_2$  (red; violet-black on development with 2 : 3-hydroxynaphthoic acid), *R*-acid (orange-brown on wool); *H*-acid  $\rightarrow$  resorcinol  $\leftarrow$  (I) (brown on leather); *p*-nitroaniline-2-sulphonic acid  $\rightarrow$  2 : 4-dihydroxybenzoic acid  $\leftarrow$  (I) (brown on chrome leather). C. H.

**Manufacture of azo dyes [green pigments and ice colours].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 383,967, 1.6.31).—An acylacetic arylamide is coupled in substance or on the fibre with a diazotised aminoindigoid obtainable by condensing a nitroisatin  $\alpha$ -chloride with a naphthol or anthrol and reducing the product. The reduced indigoid from 5-nitroisatin  $\alpha$ -chloride and 4-chloro- or 4-methoxy- $\alpha$ -naphthol is coupled, e.g., with acetoacetic tolidide or *o*-anisidide or with benzoylacetic anilide. C. H.

**Manufacture of azo dyes containing chromium.** SOC. CHEM. IND. IN BASLE (B.P. 384,767, 12.11.31. Switz., 14.11.30. Addn. to B.P. 364,147; B., 1932, 498).—The azo dyes of the prior patent are mixed with similar or different chromable azo dyes before chroming; or the Cr may be supplied already combined with one of the dyes. Examples are mixtures of: 1 : 2 : 4-aminonaphtholsulphonic acid  $\rightarrow$   $\beta$ -naphthol and  $\alpha$ -naphthol with 4-nitro-*o*-aminophenol-6-sulphonic acid  $\rightarrow$  1-phenyl-3-methyl-5-pyrazolone (prechromed, black on wool). C. H.

**Production of conversion products of azo dyes.** J. R. GEIGY SOC. ANON. (J. R. GEIGY A.-G.) (B.P. 385,471, 26.2.32. Ger., 28.2.31. Addn. to B.P. 306,447; B., 1930, 316).—The process of the prior patent is conducted in presence of a heavy metal (Fe, Cu) and/or a compound thereof (except  $MnO_2$ ). C. H.

**Manufacture of black trisazo dyes.** J. R. GEIGY A.-G. (B.P. 384,111, 15.1.32. Ger., 16.1.31).—Black shades on cotton, viscose, wool, silk, and unions are obtained with dyes of the type:  $NH_2Ar \rightarrow H$ -acid  $\leftarrow$  benzidine  $\rightarrow$  2 : 6- or 2 : 7-aminonaphthol or -naphthylenediamine, where Ar is aryl (Ph, *o*- or *p*- $C_6H_4Cl$ , *o*- or *p*- $C_6H_4Me$ ), and tolidide or dianisidine may replace the benzidine. C. H.

**Azo dyes.**—See III.

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

**Chemico-technological tests of cotton fibre at various stages of maturity.** G. A. KORSHENOVSKI and E. Z. PLIUSHKIN (Trud. Sredneziat. Nauch. Issledov. Inst. Khlop., 1931, Bull. No. 50, 81—100).—Immature cotton loses 98% and mature cotton 81% of its N when boiled with 1% NaOH. Young fibre absorbs more  $H_2O$  than fibre from mature open bolls. Dyes are fixed by young fibres as well as by old, especially after mercerisation. CH. ABS.

**Composition of cotton fibres of various stages of maturity.** V. T. IVANOVA and A. M. KURENOVA (Trud. Sredneziat. Nauch. Issledov. Inst. Khlop., 1931, Bull. No. 50, 57—71).—Cellulose accumulates rapidly up to 40 days, reaching 83% at 45 days. The total and protein-N decrease with age up to 50 days. Of the N, 65—75% is org., and the remainder nitrate and nitrite.  $P_2O_5$  decreases with maturity. The pentosan content decreases with age, becoming const. at 40 days. Cotton at 50 days is as good for industrial purposes as mature cotton. CH. ABS.

**Microscopical investigation of cotton fibres at various stages of maturity.** A. P. ZAKOSHCHIKOV, G. A. KORSHENOVSKI, and M. G. RUITKOV (Trud. Sredneziat. Nauch. Issledov. Inst. Khlop., 1931, Bull. No. 50, 15—43).—Cotton fibre at 40 days has the same structure as fibre from mature plants; it responds similarly to mercerising and has the same swelling coeff. in Schweitzer's reagent and in 80%  $H_2SO_4$ . Only 15 days after blooming, cellulose appears in cotton; as the cellulose increases, proteins decrease. The cuticle differs in composition from the cellulose of the fibre, but on hydrolysis with  $ZnCl_2$  the products are similar, a blue coloration with I being obtained. Tannins and starch were not detected in the fibres. Cotton fibre has the same industrial val., 35—40 days before the bolls open as after maturity. CH. ABS.

**Ash constituents of cotton fibres at various stages of maturity.** B. I. ARDASHEV and B. I. LEONOV (Trud. Sredneziat. Nauch. Issledov. Inst. Khlop., 1931, Bull. No. 50, 72—80).—With ripening the ash content (particularly alkalis and alkaline earths) decreases; the Ca content of the ash increases. Navrotzk cotton contains more Ca and S than American cotton. CH. ABS.

**Hygroscopic moisture of cellulose.** IX. **Hygroscopic moisture of silk and wool.** S. OGURI (J. Soc. Chem. Ind., Japan, 1932, 35, 478—481B; cf. B., 1932, 880).—The equilibrium moisture of silk and wool were determined and from these the heats of sorption were calc. These approach a max. at 6—8%  $H_2O$  and equal the heat of condensation of  $H_2O$  vapour at low or high  $H_2O$  content. V. E. Y.

**Fermentation of cellulose by thermophilic bacteria. I. Preliminary. II. Properties of the attacked cellulose fibres compared with the original. III. Fermentation tests of various cellulosic materials.**

Y. TOMODA (J. Soc. Chem. Ind., Japan, 1932, 35, 534—536B).—I. The thermophilic cellulose-decomposing bacteria were isolated as mobile long rods, thought to belong to the *Clostridium thermocellum* species (cf. A., 1926, 1278). They decompose filter-paper in 48 hr. at 65°. The natural twist of the fibre is first disentangled and the fibre gradually disintegrated until it finally becomes powdery. The velocity of fermentation of cellulose is slow for the first day, then const. for 4 days, gradually falling off over 15—20 days.

II. Filter-paper was fermented by the above type of bacteria at 65° for 1—14 days, the progress of decomp. being followed by determinations of the Cu no., viscosity in cuprammonium, and  $\alpha$ -,  $\beta$ -, and  $\gamma$ -cellulose contents; no difference was observed between the attacked cellulose and the original fibre in respect of these. Bacterial action results in neither chemical nor colloidal decomp., and is quite different from that due to acid hydrolysis.

III. Various celluloses were fermented at 65° for 3—7 days as before. Natural cellulose fibres, cotton wool, hemp, or wood cellulose, are more easily fermented than regenerated and dried cellulosic materials. The following order of fermentability of various materials is given: regenerated cellulose > standard pure cellulose from cotton wool = raw cotton wool = commercial refined cotton wool = filter-paper = tissue paper = hemp fibre = sulphite wood pulp (all ferment easily) > mercerised cotton wool = cuprammonium rayon > viscose rayon > nitro-rayon = cellophane = acetate rayon = nitro-cellulose (these last do not ferment at all). V. E. Y.

**Viscosity variations in viscose solutions. Influence of electrolyte.** T. NAKASHIMA and S. SAOTOME (J. Soc. Chem. Ind., Japan, 1932, 35, 453—455B).—The influence of NaOH on the viscosity of cellulose xanthate solutions was investigated. The relative viscosity was determined by the falling-sphere method for 4 solvents and for cellulose concns. varying from 5 to 8 g. per 100 c.c. In all cases reasonable agreement with calc. vals. was obtained. V. E. Y.

**Viscosity of cellulose esters and acetylcellulose.** I. SAKURADA (J. Soc. Chem. Ind., Japan, 1932, 35, 444—445B).—A reply to Atsuki and co-workers (B., 1932, 416). V. E. Y.

**[Viscosity of cellulose esters and acetylcellulose.]** K. ATSUKI and M. ISHIWARA (J. Soc. Chem. Ind., Japan, 1932, 35, 445B).—A rejoinder (cf. preceding abstract). V. E. Y.

**Spinning of viscose silk. X. Composition of the coagulating bath in contact with a fine stream of viscose. XI. Theory of coagulation.** S. HASE (J. Soc. Chem. Ind., Japan, 1932, 35, 458—459B; cf. B., 1933, 13).—X. The relation between the composition of the bath and that of the liquor adhering to the spun thread was investigated. For bottom-spinning this varies with the bath used, and the concn. of the adhering liquor increases with the no. of filaments and with the winding tension. For centrifugal spinning the concn. decreases with increase in denier of the thread, but increases with

spinning speed. The coagulating liquor suffers considerable change when it meets the viscose stream, but gradually returns to the initial composition as the viscose gels.

XI. Viscose spun into  $H_2SO_4$  and sulphate changes instantaneously to viscose gel by neutralisation, dehydration, and decomp., the subsequent transition of the gel to cellulose proceeding from the periphery. The dehydrating effect of sulphates increases in the order  $Mg < Na < NH_4 < Zn$ .  $ZnSO_4$  differs from others in that Zn cellulose xanthate, Zn thiocarbonate, and ZnS are formed around the viscose stream. The change of viscose into silk is shown diagrammatically. V. E. Y.

**Practical factors in the permanence of sulphite papers.** J. STRACHAN (Paper-Maker, 1933, 85, 33—34ts).—Manufacturing conditions which affect permanence are reviewed. An easy bleaching Mitscherlich pulp consuming about 3% of  $Cl_2$  is recommended as the chief portion of the furnish. Bleaching should be carried out slowly in the cold, and no anti-chlor added. Efficient washing is essential, beating must not be excessive, and the amount of  $Al_2(SO_4)_3$  used in sizing should be kept at a min.; a high free-rosin size is helpful in this respect. Max. drying temp. should be approached gradually, and should not exceed 85°. H. A. H.

**Action of ultra-violet light on gelatin in paper.** H. A. BROMLEY (Analyst, 1933, 58, 29).—The gelatin was partly hydrolysed, but no degradation products could be separated. T. McL.

**Materials used in the manufacture of tracing paper.** H. L. VINCENT (Paper Trade J., 1932, 95; T.A.P.P.I. Sect., 304—306).—The various advantages and defects of tracing papers prepared by impregnation with vegetable oils, mineral oils, waxes, abietic esters, and those from heavy beaten pulp, are described. No entirely satisfactory tracing paper is yet available. H. A. H.

**Improved wax test for measuring coated paper sizing.** H. M. ANNIS (Paper Trade J., 1933, 96; T.A.P.P.I. Sect., 20—22).—The sizing degree of coated papers is a measure of the adhesion between the coating and the body paper. By the use of a series of specially prepared waxes, applied to the coated surface under standardised conditions, it is possible to determine accurately the sizing degree, and so the behaviour of the paper on the printing presses, both as regards plucking and ink receptivity. R.H. and temp. must be controlled during the test. By means of this test the relative strengths of the bonds between fibre and fibre, fibre and coating, and adjacent particles of coating material can be discriminated. With papers coated on one side only, the wire side is the harder sized. H. A. H.

**Probable causes of variable sizing in paper-making.** J. McLAREN (Paper-Maker, 1932, 84, 28—29ts; 1933, 85, 35—37ts).—Factors affecting sizing are reviewed. H. A. H.

**Sewage plant for rayon wastes.**—See XXIII.

PATENTS.

**Apparatus for making artificial films or skins of cellulose and cellulose derivatives.** J. P. BEMBERG A.-G. (B.P. 385,783, 22.2.32. Ger., 20.2.31).—

The film passes over two positively-driven forwarding rollers (*A*, *B*) between which is a guiding member (*C*) which is displaceable in a vertical plane by the film without any appreciable stressing. A control member connected with *C* raises or lowers the speed of *A* or *B* corresponding to the changes of length of the film between *A* and *B* due to irregular shrinkage during pptn. or after-treatment. F. R. E.

**Production of artificial filaments and other products.** BRIT. CELANESE, LTD., H. DREYFUS, and W. I. TAYLOR (B.P. 385,617, 25.6.31).—A solution containing  $> 25\%$  of an org. derivative of cellulose, *e.g.*, the acetate, containing a liquid increasing its viscosity (a non-solvent ester, alcohol, aromatic hydrocarbon, or  $H_2O$ ), or a solution of a high-viscosity org. derivative of cellulose, is dry-spun at  $< 180$  m./min. into an evaporative atm. maintained at a temp.  $<$  that of the spinning solution. F. R. E.

**Manufacture of artificial silk.** S. YAMAMOTO (B.P. 385,516, 23.5.32).—An alkyl ester of an aliphatic or inorg. acid (EtOAc, MeCl, etc.) is added to known solutions of silk fibroin or other albuminous materials, *e.g.*, in alkaline Cu or Ni, in order to keep them thick and viscous, before spinning into salt or acid solution. F. R. E.

**Preparation of hollow artificial silk of low lustre.** RUTH-ALDO Co., INC. (B.P. 385,673, 15.9.31. U.S., 15.9.30).—A solution of a cellulose ester in a volatile org. solvent, *e.g.*,  $CO_2$ , containing 5–20% of the non-solvent ( $Pr_2O$ ) is dry-spun into a heated atm. F. R. E.

**Increasing the extensibility of artificial threads, especially artificial silk made from cellulose derivatives such as cellulose acetate.** ALGEM. KUNSTZIJDE UNIE N.V. (B.P. 383,028, 20.10.31. Ger., 8.11.30).—The dry and wet tensile strengths and extensibilities of acetate silk made by a stretch process are improved by treating the fully formed threads while wound on a frame (for prevention of shrinkage) with a swelling liquor (*e.g.*, a 1:3 mixture of light petroleum and  $CH_2Cl_2$ ), washing with  $H_2O$ , and drying. A. J. H.

[Device for tying up] artificial silk spun cakes and the like. ALGEM. KUNSTZIJDE UNIE N.V. (B.P. 385,911, 5.9.32. Ger., 18.9.31).

**Fatty acid amides.**—See III.  $Na_2SO_4$  from acid solutions.—See VII.

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

**Dyeing [wool fabric in] two-tone effects.** T. EGGER (Textilber., 1933, 14, 26–27).—Chlorinated wool yarn is mordanted with Cr salts and then woven into fabric with untreated fabric which is then dyed for  $\frac{1}{2}$ – $\frac{3}{4}$  hr. in a boiling bath containing 1–2% of  $H_2SO_4$  and Neolan dyes; especially fast-to-light dyeings are thus obtained. A. J. H.

**Influence of certain vat dyestuffs on the tendering of cellulose when exposed.** C. M. WHITTAKER (J. Soc. Dyers and Col., 1933, 49, 9–17).—Open-air exposure has a greater deteriorating effect on cotton and viscose-rayon yarns than has exposure under glass. Impure or industrial atm. are more harmful than sub-

urban atm., whilst pure country or seaside atm. cause least degradation. The order of relative catalytic effect of different vat dyes in the deterioration of cellulose is practically the same wherever the yarns are exposed. Certain vat dyes tend to inhibit the tendering, and no case is recorded of tendering of blue casement curtains. The extent of deterioration appears to increase with increasing concn. of dye on the material. Anti-oxidants such as  $CS(NH_2)_2$  and  $NaPO_3$  do not inhibit the tendering. Cotton is not appreciably superior to viscose rayon and, when dyed with safe vat colours, the latter material is eminently suitable for casement fabrics. B. P. R.

**Textile-aid industry.**—See XII.

### PATENTS.

**Kier [for fabrics].** E. D. JEFFERSON, ASST. to RODNEY HUNT MACHINE Co. (U.S.P. 1,853,977, 12.4.32. Appl., 2.5.30).—Intermittent upward circulation of the liquor in a modified form of the usual type of vertical kier is obtained by periodically supplying steam to a coil in the bottom of the kier; the heated liquor rises to the top of the kier and is then returned to the bottom by a suction device. A. J. H.

**Increasing the affinity of cellulose esters for dyes.** LONZA ELEKTRIZITÄTWERKE U. CHEM. FABR. A.-G., Assees. of G. R. LEVI and G. BENAGLIA (B.P. 384,403, 24.10.31. It., 24.10.30).—Cellulose is esterified with a nitrogenous carboxylic acid anhydride (cyanoacetic) or a mixed anhydride of AcOH and such an acid; the ester may be used alone or mixed with cellulose acetate etc. Any CN or  $CO \cdot NH_2$  groups present may be hydrolysed to increase further the affinity for direct dyes. C. H.

**Colouring [and parchmentisation of] fibrous materials.** R. J. HANNAY (B.P. 383,644, 29.5. and 23.6.31).—Cotton or other cellulose fabric is printed or sloop-padded with cold conc.  $H_2SO_4$  containing  $C_6H_4(OH)SO_3H$  (a retarding agent) and a fast vat or naphthol dye, and then washed free from acid after allowing sufficient time for parchmentisation; coloured linen-like transparent effects are thus obtained. A. J. H.

**Coloration of textile materials [discharge effects].** BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 384,313, 22.5.31).—A discharge agent suitable for use on acetate silk is prepared by reducing an aldehyde- or ketone-sulphoxylate with  $SnCl_2$ ,  $TiCl_3$ , or other metal salt which reduces by virtue of a valency change in the metal. C. H.

**Coloration of filaments, yarns, threads, and the like. [Effect dyeing of cellulose esters.]** BRIT. CELANESE, LTD. (B.P. 384,545, 6.5.32. U.S., 8.5.31. Addn. to B.P. 332,263; B., 1930, 944).—A hydrolysing agent (aq. NaOH) containing a dye, a mordant, or a coupling component is applied at intervals along a travelling cellulose ester (acetate) yarn, if desired, in the course of manufacture. Colour may then be applied to the mordanted material or the coupling component developed with a diazo-compound. The dye may be a leuco-vat dye or sulphuric ester thereof. Examples are hydrolysing baths containing Chlorazol-sky-blue FF,

dispersed Pontamine-fast-red F, leuco-Indanthrene-golden-yellow GK, and successive baths containing leuco-compounds of Ciba-scarlet, Indanthrene-printing-black B, and Indanthrene-golden-yellow GK. C. H.

**Manufacture of dyeings [ice colours].** SOC. CHEM. IND. IN BASLE (B.P. 383,998, 3.9.31. Switz., 3.9.30).—A sulphide vat dye of the Hydron-blue type is dyed on the fibre from the vat and coupled with a diazo-compound, *e.g.*, diazotised 5-nitro-*o*-anisidine, 2:5-dichloroaniline, 3-nitro-*p*-toluidine, aminoazotoluene, 4'-chloro-4-amino-5-methoxy-2-methylazobenzene, or tetrazotised 4:4'-diamino-5-methoxy-2-methylazobenzene. Black shades are obtained. [Stat. ref.] C. H.

**Production of improved dyeings.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 383,634, 4.5.31 and 3.2.32).—Dyes containing  $\text{CO}_2\text{H}$  or  $\text{SO}_3\text{H}$  groups are converted on the (cotton or regenerated cellulose) fibre into insol. salts by treatment with alkyl- or aralkylamines or  $-\text{NH}_4$  compounds above  $\text{C}_7$ , whereby the fastness to  $\text{H}_2\text{O}$  is improved. The amines may carry  $\text{CO}_2\text{H}$  or  $\text{SO}_3\text{H}$  groups. Examples include: Direct-deep-black EW extra with  $\text{C}_{17}\text{H}_{35}\cdot\text{NMe}_3\text{SO}_4$ ; Diamine-green B with  $\text{NEt}_2\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{NH}\cdot\text{C}_{17}\text{H}_{35}$ ; Congo-orange R with  $\text{C}_{12}\text{H}_{25}\cdot\text{NH}_2$ ; Benzo-fast-blue FR with  $\text{C}_{18}\text{H}_{37}\cdot\text{NH}\cdot\text{CH}_2\cdot\text{CO}_2\text{H}$ ; Congo-orange R with the sulphobenzoylation product of aminated hexochlorinated hard paraffin; Benzopurpurine 4B with product from anhydro-*p*-aminobenzyl alcohol and  $\text{CH}_2\text{Cl}\cdot\text{CH}_2\cdot\text{SO}_3\text{Na}$ . C. H.

**Transfer printing [of fabrics] and transfer pattern therefor.** P. YOHNS, Assr. to BUTTERICK PUBLISHING Co. (U.S.P. 1,849,996, 15.3.32. Appl., 10.11.25).—In preparing a transfer, paper or other suitable material is printed with a very thin layer of a coloured transfer medium (*e.g.*, a paste containing a basic dye, a neutral gum, glycerin, and dextrin) which retains permanently its solubility in the liquid solvent (*e.g.*, benzine, EtOH, used for effecting the transference) then dusted with a powder which may be a mordant but which during the transference prevents spreading of the colour and penetrates and envelops the threads of the fabric being decorated; transference is effected by forcing the liquid solvent through the fabric while in contact with the transfer. A. J. H.

**Mothproofing [of textiles].** W. J. MCGILL, Assr. to STANDARD OIL Co. (U.S.P. 1,854,948, 19.4.32. Appl., 7.11.27).—The impregnant is a solution of rotenone in  $\text{C}_6\text{H}_6$ , EtOH, or  $\text{Et}_2\text{O}$ . A. J. H.

**Fatty acid amides.**—See III. **Dyeing.**—See IV. **Coloured lacquers etc. Resin emulsions.**—See XIII.

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

**Rate of absorption of nitrous gases by sulphuric acid.** II. L. SZEGÖ and M. LOMBARDI (Giorn. Chim. Ind. Appl., 1932, 14, 492—496; cf. B., 1932, 304).—The rates of absorption of  $\text{N}_2\text{O}_3$  and  $\text{N}_2\text{O}_4$  in 0—95.6%  $\text{H}_2\text{SO}_4$  at room temp. have been determined. The rate is greater for  $\text{N}_2\text{O}_3$  in conc. (>23%) acid, and for  $\text{N}_2\text{O}_4$  in  $\text{H}_2\text{O}$  and dil. acid; the  $\text{N}_2\text{O}_3$  curve passes through a min. at about 17%, and the  $\text{N}_2\text{O}_4$  curve at about 80%.

The results are discussed, particularly in relation to the chamber process. H. F. G.

**Ammonia-soda process under high pressure of carbon dioxide.** N. KAMEYAMA and E. MUNAKATA (J. Soc. Chem. Ind., Japan, 1932, 35, 533—534B).—Aq. solutions, simultaneously saturated with  $\text{NH}_4\text{HCO}_3$ ,  $\text{NaHCO}_3$ , and  $\text{NH}_4\text{Cl}$ , were treated with  $\text{CO}_2$  at 20 and 40 atm. pressure and at 10° and 20°. The results show that no improvement in the conversion of NaCl can be expected by increasing the pressure of  $\text{CO}_2$ . At 10° the equilibrium solution is incongruently saturated with  $\text{NH}_4\text{HCO}_3$  at high pressures just as at atm. pressure, but at 20° it becomes congruently saturated with this salt under 20 or 40 atm.  $\text{CO}_2$ , although other investigators have shown that, at moderate pressures, there is incongruent saturation at 20°. M. S. B.

**Phosphorescence of zirconium oxide preparations.** K. HOLZINGER (Chem.-Ztg., 1932, 56, 1022).—Several commercial preps. of  $\text{ZrO}_2$  have been found to exhibit phosphorescence after exposure to light from the quartz-Hg-vapour lamp. The intensity of the effect falls to zero after 1 hr., but the material will again phosphoresce after exposure to sunlight or light from a filament lamp provided that such exposure occurs > 30 hr. after the exposure to the quartz lamp. Admixture of small quantities of  $\text{K}_2\text{SO}_4$ ,  $\text{Na}_2\text{SO}_4$ ,  $\text{TiNO}_3$ ,  $\text{Li}_2\text{CO}_3$ ,  $\text{CaSO}_4$ , and  $\text{Bi}(\text{NO}_3)_3$  increases, and of small quantities of  $\text{RbCl}$ ,  $\text{MnSO}_4$ , and  $\text{UO}_2(\text{NO}_3)_2$  reduces, the phosphorescence. A. R. P.

**KOH as fusion agent. Corrosion of steels by  $\text{H}_2\text{S}$ .**—See X. **Rapid test for Hg.**—See XX.

## PATENTS

**Manufacture of chlorosulphonic acid.** SOC. CHEM. IND. IN BASLE (B.P. 384,615, 8.8.32. Switz., 17.8.31).—Powdered dry NaCl is added to 62—70% oleum under reflux in proportions to give  $\text{NaHS}_2\text{O}_7$  or  $\text{Na}_2\text{S}_2\text{O}_7$ , and 1 or 2 mols. of  $\text{ClSO}_3\text{H}$ , which is distilled off. C. H.

**Production of phosphoric acid and calcium aluminate slag.** J. N. CAROTHERS and S. P. HUGER, Assrs. to SWANN RESEARCH, INC. (U.S.P. 1,853,406, 12.4.32. Appl., 28.2.27).—A mixture of bauxite, phosphate rock, coke, and scrap Fe is smelted in an electric furnace to produce ferrophosphorus and a fluid  $\text{CaO}-\text{Al}_2\text{O}_3-\text{SiO}_2$  slag having hydraulic properties and suitable for the manufacture of cement. The ferrophosphorus is subsequently oxidised to expel  $\text{P}_2\text{O}_5$ , which is converted into  $\text{H}_3\text{PO}_4$ . A. R. P.

**Manufacture of ammonia.** E. RABETRANO (B.P. 385,330, 20.6.31. Fr., 12.7.30).—A mixture of C and an oxide of Si, B, Ti, or V is heated at 1200—1500° in a strong current of  $\text{N}_2$  free from  $\text{O}_2$  and the product is decomposed by treatment with aq. acids, alkalis, or alkaline-earth hydroxides, or with  $\text{H}_2\text{O}$  vapour and  $\text{CO}_2$ , with the addition, if desired, of finely-divided Al, Zn, or Fe to facilitate the decomp. L. A. C.

**Removal of sodium sulphate from solutions containing sodium sulphate and sulphuric acid.** COURTAULDS, LTD., and H. J. HEGAN (B.P. 384,726, 19.9.31).—The solution is conc. until it contains

21—22% of  $\text{Na}_2\text{SO}_4$  and  $\text{H}_2\text{SO}_4$  is then added until > 30% (40%) is present. On cooling,  $\text{NaHSO}_4$  aq. crystallises. The process is applicable to the removal of  $\text{Na}_2\text{SO}_4$  from rayon spinning baths. A. R. P.

**Sodium hydrosulphite preparations.** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 384,903, 13.5.32).— $\text{Na}_2\text{S}_2\text{O}_4$  (1 mol.) is intimately mixed with at least 2 mols. of  $\text{NaOH}$ ,  $\text{KOH}$ ,  $\text{Na}_2\text{CO}_3$ ,  $\text{Na}_2\text{SiO}_3$ , or  $\text{Na}_3\text{PO}_4$ , which act as stabilisers. A. R. P.

**Manufacture of anhydrous alkali polysulphides.** I. G. FARBENIND. A.-G. (B.P. 385,895, 26.7.32. Ger., 5.9.31).—An anhyd. alkali sulphide prepared by the gaseous reduction of the sulphate is added to S at about 200° in quantity sufficient to yield the desired polysulphide. L. A. C.

**Production of sulphocyanide compounds.** E. HENE (B.P. 384,662, 22.5.31).—A mixture of  $\text{CaCN}_2$  with  $\text{CaS}$ ,  $\text{Ca}(\text{SH})_2$ , or  $\text{CaO}$  and  $\text{NaHS}$  is heated at 100—110° until the formation of  $\text{CS}(\text{NH}_2)_2$  is complete, whereupon the temp. is raised to 150—250° (180°) until  $\text{NH}_3$  ceases to be evolved and the  $\text{CS}(\text{NH}_2)_2$  is converted into  $\text{Ca}(\text{CNS})_2$  or  $\text{NaCNS}$ . If desired, the  $\text{CS}(\text{NH}_2)_2$  may first be separated and purified, then heated with  $\text{CaO}$  to obtain  $\text{Ca}(\text{CNS})_2$ . A. R. P.

**Preparation of titanium oxide.** J. BLUMENFELD (B.P. 385,315, 17.4.31).—Calcined  $\text{TiO}_2$  is ground wet in the presence of a dispersion agent, e.g.,  $\text{NaOH}$ ,  $\text{KOH}$ , aq.  $\text{NH}_3$ , or an alkali silicate, sufficient to produce a suspension having  $p_H$  9—12. The thick, milky liquid is decanted from the coarser particles and coagulated by neutralisation with  $\text{H}_2\text{SO}_4$  or by addition of finely-ground  $\text{BaSO}_4$ . A. R. P.

**Separation of titanium dioxide hydrate from hydrolysable solutions of titanium salts.** J. BLUMENFELD, Assee. of VER. F. CHEM. U. MET. PROD. (B.P. 384,875, 21.3.32. Ger., 20.3.31. Addn. to B.P. 310,949; B., 1930, 143).—A  $\text{HCl}$  solution containing  $\text{Ti}$  and  $\text{Cl}$  in the ratio 1:3 and about 150 g. of  $\text{TiO}_2$  per litre is hydrolysed at 100° with the addition of 7 g. per litre of  $\text{TiO}_2$  aq. prepared by hydrolysis at 80° of a solution of  $\text{TiO}_2$  in  $\text{HCl}$  having  $p_H$  2—3.5 (2.6). The  $\text{TiO}_2$  is quantitatively pptd. in 10 min. and after calcination with 2%  $\text{KHSO}_4$  at 900—950° has a very high covering power after grinding in oil. A. R. P.

**Preparation of beryllium compounds.** DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 384,926, 27.6.32. Austr., 31.7.31).—Finely-powdered beryl is heated at 1200° for 2 hr. with 9—13 mols. of  $\text{CaCO}_3$  and the product is leached with 7—10%  $\text{HCl}$ , whereby a residue of  $\text{BeO}$  with some  $\text{SiO}_2$  is obtained from which the  $\text{BeO}$  can be extracted by heating with  $\text{H}_2\text{SO}_4$ . Up to half of the  $\text{CaCO}_3$  may be replaced by  $\text{Na}_2\text{CO}_3$  and the heating carried out at 1000° for 2—3 hr. A. R. P.

**Production of a catalytic material.** A. T. LARSON, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,853,771, 12.4.32. Appl., 30.1.28).—Claim is made for briquetted mixtures of a catalytically active oxide, e.g.,  $\text{Fe}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ , or  $\text{ZnO}$ , or oxide mixture, e.g.,  $\text{Cr}_2\text{O}_3$  +  $\text{CeO}_2$ , or  $\text{CrO}_3$  +  $\text{MnO}$ , bonded with a plastic metal, e.g.,  $\text{Fe}$ ,  $\text{Cu}$ ,  $\text{Zn}$ ,  $\text{Al}$ ,  $\text{Sn}$ ,  $\text{Pb}$ , or  $\text{Ag}$ . The briquettes may

be made by compressing mixtures of the metal powder and oxide until a coherent tablet is obtained or by the pptn. of two oxides together followed by reduction of one of them to metal by heating the mixture in  $\text{H}_2$ .

A. R. P.

**Production of base-exchange gels.** UNITED WATER SOFTENERS, LTD. From PERMUTIT Co. and (in part) PERMUTIT A.-G. (B.P. 384,817, 24.12.31).—A mixture of the reacting substances, e.g.,  $\text{Na}_2\text{SiO}_3$ ,  $\text{Al}_2(\text{SO}_4)_3$ ,  $\text{Fe}_2(\text{SO}_4)_3$ , and  $\text{Na}_2\text{CO}_3$ , is intimately ground in a dry state, steamed at 100°, moistened with  $\text{H}_2\text{O}$ , dried at 80°, and washed with  $\text{H}_2\text{O}$  to remove sol. salts. A. R. P.

**Recovery of sulphur [from pyrites].** H. G. C. FAIRWEATHER. From R. F. BACON (B.P. 382,697, 28.8.31).—The ore is heated at 300—400° in a rotary furnace into which is passed a mixture of air, steam, and  $\text{HCl}$ , whereby  $\text{FeCl}_3$ ,  $\text{NiCl}_2$ , and  $\text{CuCl}_2$  are produced, and a mixture of  $\text{S}$ ,  $\text{H}_2\text{S}$ , and  $\text{SO}_2$  together with a little  $\text{HCl}$  passes out of the furnace through a condenser to remove  $\text{S}$  and then through a reaction tower to recover the  $\text{HCl}$  as dil. acid and to cause the  $\text{SO}_2$  and  $\text{H}_2\text{S}$  to react to form more free  $\text{S}$ . The mixture of chlorides and gangue in the furnace is further treated with  $\text{Cl}_2$  to volatilise  $\text{FeCl}_3$ , which is hydrolysed with steam to regenerate  $\text{HCl}$  for further use. A. R. P.

**Recovery of sulphur from roaster gases.** R. F. BACON (U.S.P. 1,842,230, 19.1.32. Appl., 9.5.27).—Pyritic ores are roasted in a current of preheated air to obtain a gas mixture with a high content of  $\text{SO}_2$  and a low content of  $\text{O}_2$ ; the gases are passed through a column of red-hot coke to reduce the  $\text{SO}_2$  to  $\text{S}$ , and the heat of the gases issuing from the reduction furnace is utilised to preheat air for the roaster. A. R. P.

**Purification of sulphur.** J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 385,352, 24.8.31).—Solutions of  $\text{S}$  in  $\text{CS}_2$ , obtained, e.g., by the extraction of spent gas-purifying material, are treated with  $\text{H}_2\text{SO}_4$ ,  $d < 1.84$ , and  $\text{HNO}_3$ , or with  $\text{ClSO}_3\text{H}$ ,  $\text{SO}_3$ , or, preferably, > 5 vol.-% of oleum; the solution, after separation from the acid and before distillation to recover the  $\text{S}$ , is treated with aq.  $\text{NH}_4\text{HSO}_3$  and  $(\text{NH}_4)_2\text{SO}_3$ , with adsorption agents (active  $\text{C}$ , clay,  $\text{SiO}_2$  gel), or with an aq. paste of  $\text{Ca}(\text{OH})_2$  to remove  $\text{SO}_2$ . L. A. C.

**Fire-proofing sulphur.** W. H. KOBÉ, Assr. to TEXAS GULF SULPHUR Co. (U.S.P. 1,853,818, 12.4.32. Appl., 25.6.31).—A mixture of molten  $\text{S}$  and  $\text{Ph}_2$  or one of its chlorination products is sprayed into a cool atm. to obtain a fine powder of spherical particles of  $\text{S}$  coated with the fire-proofing agent. A. R. P.

**Removal of free chlorine from chlorine-containing materials and production of chlorine-removing material therefor.** DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 385,455, 26.1.32. Ger., 26.1.31).— $\text{H}_2\text{O}$  is filtered through  $\text{C}$  which has been activated with  $\text{H}_2\text{O}$  vapour at 800—1000° and non-aq. liquids and gases are treated with the  $\text{C}$  in the presence of  $\text{H}_2\text{O}$  to remove free  $\text{Cl}_2$ . L. A. C.

**Extraction of iodine [from brine].** C. W. JONES and J. J. GREBE, Assr. to JONES CHEM. Co., INC. (U.S.P. 1,853,621, 12.4.32. Appl., 26.8.29).—The brine is

acidified to about  $p_H$  3.5 and electrolysed to liberate I, which is then expelled with a current of air and absorbed in an aq. alkali. A. R. P.

[Apparatus for] production of asbestos [from rock]. M. P. BRODSKY (B.P. 385,803, 22.3.32).

Gas purification.  $NH_3$  and  $(NH_4)_2SO_4$  from gases.—See II. Protecting metal surfaces.  $NH_3$ -oxidation catalyst.—See X. Pigments.—See XIII. Weed killer.—See XVI.

### VIII.—GLASS; CERAMICS.

Solubility of lead glasses in relation to their alkali content. O. KNAPP (J. Soc. Chem. Ind., Japan, 1932, 35, 520—522B).—Owing to the contradictory nature of the data available it is impossible to give a general rule for the solubility and weathering of Pb glasses. M. S. B.

Solubility of clays in alkaline solution. K. KOBAYASHI and K. YAMAMOTO (J. Soc. Chem. Ind., Japan, 1932, 35, 522—523B).—The proportion of material sol. in alkaline solution of varying concn. has been determined for 70 different samples of clay. The free hydrated  $SiO_2$  and  $Al_2O_3$  are more readily sol. than is the hydrated Al silicate. The method thus affords a means of distinguishing between different types of clay. M. S. B.

Refractory service, with particular reference to boiler furnaces. J. WALKER (Engineering, 1933, 135, 4—7, 34—37).—The properties and service behaviour of boiler-furnace refractories (generally fireclays) are described. Refractoriness tests and the effect of impurities on the refractoriness of fireclays are discussed. The mechanical strength of boiler-furnace refractories, usually adequate in the cold state, is reduced at high temp. The testing of refractories under load is described, and the behaviour under load of aluminous (44%  $Al_2O_3$ ) and semi- $SiO_2$  bricks is compared. Aluminous bricks are likely to be more satisfactory for use under conditions of moderate temp. and mechanical load, being safer and generally more resistant to corrosion by slag than semi- $SiO_2$  bricks. Conditions of heavy load and high temp. may necessitate modified furnace design or the use of bricks of high load-bearing capacity. Bricks made from a sillimanite base are stronger than ordinary firebrick at high temp., but are not very resistant to erosion by ferruginous slags. An aluminous brick (52—54%  $Al_2O_3$ ), very resistant to slag attack and showing great strength up to approx. 1810°, is described. To obtain max. rigidity of walls at top temp. joints must be as thin as possible, and highly-refractory cements giving joints strong at high temp. should be used. The factors influencing the abrasion of boiler-furnace refractories are described; for resistance to abrasion at high temp. well-burnt, highly-aluminous bricks of close texture and good skin are recommended. The resistance of refractories to thermal spalling (caused by steep temp. gradients or rapid changes of temp.) is discussed; bricks containing little free  $SiO_2$  (e.g., aluminous) are preferable for positions subject to rapid temp. changes. Mullite bricks are highly resistant to thermal spalling.

Physical spalling, caused by changes in the refractory in service (e.g., slag penetration) and mechanical spalling, due to localised stresses, are considered. Clinkering may be avoided by the use of  $SiC$  bricks in the appropriate positions. The slagging of boiler-furnace refractories is considered in detail, and factors influencing the extent of slag action are discussed. The rate of reaction with coal-ash slag is slower for aluminous than for more siliceous bricks; the slag produced is more refractory, more viscous, and hence less corrosive. A. L. R.

Aluminium oxide as a highly-refractory material. H. GERDIEN (Z. Elektrochem., 1933, 39, 13—20).—A lecture. F. L. U.

Open-hearth furnace crowns.—See X.

### PATENTS.

Apparatus for drying pottery, tiles, and the like. W. H. GRINDLEY, D. G. NORMAN, G. GREATBATCH, and A. MASSEY (B.P. 385,402, 14.11.31).—In an apparatus in which the goods are kept in motion on vertical conveyors, means for the uniform (or non-uniform, as desired) distribution of drying air is described. The suspended trays are provided with rubber curbs. B. M. V.

Production of grinding or abrading bodies. H. A. GILL. FROM DEUTS. GOLD- U. SILBER-SCHNEID-ANSTALT VORM. ROESSLER (B.P. 384,854, 26.2.32).—A mixture of powdered abrasive (100 pts.) with a suitable plastic binder (20—50 pts.), which can be subsequently hardened by heating and/or chemical treatment, is mixed with a proportion of a substance which generates gas, e.g.,  $O_2$ ,  $C_2H_2$ ,  $H_2$ , or  $CO_2$ , and the mixture is formed into the desired shape and heated to cause generation of the gas and hardening of the product to a cellular solid which absorbs cooling or lubricating media during use. A. R. P.

Production of fibres or threads from glass, slag, and like meltable materials. N.V. MAATS. TOT BEHEER EN EXPLOIT. VAN OCTROOIEN (B.P. 385,927, 24.10.32. Ger., 5.11.31. Addn. to B.P. 373,932).

Abrasive wheels. Filtering [bottle-washing] liquids.—See I.

### IX.—BUILDING MATERIALS.

Electroconductivity method of studying the processes of setting and hardening of lime-kieselguhr cements. N. PETIN, M. HIGEROVITSCH, and E. GAJSINOVITSCH (J. Gen. Chem. Russ., 1932, 2, 614—629).—The conductivity ( $C$ ) of lime-kieselguhr suspensions varies with time,  $H_2O$  content, and catalyst (alum,  $FeSO_4$ ); setting is characterised by an abrupt fall in the val. of  $C$ , and hardening by further gradual diminution in  $C$ . In the case of lime-clay suspensions the abrupt change in  $C$  does not occur. The above phenomena are more marked at higher than at lower temp. The characteristic change in  $C$  is more distinct when  $H_2O$  is allowed to evaporate than in closed systems.  $C$  attains a min. val. after 15 days in the presence of alum, thereafter remaining const. during 3 months. The above processes are irreversible. R. T.



**Formation of carbonate in litharge-glycerol cement.** TYPKE (*Angew. Chem.*, 1933, 46, 20—21; cf. B., 1929, 520).—The formation of carbonate is least when the ratio of PbO to glycerol is high, when the H<sub>2</sub>O content of the glycerol is low, and the temp. is 15°.

E. S. H.

**Road-tar emulsions.**—See II. Bitumen paints.—See XIII.

## PATENTS.

**Utilisation of free oxygen in the production of cement and iron.** C. B. HILLHOUSE (U.S.P. 1,842,609, 26.1.32. Appl., 12.3.29. It., 14.4.28).—Fe ore is reduced with hot CO from a producer-gas plant in which CO<sub>2</sub> is passed over hot coke, the Fe sponge is mixed with CaO sufficient to slag off the contained gangue, and the mixture is fused in a furnace fired with hot CO and O<sub>2</sub> to give Fe and cement clinker. The flue gases from this furnace are utilised partly in burning limestone to obtain the CaO necessary, and partly for regenerating CO in the producer, in which process the gases from the CaO kiln may also be employed.

A. R. P.

**Production of hydraulic cement.** G. O. CASE, E. M. ELLIS, and L. H. MONTIGUE (B.P. 385,664, 26.8.31).—Pulverised CaCO<sub>3</sub> (limestone) is mixed with a clinker produced by calcining a mixture of CaCO<sub>3</sub> and Al silicate in the proportion of 8 pts. of CaCO<sub>3</sub> to 1 pt. of the CaO liberated from the clinker on adding H<sub>2</sub>O. The total content of CaCO<sub>3</sub> is < 85 wt.-% of the mixture.

C. A. K.

**Cements and materials formed from them.** W. W. TRIGGS. From SOC. D'APPLICATIONS DES PÂTES DE CIMENT (S.A.P.A.C.I.). (B.P. 385,766, 28.1.32).—A composition consisting of a mixture of Portland cement 77, CaCO<sub>3</sub> 17, tripoli 3, and CaSO<sub>4</sub> 3 wt.-% is claimed. Cement mixtures may contain fibrous materials and supplementary SiO<sub>2</sub>.

C. A. K.

**Calcium sulphate plaster mixes and application of same.** V. LEFEBURE, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 385,611, 23.6.31 and 4.1.32. Cf. B.P. 381,155; B., 1932, 1082).—The gas-producing agents (or their reaction products) used in the production of porous sets from accelerated anhydrite plasters act as the accelerators; e.g. the agents comprise CaCO<sub>3</sub> and Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, or finely-divided Zn or Al. Additional accelerators, e.g., K<sub>2</sub>SO<sub>4</sub>, K<sub>2</sub>CO<sub>3</sub>, may be added, or setting may be accelerated by the admixture of > 20% of a hemihydrate plaster, e.g., plaster of Paris. Expansion may be delayed by the addition of a protective colloid (glue, bentonite), by the use of compact particles of the gas-producing agent, or by using ice-cold H<sub>2</sub>O for gauging the plaster.

L. A. C.

**Dental models.** I. G. FARBENIND. A.-G. (B.P. 385,896, 27.7.32. Ger., 27.7.31).—A plaster of Paris composition is used comprising < 75% of a gypsum hemihydrate the wt. by vol. of which is > 0.9 g. per c.c. and > 1.45 g. per c.c. "loosely put in" and "shaken in," respectively, the "strewed-in quantity" of which is < 230 g. per 100 c.c. to yield a pourable paste, and the "thickest consistency" is < 300 g. per 100 c.c.

L. A. C.

**Preparing a [sound-proof] mortar for forming or covering building materials.** BRIT. BRICK-AND-

IRON LATHING, LTD. (B.P. 385,818, 21.4.32. Holl., 21.4.31).—A mortar consisting of 1 vol. of calcined gypsum, 1 vol. of shell CaO, and 3—4 vols. of granulated blast-furnace slag is suitable for covering partitions.

C. A. K.

**Production of hard masses [plaster of Paris bandages] having a certain elasticity.** A. EICHENGRÜN (B.P. 385,658, 6.8.31. Ger., 6.8.30).—Plaster of Paris is mixed with a solution of a binding medium in an anhyd. solvent, then spread on muslin or other bandage material, and the solvent is evaporated. The binder should either be sol. in or swell rapidly in H<sub>2</sub>O e.g., artificial resins, so that after treatment with H<sub>2</sub>O the bandage sets with a certain amount of resiliency.

C. A. K.

**Reproducing the surface markings of wood and other material.** OXFORD VARNISH CORP., ASSEES. of L. V. CASTO (B.P. 374,205—6, 15.4.31. U.S., 16.4.30).—(A) The wood is stained to a colour corresponding with the ground colour of the wood to be copied, and then separately printed with two-design patterns out of register in such a way that one of the imprints emphasises the tone portions of the original design with the grain subdued and the other emphasises the grain only by making it with a heavier thickness of pigment material. These effects are obtained by etching the first plate lightly with a dil. acid and the second plate heavily and slowly with a more conc. acid. (B) The photographic reproduction used to make the second plate constitutes a process film on a suitable base, and that used for the first plate a portrait film of soft emulsion on a suitable base.

A. R. P.

**Impregnation of wood.** C. POLYSU (B.P. 385,327, 19.6.31. Rumania, 29.8.30).—Railway sleepers etc. are immersed in a suitable liquid which is subjected to a no. of alternations of pressure of short duration, each variation covering the whole range of pressure employed. [Stat. ref.]

L. A. C.

**Conveying cement sludge.**—See I. Products from asphalt emulsions.—See II. Ca aluminate slag.—See VII. Elastic bricks etc.—See XIV.

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

**Heat effects in metallurgical reactions.** C. SCHWARZ (*Arch. Eisenhüttenw.*, 1932—3, 6, 227—230).—Recent determinations of thermochemical data in reactions of importance in the metallurgy of Fe and steel are tabulated with full references.

A. R. P.

**Recent patterns in open-hearth furnace crowns.** E. MAASE (*Feuerfest*, 1932, 8, 177—178).—A thick lining to the crown of an open-hearth furnace prolongs the life considerably and reduces the loss of heat by radiation. Further improvements in these respects are obtained by riffling the lining, making alternate courses of brick protrude below the general level of the crown, and by efficient keying of the courses. MgO bricks have recently been manufactured to withstand prolonged firing at 1750° with intermittent cooling to 1000° without spalling, and crowns built of such bricks are much more refractory than those built of SiO<sub>2</sub> brick.

A. R. P.

**Economical heat regenerators for open-hearth furnaces.** H. TRINIUS (Arch. Eisenhüttenw., 1932—3, 6, 231—238).—The construction of the most economical type of regenerator for steel furnaces is discussed mathematically. A. R. P.

**Evaluation of iron ores for the blast furnace.** E. DIEPSCHLAG, M. ZILLGEN, and H. POETTER (Stahl u. Eisen, 1932, 52, 1154—1162).—The size and no. of pores in various Fe ores have been determined by means of a microscope with integrating stage and the results correlated with the reducibility of the ore in blast-furnace gas. Curves showing the relation between the size of the ore pieces and the reducibility are hyperbolæ for all ores, and the reducibility of an ore is a linear function of the surface area of pores per unit vol. of ore. With a certain degree of fineness, which varies for every ore, a const. max. reducibility is obtained; for minette this occurs with 23-mm. pieces, for hæmatite with 10-mm. pieces, and for limonite with 12-mm. pieces. Examples are given of the economy effected in coke by crushing the ore to a suitable size. A. R. P.

**Influence of the slag on cast iron in the electric furnace.** E. PIVOVARSKY and W. HEINRICH (Arch. Eisenhüttenw., 1932—3, 6, 221—226).—In refining cast Fe in the electric arc furnace under a CaO—CaF<sub>2</sub>—coke slag there is a reduction of about 12% in the Si content of the metal due partly to oxidation by constituents of the slag, partly to conversion into SiC, and partly to loss as SiF<sub>4</sub>. The liberated Ca combines with some of the S of the Fe, but the P in the Fe remains unchanged. Relatively little change in the Si content occurs under a CaC<sub>2</sub> slag. The oxidising slags, *i.e.*, those containing Al<sub>2</sub>O<sub>3</sub> or CaO, act unfavourably on the mechanical properties of the metal and tend to nullify or completely to inhibit the beneficial effects of overheating on the graphite distribution in the castings. A. R. P.

**Effects of molybdenum and chromium on the malleabilisation of white cast iron.** E. L. HENDERSON (Iowa State Coll. J. Sci., 1932, 6, 435—437).—1% Cr prevented graphitisation; with 5% Mo graphitisation was incomplete in 255 hr. Mo produces refinement in grain structure and imparts increased hardness, toughness, and tensile strength to the Fe. (Cf. B., 1932, 26.)

CH. ABS.

**Phosphorus in cast iron.** W. WEST (Metallurgia, 1932, 7, 83—88).—In cast Fe (3.3% C, 2% Si, 0.9% Mn, 0.05% S, 0.3% Cr, 0.05—1.5% P) tensile strength, transverse strength, and soundness are maximal with < 0.3% P. E. H. B.

**Rapid determination of carbon in iron and steel.** S. P. LEJBA (J. Gen. Chem. Russ., 1932, 2, 697—706).—The methods of Würtz and of O. Meyer give satisfactory results. R. T.

**Nitrogenisation of iron and iron alloys. III.** O. MEYER, W. EILENDER, and W. SCHMIDT (Arch. Eisenhüttenw., 1932—3, 6, 241—245; cf. B., 1932, 184).—Max. hardness of the surface layer after heating in NH<sub>3</sub> is obtained at 500°; rise in temp. reduces the hardness linearly in steels free from Al, but in those containing Al nitrogenising at up to 700° results in only a slight reduction in the hardness of the case. Increase

in pressure of the NH<sub>3</sub> increases the depth of penetration of the N, but reduces the surface hardness and has no effect on the time of hardening. When the nitrogenisation is carried out in a high-frequency alternating magnetic field diffusion of N into the steel is greatly accelerated, probably owing to the effects of magnetostriction. This process may also be applied to the tempering of steels which undergo pptn.-hardening: thus a steel with 6.19% Ti, 0.07% C, 0.87% Mn, and 0.58% Si quenched in H<sub>2</sub>O from 1250° reached max. hardness after 48 hr. at 600° in the usual furnace, but the same val. was obtained in 6 hr. with the aid of the magnetic field. A. R. P.

**Cause of sand inclusions in heavy [steel] forgings and means for their reduction.** K. DAEVES (Stahl u. Eisen, 1932, 52, 1162—1168).—The sandy inclusions often found in heavy forgings consist chiefly of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and MnO and their presence is attributed to a reaction between a highly manganiferous constituent (probably MnO) of the steel and the refractory lining of the furnace; hence in high-Mn steels it is essential to prevent inclusion of MnO as far as possible during melting or to use refractories which do not readily react with MnO. A. R. P.

**Limits of error in works determination of Brinell hardness [of steel].** M. MOSER (Stahl u. Eisen, 1933, 53, 16—18).—With a 10-mm. ball and a load of 3000 kg. measurements of the Brinell hardness of a steel should agree within ±5% unless the hardness is > 600, when larger variations may occur. Hence it is recommended that for very hard steels a cone or, better still, a square pyramid be used in place of the ball. A. R. P.

**Protection of iron and steel from corrosion.** E. S. HEDGES (Metallurgia, 1932, 7, 89—92).—Methods of protecting Fe and steel by oxide, phosphate, and other non-metallic coatings are briefly described and the prep. and properties of metallic coatings (Al, Cu, Cr, Pb, Ni, Sn, Zn) deposited on Fe by hot-dipping, electroplating, metal-spraying, or cementation are more fully discussed. E. H. B.

**Effect of pressure on corrosion of steels by hydrogen sulphide.** E. DITTRICH (Chem. Fabr., 1933, 6, 25—30).—A H<sub>2</sub>S—H<sub>2</sub> mixture under pressure was prepared by heating S in H<sub>2</sub> at 50 atm. to 350° and the corrosion of a no. of samples of alloy steel in an electric furnace exposed to this gas determined by the increase in wt. Effect of pressure up to 75 atm., temp. to 600°, and concn. of H<sub>2</sub>S is summarised in curves for each class of steel. Steels containing > 13% Cr are stable up to about 300° and the effect of pressure and concn. is slight. Steels with < 13% Cr show a rapid increase in corrosion with increasing pressure and H<sub>2</sub>S concn. and are also affected by H<sub>2</sub> at high pressures. Ordinary cast steel commences to corrode at 80°. Addition of Cr increases the temp. of corrosion, but corrosion of all alloys increases with temp. when a given min. has been passed. At temp. above 500° the protective effect of Cr is much less. At high pressures < 6% Cr gives a steel more corroded than ordinary cast steel. Ni lowers the temp. at which corrosion starts, but in conjunction with Cr seems to be beneficial. Si—Cr—Al steel samples were not resistant above 200°. C. I.

**Effect of oxygen pressure on corrosion of steel.**

A. R. LEE (Trans. Faraday Soc., 1932, 28, 825—826).—A reply to criticisms by Evans and Borgmann. (B., 1933, 64).  
F. L. U.

**Reduction of metals with gas.** G. V. RUIKOV (Tzvet. Met., 1931, 1442—1454).—The charge, consisting of powdered slag, coal, and fluxes, is blown into a preheated furnace; CO may also be used. In passing through the reducing zone the metals are vaporised and then oxidised, the oxides being caught in dust collectors. Difficultly oxidisable metals (Cu, Ni) are tapped from the bottom of the furnace. Experiments were made with Zn-Pb ore, Zn- and bronze-bearing slags, Zn concentrates, and Pb ores. The % recoveries were: Zn 95, Pb 96, Sn 85, Cu 95. The Zn contained no Fe or slag.  
CH. ABS.

**Froth formation and flotation ability of powders in solutions of surface-active substances.** N. M. LUBMAN (Tzvet. Met., 1931, 854—866).—Max. frothing of aq. isoamyl alcohol (I),  $\alpha$ -C<sub>10</sub>H<sub>7</sub>NH<sub>2</sub>, and *m*-cresol (II) occurred at small concns. before the saturation of the adsorption layer had been reached. In saturated aq. solutions of the reagent the froth formation is zero in (I) and (II). For acid and basic frothers neutralisation causes a sharp decrease in frothing ability. The max. flotation ability either coincides with max. froth formation or differs slightly on account of adsorption.  
CH. ABS.

**Flotation process. IV. Determination of contents of flotation agents in aqueous media by means of surface-tension measurements.** M. E. LIPÉTZ and M. M. RIMSKAYA (Tzvet. Met., 1931, 1432—1442).—The application of the capillary manometer to the determination of the composition of various mixtures of flotation agents is described.  
CH. ABS.

**Properties of copper deoxidised with calcium.** L. ZICKRICK (Amer. Inst. Min. Met. Eng., Preprint, 1932).—A high-Ca-Cu alloy is a satisfactory deoxidiser for Cu. Residual Ca raises the annealing temp. for dead-soft Cu from 250° to 350° or 400°. The electrical conductivity of the Cu is not greatly diminished.  
CH. ABS.

**Copper embrittlement. II.** L. L. WYMAN (Amer. Inst. Min. Met. Eng., Preprint, 1932).—Addition of small amounts of Ca (e.g., 0.0375%) increases resistance to embrittlement; large additions are detrimental to physical properties. Double-deoxidised Cu is superior to single-deoxidised Cu for similar or larger amounts of the same deoxidants.  
CH. ABS.

**Potential measurements and dissolution tests on tin- and zinc-copper alloys.** O. BAUER, O. VOLLENBRUCK, and G. SCHIKORR (Mitt. Materialprüf., 1932, 214).—Potential measurements do not always afford a correct insight into the behaviour of metals and alloys in a corrosive electrolyte since the fall in potential and the rate of dissolution of the material may be affected by the formation of oxidic films by the action of O<sub>2</sub> in the air, by the secondary deposition of one of the constituents of the alloy on the whole surface, and by the nature of the sol. products formed by the corrosion. Free access of air plays an important part in determining the extent and nature of corrosion; owing to the

depolarising action of the O<sub>2</sub> corrosion is generally promoted by access of air, but in some cases it is retarded by the formation of oxide films. In the absence of O<sub>2</sub> corrosion of Cu and high-Cu brasses by *N*-HCl is retarded. Determinations of the loss in wt. in corrosive media do not give a clear idea of the nature of corrosion of heterogeneous alloys or in some cases, e.g.,  $\alpha$ -brass in *N*-NaOH, of homogeneous alloys, hence the amount and nature of the material dissolved should always be ascertained. In corrosion tests made with a limited quantity of solution, which is not agitated during the tests, the continuous change in the composition of the electrolyte has a considerable effect on the results obtained; in some cases the rate of corrosion is continuously decreased and in others rapidly accelerated, e.g., Cu and brass in HCl, in the presence of O<sub>2</sub>.  
A. R. P.

**Recovery of metals from copper-zinc ores.** S. C. TONAKANOV (Tzvet. Met., 1931, 1455—1470).—The Zangezur ore is roasted at 850—900° (to S 7—9%), ground (30-mesh), and reduced at 1200—1400° with 100% excess of powdered coal. The CO content of the discharged gas should be 5—7%. 95% reduction of Zn and Cu was obtained; noble metals were recovered completely.  
CH. ABS.

**Electrolytic separation of lead as peroxide in non-ferrous alloys. I. Determination of small amounts of lead in copper and copper-rich alloys.** B. JONES (Analyst, 1933, 58, 11—26).—The sample is dissolved in HNO<sub>3</sub> (*d* 1.29) and evaporated to incipient crystallisation, when H<sub>2</sub>O is added. Sb and Sn are removed by filtration, if necessary, and heated with H<sub>2</sub>SO<sub>4</sub> and HNO<sub>3</sub> until fumes of SO<sub>3</sub> are evolved, when Pb, Cu, and Fe are pptd. by pouring into aq. NaOH and Na<sub>2</sub>S, a little CuSO<sub>4</sub> being added, if necessary, to aid pptn. of Pb. The sulphides are dissolved in dil. aqua regia and, after removal of HCl, the solution is added to the first filtrate. The electrolysis is carried out in a current of 2 amp. and 10—20 volts, with the anode rotating at 400 r.p.m. The deposit is dissolved in warm dil. (1:1) HCl, excess of which is removed on the water-bath, and Pb is pptd. as PbCrO<sub>4</sub> (cf. A., 1930, 881). If < 2% Mn is present in the alloy, Mn<sub>2</sub>O<sub>7</sub> formed during electrolysis is reduced by the cautious addition of 50% N<sub>2</sub>H<sub>4</sub>·2HNO<sub>3</sub>. When > 2% Mn is present, most of the Cu is deposited on the cathode, using a current of 3—4 amp. and reducing the Mn<sub>2</sub>O<sub>7</sub> as before. The deposit on the anode is dissolved in HCl and, after addition of the electrolyte and, if necessary, Fe<sup>+++</sup>, PbO is pptd. with Fe(OH)<sub>3</sub>, Mn being held in solution by NH<sub>4</sub>Cl. The Cu is dissolved in HNO<sub>3</sub> and electrolysed with the solution of the pptd. Fe and Mn. Special precautions are necessary when Ag, P, As, and Bi are present in quantity.  
T. McL.

**Effect of temperature and the presence of impurities on the rolling of zinc.** O. BAUER and P. ZÜNKER (Mitt. Materialprüf., 1932, 216).—Both electrolytic and refined Zn can be rolled at > 110°, but refined Zn has a tendency to fracture at the edges at 110—130°. With increasing degree of deformation and higher rolling temp. the rate of transformation of the coarse-grained casting structure into fine-grained rolling structure rapidly increases; this change takes place

more readily with electrolytic Zn owing to the absence of the restraining effect of impurities. Up to 1% Pb, 1% Cu, 0.25% Cd, or 1% Sb alone has no effect on the rolling properties, but  $> 0.1\%$  Fe,  $> 0.25\%$  Cd, or even less Sn has a very deleterious effect thereon; the effects of most impurities can be overcome, at least partly, by raising the rolling temp., but in the presence of Sn this is impossible since a readily fusible ternary eutectic is formed. A. R. P.

**Properties of nickel-chromium alloys and their manufacture.** A. M. KOROLKOV (Tzvet. Met., 1931, 1471—1474).—Alloys containing Ni 61.84, Cr 9—22, Fe 10—22, Mn 1.7% were prepared; the loss of Cr was 15—23%. Mechanical properties and microstructures of the cold-rolled, annealed alloys are described; the alloys are suitable for use in heating coils, ternary Ni-Cr-Fe alloys being the best. CH. ABS.

**Ageing of nickel chromium-nickel thermo-elements.** A. GRUNERT (Chem. Fabr., 1933, 6, 39—40).—Little change occurs in the serviceability after use for several years at 1000°. The ageing effect varies with composition, being considerable in elements containing little Cr. E. S. H.

**Prevention of liquation in white anti-friction alloys by addition of nickel.** A. M. BOCHVAR, F. P. BORIN, and M. YOSELEVICH (Tzvet. Met., 1931, No. 7, 850—854).—Ni (1—2.5%) prevents liquation and ensures uniform distribution of hard crystals in Sn-Sb alloys.  $Ni_2Sn_3$  is formed. CH. ABS.

**Roasting of concentrates containing mercury and antimony and their separation by successive roasting.** G. A. SHAKHOV and Y. Y. SLOBODSKA (Tzvet. Met., 1932, No. 1, 23—28).—The separation is technically and economically possible. CH. ABS.

**Modern tin refining.** W. SAVELSBERG (Metallbörse, 1932, 22, 833—834, 865, 897—898; Chem. Zentr., 1932, ii, 2232).—Removal of Cu, As, and Sb from crude Sn (98—99%) with Al (as an alloy containing 30% Al; 0.1% Al for 1% of impurity), with simultaneous action of atm.  $O_2$  is described. Difficulties ( $AsH_3$ ) attend the working up of the scum. Pb forms no compound with Al, and must be separated from Sn by liquation. The remaining Pb, retained in the Sn in solid solution, is removed by treatment with  $SnCl_2$  at 250—260°;  $SnCl_2$  is regenerated by chlorination to  $SnCl_4$  followed by interaction with Sn. Sn-Fe alloys are treated with Fe-Si to remove Fe. A. A. E.

**Use of high-lead tin bronzes as bearing metal.** R. SCHULZE (Giesserei Maschinenbau-Ztg., 1932, 5, No. 6—7, 10—11; Chem. Zentr., 1932, ii, 2362).—Up to 3% Pb is permissible; it improves working qualities and thermal resistance. In special bronzes 3—20% Pb may be present. A. A. E.

**X-Ray examination of electrolytically oxidised aluminium.** E. SCHMID and G. WASSERMANN (Mitt. Materialprüf., 1932, 218).—Anodic films of oxide on Al consist entirely, irrespective of the mechanical state of the metal, of extremely fine-grained but cryst.  $\gamma-Al_2O_3$ . Annealing of the film above 600° causes grain growth to occur, but no change takes place in the crystal structure. Treatment of the freshly formed film with high-

pressure steam to reduce its porosity converts it into  $Al_2O_3 \cdot H_2O$  (böhmite). A. R. P.

**Effect of cadmium and lead on the properties of aluminium.** B. BLUMENTHAL and M. HANSEN (Mitt. Materialprüf., 1932, 218).—All the tests were made on alloys in which the Cd and Pb were regularly distributed throughout the Al; in cases where the liquid phase was heterogeneous the two liquids were thoroughly mixed at such a temp. that the rate of separation and agglomeration of the Pb and Cd particles was small so that a homogeneous alloy was obtained on casting. Pb has no effect on the mechanical or corrosion-resistant properties of Al but improves its machinability. Cd increases slightly the tensile strength, but has no effect on the elongation or bending properties of normalised Al; the strength and hardness are, however, considerably increased and the ductility is reduced by quenching and ageing Cd-Al alloys. Cd has little effect on the electrical conductivity of Al. A. R. P.

**Determination of phosphorus in aluminium.** K. STEINHÄUSER and J. STADLER (Z. anal. Chem., 1932, 91, 165—170; cf. B., 1930, 1157).—Traces of P can be determined accurately by the  $NH_4$  phosphomolybdate method only if the ppt. is heated for some time and subsequently boiled with  $H_2O$  for 1 hr. J. W. S.

**Detection of free metal particles in dust etc.** T. J. WARD (Analyst, 1933, 58, 28).—The sample is freed from grease, if necessary, and treated with  $AgNO_3$  solution on a microscope slide. Ag "trees" are formed by Cu, Hg, Bi, Pb, Zn, Al, and Mg, but not always by Sn and Fe. T. McL.

**Use of potassium hydroxide as a fusion agent.** P. F. THOMPSON (J. Soc. Chem. Ind. Victoria, 1932, 32, 699—703).—KOH with a little  $H_2O$  is much more effective than anhyd. KOH for decomposing pyritic ores, mattes, Sn ores, wolfram, chromite, silicates, ferrotungsten and -chromium, and rock phosphate (for  $Al_2O_3$ ) by fusion in Ni crucibles. Sulphides are hydrolysed by the  $H_2O$  and the  $K_2S$  first formed is rapidly oxidised to  $K_2SO_4$ , the alloys yield  $H_2$  and K salts of the metal acids, and the rock phosphate solutions of  $KAlO_2$  and a ppt. of  $Ca_3P_2O_8$  after leaching. A. R. P.

**Electrodeposition of nickel with insoluble anodes.** V. I. LAINER, S. A. PLETENEV, V. A. KUZNETZOV, and B. I. ROZOV (Tzvet. Met., 1931, 1294—1310).—The (Khalila) ore contained much Fe. Ni was deposited from  $NiSO_4$  (20 g. Ni per litre) solutions with Al cathodes and Pt anodes at 80° with c.d. 600 amp. per sq. m., using acid-proof diaphragms; the anodic solution contained  $H_2SO_4$  and  $Na_2SO_4$ . With rotating cathodes the deposition of Ni is possible when Fe: Ni  $\geq 1:10$ . The presence of  $\geq 0.1$  g. Al per litre is desirable. CH. ABS.

**Modern chromium-plating plant.** W. BIRETT (Z. Metallk., 1932, 24, 289—295).—Fe tanks the sides of which are protected by loose glass plates are the most suitable for Cr-plating plant provided that the free  $H_2SO_4$  content of the electrolyte is kept within the correct limits. With a c.d. of 20 amp. per sq. dm. a max. current yield of  $>16\%$  is obtained when the  $H_2SO_4$  content corresponds with 0.04N-acid; with a c.d. of 10 amp. per sq. dm. the max. yield is 12% with 0.05N-acid.

With increasing  $\text{CrO}_3$  content  $> 25\%$  the temp.-c.d. range within which bright deposits are obtained is considerably narrowed and displaced towards lower temp. and lower c.d. Methods of controlling the composition of the bath and the val. of intermediate Ni plates are discussed. A. R. P.

**Varnish-kettle manufacture.**—See XIII.

#### PATENTS.

**Blast furnaces and cupola furnaces.** L. F. T. FONDERIES (B.P. 374,847, 6.3.31. Fr., 22.11.30).—The furnaces are provided with devices whereby a portion of the flue gases is withdrawn by means of a hot-air injector and introduced with the hot air into the fusion zone of the furnace, while the remainder of the flue gases is burned to preheat the air for the injector. A. R. P.

**Operation of cupola furnaces.** VEREIN. STAHLWERKE A.-G. (B.P. 379,361, 29.5.31. Ger., 15.7.30).— $\text{H}_2\text{O}$  is introduced into the furnace just above the melting zone by means of an adjustable pipe passing downwards through the centre of the charge. A. R. P.

**Reduction of metals from their oxides.** W. M. GRANT (B.P. 384,327, 19.5.31).—A mixture of the oxide with a carbonaceous reducing agent is mixed with CO or gas mixtures containing it to obtain a "fluidised" product which is passed continuously through tubes heated to a temp. at which the oxide is reduced to sponge metal. A. R. P.

**Production of insulated ferromagnetic [iron] powder and of cores therefrom.** HARTSTOFF-METALL A.-G. (HAMETAG) (B.P. 384,778, 23.11.31. Ger., 24.11.30).—Fe powder (100 kg.) is intimately coated with 30 g. of olive oil and made into a paste with a suspension of 500 g. of finely-powdered asbestos in 10–15 litres of  $\text{H}_2\text{O}$  containing 500 g. of  $\text{Na}_2\text{B}_4\text{O}_7$ . The paste is dried and pressed into cores of the desired shape. A. R. P.

**Production of metal [iron, nickel, or cobalt, or alloys thereof] sponge.** E. G. T. GUSTAFSSON (B.P. 373,298, 28.2.31. Swed., 6.3.30).—The ore is briquetted with a binder and an excess of carbonaceous reducing agent and heated to the reducing temp. by passing over it hot combustion gases containing an excess of oxidising gas sufficient to burn out most of the excess C in the briquettes. The briquettes are finally completely reduced by transferring them to another zone of the furnace and passing over them heated reducing gases while supplying heat externally to this zone of the furnace. A. R. P.

**Purification of iron [sponge].** A. A. JOHNSON (A. JOHNSON & Co.), B. M. S. KALLING, and C. VON DELWIG (B.P. 385,639, 22.5.31).—The sponge is heated in a reducing atm. with  $\text{BaO}$ ,  $\text{CaO}$ , or  $\text{MgO}$  and C or with  $\text{CaC}_2$ , whereby the S is eliminated. A. R. P.

**Low-carbon steel.** A. FRY, Assr. to F. KRUPP A.-G. (U.S.P. 1,855,105, 19.4.32. Appl., 16.12.27. Ger., 27.8.26).—Mild steel containing  $< 0.4\%$  C is rendered immune to caustic embrittlement by annealing at  $650\text{--}950^\circ$  to obtain a homogeneous solid solution, quenching, and re-annealing at  $< 750^\circ$  ( $730^\circ$ ). A. R. P.

**Making silicon steel from silicon scrap.** V. B. BROWNE (U.S.P. 1,853,544, 12.4.32. Appl., 31.7.31).—

The scrap is melted in an acid-lined arc furnace under a flux formed by fusing together  $\text{CaO}$ ,  $\text{SiO}_2$ ,  $\text{MnO}_2$ , and  $\text{Na}_2\text{CO}_3$  and with the exclusion of air (cf. U.S.P. 1,842,536; B., 1932, 1122). A. R. P.

**Ferro-aluminium-silicon alloy.** B. D. SAKLATWALLA, Assr. to VANADIUM CORP. OF AMERICA (U.S.P. 1,853,229, 12.4.32. Appl., 6.2.29).—An alloy for use in the removal of gases and oxides from steel comprises Fe with 5–30% Al, 10–48.5% Si, and  $\geq 0.4\%$  C, the Si content being always  $>$  the Al content. A. R. P.

**Treatment of steel sheets.** H. E. SHELDON, Assr. to ALLEGHENY STEEL CO. (U.S.P. 1,854,628–9, 19.4.32. Appl., 26.9.29).—Hot-rolled Si-steel sheets are annealed quickly by passing them in a vertical position through a furnace heated at  $980\text{--}1060^\circ$ , quenched by spraying them with an acid pickle, washed by passing them through high-pressure jets of  $\text{H}_2\text{O}$ , dried, and, in (A), passed through a series of 4-high cold-rolling mills and again subjected to the same series of operations, or in (B) packed in a box and annealed in the usual manner. A. R. P.

**Non-magnetic iron-nickel-copper alloy.** N. B. PILLING, Assr. to INTERNAT. NICKEL CO., INC. (U.S.P. 1,853,575, 12.4.32. Appl., 16.11.28).—The alloys consist of Fe with 1–20% Cu,  $\geq 22\%$  Ni, and  $> 0.4$  ( $0.5\%$ ) C. The % Ni should be  $\geq 1.5 \times \% \text{Cu}$  and the % C =  $6 [0.27 - \% \text{Ni} / (\% \text{Ni} + \% \text{Fe})]$ . A. R. P.

**[Inhibitor for] preserving metal [iron] surfaces [during pickling].** J. C. VIGNOS, Assr. to RUBBER SERVICE LABS. Co. (U.S.P. 1,855,035, 19.4.32. Appl., 14.1.28).—A thiuram disulphide, especially the diethyl-di-*o*-tolyl compound, is claimed. A. R. P.

**Treatment of corrosion- or heat-resisting alloys to remove oxide scale therefrom.** H. WIGGIN & Co., LTD., and J. STOCKDALE (B.P. 385,429, 31.12.31).—Cr and Ni-Cr steels after heat-treatment are pickled at 65–75% in a solution containing 0.25–10% HCl or  $\text{H}_2\text{SO}_4$  and 0.25–10%  $\text{CuSO}_4$ . A. R. P.

**Protecting an immersed [metal] surface from corrosive action.** S. GILL and S. U. MCGARY, Assrs. to GULF PRODUCTION Co. (U.S.P. 1,854,898, 19.4.32. Appl., 11.9.30).—The corrosion of metal parts in mixtures of brine and crude petroleum is prevented by adding to the latter substances which cause it to wet the metal surface preferentially to the brine, e.g., the higher aliphatic acids or their salts, or naphthenic acids or certain of their salts. A. R. P.

**Methods of intimately uniting a body of metal having a low m.p. [brass] with a body of metal having a high m.p. [steel].** E. F. MATTHEWS (B.P. 384,367, 27.8.31. Ger., 1.9.30).—The steel is heated to such a temp. that the expansion undergone in this heating is equal to the contraction undergone by the brass between its m.p. and room temp., and the brass is then cast on to the steel by the use of suitable moulds and cores. A. R. P.

**Blowing cupriferous metallic sulphide melts, especially low-grade copper mattes, direct to black copper.** H. MASCHMEYER, Assr. to AMER. LURGI CORP. (U.S.P. 1,853,627, 12.4.32. Appl., 20.4.31. Ger., 30.4.30).—The ordinary bessemerising treatment

is carried out with a slow current of air and the converter is heated externally during the operation so that the whole operation takes twice as long as usual. In this way the slag is free from magnetite, and being much more mobile does not retain large amounts of partly or completely converted matte in the form of prills.

A. R. P.

**Casting of alloys.** W. PEYINGHAUS and J. KUPFERBERG, Assees. of G. PEMETZRIEDER (B.P. 381,413, 29.12.31. Ger., 30.12.30).—The alloys are cast centrifugally into the bottom of moulds radiating outwards and upwards from the bottom of the rotating part of the centrifuge, so that the cooling is extremely rapid and there is no time for segregation to occur. Alloys of the following compositions cast in this way are claimed: (a) Cu 53, Pb 19, Fe 28%; (b) Cu 85.6, Pb 14.4%; (c) Fe 95.3, Al 4.7%; (d) Fe 65.5, Al 34.5%.

A. R. P.

**Copper alloys capable of being age-hardened.** INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 385,652, 22.7.31. Ger., 31.7.30).—Alloys of Cu with 0.1–6% of  $M_2Be$  or  $MBe$ , where M is Co or Cr, are claimed; the heat-treatment comprises quenching from a high temp. and ageing at 300–600°.

A. R. P.

**Rolling compounds of metals with non-metals or alloys of these compounds with metals [e.g., phosphor-copper].** METALLGES. A.-G. (B.P. 385,119, 6.1.32. Ger., 9.1.31).—Alloys of Cu with > 6.5% P, > 7% Si, > 17% Sb, or > 13% As are rendered ductile by plastic deformation at just below the eutectic temp.

A. R. P.

**Treatment of bronze scrap for separating from it copper, tin, lead, and precious metals.** SOC. ANON. SUD MÉTAUX (B.P. 385,413, 30.11.31. Fr., 1.12.30).—The bronze scrap is packed between conducting, non-corrodible grids covered with flannel and electrolysed in an acid  $CuSO_4$  bath containing Cu-sheet cathodes between the grid anodes. The anode mud is roasted at 200–300°, leached with hot dil.  $H_2SO_4$  to remove Cu, and fused with reducing agents and fluxes to obtain a crude Sn which is subsequently dissolved in HCl, whereby a residue containing Pb, Au, and Ag is obtained.

A. R. P.

**Distillation processes for production of zinc or similar volatilisable metals.** F. KRUPP GRUSONWERK A.-G. (B.P. 374,900, 17.3.31. Ger., 20.12.30).—The material, e.g., roasted Zn ore, mixed with a large excess of carbonaceous reducing agent, is heated in a muffle which is built into a rotary furnace, until 60–90% of the Zn is volatilised. The residue is then discharged into the furnace and the excess C burned therein to supply heat for the muffle and to volatilise the remaining Zn and other volatilisable metals, which are then recovered from the flue gases. The excess of coke in the muffle prevents slagging, and the two-stage heating yields a residue much freer than usual from volatilisable metals.

A. R. P.

**Production of coloured coatings [on zinc].** J. SCHULEIN (U.S.P. 1,853,323, 12.4.32. Appl., 24.9.28).—A.c. with a frequency > 25 (60) cycles/sec. is passed between two Zn articles immersed in a hot (80°) solution

containing 250 g. of  $CrO_3$  and 3 g. of  $Cr_2(SO_4)_3$  per litre.

A. R. P.

**Mercury condenser.** F. B. PRESCOTT (U.S.P. 1,841,913, 19.1.32. Appl., 5.8.30).—The apparatus comprises a series of adjacent chambers connected to one another at the top by  $\Omega$ -shaped cooling pipes and an exhaust suction fan for drawing the gases from the roasting furnace through the condensers and for discharging the residual gases from the condensers centrifugally through collecting  $H_2O$ -spray tanks.

A. R. P.

**Formation of (A) chromium- (B) silicon-alloy coatings.** L. H. MARSHALL, Ass. to TECHNIMET CO. (U.S.P. 1,853,369—70, 12.4.32. Appl., 27.12.27).—(A) Fe and steel articles are superficially coated with a Cr alloy by heating them at 1000–1100° for 20 hr. in a mixture of ferrochromium and chromite with 5% of  $CaOCl_2$ . (B) Similar Si coatings are obtained with a mixture of Si powder, coarse  $SiO_2$  sand, and 5–10% of  $FeCl_3$  or substances which react to produce  $FeCl_3$  on heating.

A. R. P.

**Production of hard metal compositions.** BRIT. THOMSON-HOUSTON CO., LTD., Assees. of G. F. TAYLOR (B.P. 385,629, 19.6.31. U.S., 19.6.30).—The powdered constituents are pressed into a bar which is sintered under pressure in an atm. of  $H_2$  under low pressure or in vac. The bar is heated by an electric current passed through it, and pressure is applied by means of steel or Mo plungers; means are provided for actuating the plungers as the bar shrinks and for cutting off the current after a predetermined time.

A. R. P.

**Production of large-crystalled metallic bodies.** VEREIN. GLÜHLAMPEN U. ELEKTRICITÄTS A.-G. (B.P. 384,309, 22.4.31. Ger., 14.2.31).—Blocks, bars, wires, or filaments of large crystals of Re, W, Mo, etc. are obtained by reducing an intimate mixture of an oxide of one of these metals with  $Zr_3(PO_4)_4$ ,  $Th_3(PO_4)_4$ ,  $FeSiO_3$ ,  $Al_2SiO_5$ ,  $ThSiO_4$ ,  $MgSiO_3$ ,  $ZrSiO_4$ ,  $CaTiO_3$ ,  $AlBO_3$ ,  $CaF_2$ ,  $BaF_2$ ,  $ZrOF_2$ , or mixtures of two or more of these or similar compounds, forming the reduced mixture into pressed shapes, sintering them just below the m.p., and then heating them at a temp. at which crystal growth is rapid.

A. R. P.

**Light-metal [magnesium-silicon-aluminium] alloy.** STERLING METALS, LTD., and E. PLAYER (B.P. 384,889, 20.4.32).—The alloy consists of Al with 5–15 (6%) Mg, 8–18 (11%) Si, and 0.5–3 (1%) Cr; it is hardened by quenching from 500–550° and ageing at 180–250°.

A. R. P.

**Treatment of oxide coatings on aluminium and its alloys.** VEREIN. ALUMINIUM-WERKE A.-G. (B.P. 385,763, 25.1.32. Ger., 31.12.31).—Anodic oxide layers on Al are impregnated with an "I.G. wax." [Stat. ref.]

A. R. P.

**Production of firmly adherent galvanic deposits on aluminium and its alloys.** SIEMENS & HALSKE A.-G. (B.P. 385,067, 6.11.31. Ger., 6.11.30).—The articles are anodically oxidised in a  $CrO_3$  bath, then plated with brass in an alkaline cyanide bath, and finally plated with Ni or Cr in the usual way.

A. R. P.

**Manufacture of metal catalysts.** SCHERING-KAHLBAUM A.-G. (B.P. 384,175, 27.4.32. Ger., 5.5.31).—Powders consisting of carriers impregnated with the metal oxide are allowed to fall in a thin stream through a heated tube through which  $H_2$  is passed, preferably countercurrentwise. A. R. P.

**Catalytic reactions and [rhenium] catalysts for use therein. [Oxidation of ammonia.]** G. SIEBERT GES.M.B.H. (B.P. 385,859, 8.6.32. Ger., 22.6.31).—The gauze catalysts are made of a Re-Pt alloy or of a Pt alloy coated with Re or a Re alloy. A. R. P.

**Anti-rust solution for radiators.**—See I. S from roaster gases.—See VII. Producing cement and Fe.—See IX. Electrolysis [for metal deposition].—See XI. Anticorrosive paints. Resin lacquers [for wire]. Decorating metal objects.—See XIII. Photographs on Al.—See XXI.

### XI.—ELECTROTECHNICS.

**Electrostatic pptn. and carbonisation.**—See II. **Hardening of cements.**—See IX. **Nitrogenisation of Fe. Protecting Fe etc. Measurements on Sn- and Zn-Cu alloys. Separation of Pb as  $PbO_2$  in alloys. Ni-plate. Cr-plating plant.**—See X. **Rubber insulation.**—See XIV. **Refuse disposal.**—See XXIII.

#### PATENTS.

**[Alkaline] electric storage batteries.** J. J. DRUMM, and DRUMM BATTERY CO., LTD. (CELIA, LTD.) (B.P. 385,614, 24.6.31).—Positive plates comprising perforated tubes or flat, rectangular, perforated Ni pockets containing  $NiO_2$  or Ag oxide mixed with Ni flake and/or graphite, and negative plates of perforated plate or expanded metal having a Ni or Co surface are arranged horizontally in a container. The active material consists of Zn plated out of the electrolyte. J. S. G. T.

**Electric gaseous [arc-]discharge devices.** CLAUDE-LUMIÈRE, STÉ. AME. POUR LES APPLICATIONS DES GAZ RARES À LA LUMIÈRE, PROC. G. CLAUDE (B.P. 385,311, 17.3.31. Fr., 5.11.30).—The pressure range ( $p$  mm. Hg) of the rare-gas filling is 0.1–3 mm. and is adjusted according to the diam. ( $d$  cm.) of the discharge tube so that  $p > 6/d$  and  $< 8/d$ . Constructional details of cathodes and anodes are claimed. J. S. G. T.

**Manufacture of [cold cathodes for] thermionic valves and like electric-discharge devices.** TELEFUNKEN GES. F. DRAHTL. TELEGRAPHIE M.B.H. (B.P. 385,496, 4.4.32. Ger., 2.4.31).—The surface of the electrodes is coated, e.g., sprayed, with an emulsion of the oxide or oxides of refractory metals, e.g., W, Mo, or Ta, which is then reduced to metal by heating in  $H_2$  and coated, e.g., with collodion solution. J. S. G. T.

**Electrolysis with the use of a mercury cathode.** K. W. PALMAER (B.P. 385,756, 18.1.32. Swed., 29.4.31).—Hg employed as cathode in the electrodeposition of metals of group VIII is continuously purified by fluid, e.g., mercurous salt solution, in a separate compartment (A). Apparatus for transferring the liquids to A is claimed. [Stat. ref.] J. S. G. T.

**Regulating the viscosity of liquids. Separating particles from gases. Bubbling gases through liquids [for electric motors]. Hg-vapour vac. pump.**—See I.  $C_5H_5N$  derivatives.—See III. I from brine.—See VII. Ferromagnetic cores. Non-magnetic Fe alloy. Bronze scrap. Coloured coatings on Zn. Hard metal compositions. Oxide coatings on Al. Deposits on Al.—See X. Vitamin-D.—See XX.

### XII.—FATS; OILS; WAXES.

**Alkalinity of dilute aqueous soap solutions and effect thereon of addition of free alkali.** J. DAVIDSOHN (Chem. Umschau, 1932, 39, 275–276).—The work of Bleyberg and Lettner (B., 1933, 74) endorses the author's previously expressed opinions that a small excess of alkali in toilet soap is harmless and helps to prevent rancidification. E. L.

**Significance of the fatty acid carboxyl group in the textile-aid industry.** K. LINDNER (Chem. Umschau, 1932, 39, 274–275).—A short discussion, supplementing the paper by Stadlinger (B., 1933, 28) and including a description of some modern commercial wetting agents. E. L.

**“Umesterung” [double decomposition]. I. Double decomposition of fatty oils with certain alcohols, and preparation of alcohol-soluble boiled oils and a new emulsifier.** R. ODA (J. Soc. Chem. Ind., Japan, 1932, 35, 515–518B).—Double decomp. (trans-esterification) between olive oil and EtOH is very slow; even when excess EtOH was present, and at  $200^\circ$  under 20–25 atm., esterification was only partial after 9 hr. (Ac val. of reaction product, e.g., 72.4). Increase of Ac val. of the products of the treatment of olive oil under pressure at  $200^\circ$  with excess glycerol is very slow; some solid products are also formed. Reaction between olive (or fish, linseed, or coconut) oil and excess of ethylene glycol occurs readily and rapidly; glycol mono-esters appear to be formed. Reaction is slower when a smaller proportion of glycol is used; in both cases the products are EtOH-sol., emulsify readily in  $H_2O$ , and yield sulphonation derivatives of high emulsifying power. Films of an EtOH-sol. boiled oil prepared from linseed oil (100 g.), ethylene glycol (7–10 g.), and Mn borate dried well, but the adhesion to the glass support was somewhat defective. E. L.

**Oil from Niger seed (*Guizotia Abyssinica*).** D. L. SAHASRABUDDHE and N. P. KALE (J. Univ. Bombay, 1932, 1, 37–47).—A detailed examination of this oil is described. The insol. acids contain lauric and myristic (3.35%), palmitic (8.41%), stearic (4.89%), arachidic and lignoceric (0.48%), oleic (31.06%), and linoleic (54.34%) acids. The oil contains 3–4% of a triolein (Br derivative, m.p.  $76.5^\circ$ ) and 20–25% of an oleo-dimolein (Br derivative, m.p.  $54$ – $56^\circ$ ). R. S. C.

**Falsification of train oils.** M. AUERBACH (Chem. Umschau, 1932, 39, 273).—A train oil which had been “improved” by addition of alkali was of a clear reddish-yellow colour, and on keeping deposited a small amount of soap which redissolved on warming; on acidification, however, the oil became very dark and turbid. E. L.

**New compounds produced during the hydrogenation of fish oils.** IV. **Fatty acids.** S. UENO and R. YAMASAKI (J. Soc. Chem. Ind., Japan, 1932, 35, 492—495 B; cf. B., 1931, 727).—The 0.2% of distillate obtained on hydrogenating fish oils has been separated into unsaponifiable matter (about 13.6%, consisting mainly of branched-chain  $C_{10-20}$  hydrocarbons, with minor amounts of  $C_9$ ,  $C_{10}$ , and  $C_{13}$  alcohols, and malodorous  $C_{10}$ ,  $C_{12}$ ,  $C_{14}$ , and  $C_{16}$  aldehydes; cf. *loc. cit.*), about 10% of lower saturated fatty acids ( $C_7$ ,  $C_8$ ,  $C_9$ , and  $C_{10}$  in the proportions 5:3:3:1), and about 60% of higher saturated acids from which myristic, palmitic, and stearic acids (in the proportions 2:7:1) and very small amounts of behenic acid have been isolated. E. L.

**Cane wax in sugar.**—See XVII.

#### PATENTS.

**Manufacture of derivatives of soap-forming carboxylic acids.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 384,314, 23.5.31).—Soap-forming carboxylic acids above  $C_7$ , alone or admixed with lower acids, are converted into the corresponding ketones (e.g., by passing the vapour over  $ThO_2$  at 400—450°), which are then treated in liquid phase with  $NH_3$  or primary amines and  $H_2$  to give amines suitable for manufacture of washing agents. Examples are amines from ketones derived from: coconut oil acids with  $AcOH$  (ketone, b.p. 80—185°/12 mm.; amine with  $NH_3$ , b.p. 88—200°/12 mm.); naphthenic acid and  $AcOH$  (ketone, b.p. 75—103°/12 mm.; amine with  $NH_3$ , b.p. 70—120°/11 mm.); stearic acid and  $AcOH$  (amine with  $NH_3$ , b.p. 187—193°/13 mm.; amine with  $NH_2Bu$ , b.p. 205—220°/11 mm.; amine with  $NH_2Ph$ , b.p. 200—220°/4 mm.). C. H.

**Improving the taste and smell of fatty oils.** I. G. FARBENIND. A.-G. (B.P. 385,774, 12.2.32. Ger., 12.2.31. Addn. to B.P. 382,060; B., 1933, 29).—Fatty oils other than fish oils, e.g., castor oil, are hydrogenated over a non-noble metal (Ni) catalyst under pressure and at temp. < 100°, to such a slight extent that the physical properties of the oils are practically unaltered, the amount of  $H_2$  absorbed being > 5—10 litres per 500 g. of oil. E. L.

**Reduction of viscosity of greasy oils for cosmetics, etc.** H. EISNER and H. VOLLMER (B.P. 385,306, 18.6.31).—Admixture of 3—10% of essential oil(s) (e.g., lavender oil) with a mineral or fatty oil, or sulphurised oil, reduces the  $\eta$  remarkably; cloudiness is prevented or removed by irradiating the essential oil or the mixture with ultra-violet light, preferably in the absence of  $O_2$ . E. L.

**Stabilised greases.**—See II. **Fatty acid amides.**—See III. **Oxide coatings on Al.**—See X. **Kneading of edible fats.**—See XIX. **Vitamin-D.**—See XX.

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

**Bitumen paints and their importance as protective agents.** A. W. RICK (Farben-Chem., 1933, 4, 9—10).—A general account is given of the properties of such paints. Particular reference is made to their suitability for protecting concrete. S. M.

**Alumina lakes.** W. D. BANCROFT and E. C. FARNHAM (J. Physical Chem., 1932, 36, 3127—3137).—The adsorption of alizarin by  $Al_2O_3$  varies with the nature and thermal treatment of the  $Al_2O_3$ . The lakes are adsorption complexes, no chemical compounds being formed. Orange II acid forms a definite compound of the type  $AlX_3$  with  $Al_2O_3$ , both in aq. and EtOH solution. At low concns. the acid dye is adsorbed. The Na salt of Orange II is adsorbed without formation of a definite compound.  $NaOH$  and Na alizarate are nearly equiv. in their pptg. and dispersing effects on  $Al_2O_3$ . Negatively-charged lakes peptised by Na alizarate are flocculated by  $NaCl$  or  $Na_2SO_4$ , owing to adsorption of  $Na^+$ . The colour of an alizarin solution can be varied in the series yellow, red, violet, blue by progressively increasing the  $p_H$ . The colour of alizarinic acid dissolved in  $C_5H_5N$  can be varied similarly by adding  $H_2O$ , which increases the  $p_H$ . Under suitable conditions  $Al_2O_3$  and  $SnO_2$  lakes of all these colours can be prepared. The red is stable over a wide range of  $p_H$  with  $Al_2O_3$  and over a narrow range with  $SnO_2$ . E. S. H.

**Structure of ultramarines.** I. **Technical ultramarines as basic material for research.** M. DOMINKIEWICZ (Rocz. Chemi., 1932, 12, 913—924).—Commercial ultramarines are solid solutions of ultramarine ( $Na_6Al_4Si_6O_{20}S_4$ ,  $Na_7Al_6Si_6O_{24}S_2$ ,  $Na_{7.5}Al_6Si_6O_{24}S_3$ , or  $Na_3Al_6Si_6O_{20}S_4$ ) and isomorphous Na kaolinite,  $Na_2Al_6Si_6O_{24}$  (0—40%). R. T.

**"Catalysis" in mixed pigments.** H. HEBBERLING (Farben-Ztg., 1933, 38, 402).—The discoloration of S-containing mixed pigments, e.g., the "greying" of lithopone-white lead mixtures, is considered not to be due to catalytic influence of traces of  $Fe_2O_3$  as has been suggested. The S content of modern lithopones, ultramarine blues, etc. is present in inactive forms, and the relatively few cases of discoloration are at present without explanation. S. S. W.

**Determination of "critical oil absorption" of pigments.** G. ZEIDLER and B. ROSEN (Farben-Ztg., 1933, 38, 403—404, 427—429).—The "crit. oil absorption" postulated empirically by Wolff (B., 1932, 194) is discussed and its practical significance indicated. Full details of experimental work and calculation of results are given for two samples of  $ZnO$  of crit. oil absorption 50.3 and 45.0%, respectively. S. S. W.

**Suitability of metals, alloys, etc. for varnish-kettle manufacture.** J. SOMMER (Farben-Ztg., 1933, 38, 426—427).—The properties of a range of materials are tabulated under the headings: price, durability, influence on colour of varnish, ease of cleaning, heat-transference. Separate data relating to suitability for kettle bottoms and bodies are collectively evaluated; it is shown that a monel-metal body and Sn-plated steel bottom is the best combination, whilst Cu body and Al bottom is the worst. S. S. W.

**Use of rubber resins in nitrocellulose lacquers.** A. KRAUS (Farbe u. Lack, 1933, 17—18, 28).—The solubilities in common nitrocellulose solvents and other properties of two resins obtained from gutta-percha by solvent extraction are tabulated. Neither resin enhances the mechanical properties of a nitrocellulose



film, but considerable improvement is observed if a plasticiser, *e.g.*, tolyl phosphate, is present, especially in the case of the softer resin, which thus resembles the original gutta-percha. The softer varieties can be used in the prep. of finishing lacquers for leather and wood.

S. M.

**Testing light-resistance of nitrocellulose in lacquers.** W. TOELDT (Farben-Chem., 1933, 4, 5—7).—Erdmann's reaction (B., 1900, 277A) for the determination of  $\text{HNO}_2$  is used to determine the relative stabilities of nitrocellulose films when exposed to light. Although a film heated for 35 hr. at  $100^\circ$  gave a negative reaction, the presence of  $\text{N}_2\text{O}_3$  was detected after exposure for 1 hr. to the light from a Hg-vapour lamp, which is in fact considerably more effective than summer sunlight. Plasticisers, *e.g.*, Bu phthalate, absorb the light and hence reduce the amount of decomp.; resin alone had no effect. The  $\text{N}_2\text{O}_3$  content of the undercoat is also lowered by increasing the thickness of the covering coats. Basic pigments, *e.g.*, ZnO, prevent secondary reactions and consequent destruction of the mechanical properties of the film.

S. M.

**Xanthorrhœa and its resin.** H. T. COLE (Chem. Eng. Min. Rev., 1932, 25, 92—95).—Red resin from *X. Preissii* had acid val. 20, sap. val. 47, I val. 94.6—100.8. It softens at  $90$ — $100^\circ$ , decomposes readily at  $100^\circ$ , is sol. in common solvents containing O, but is insol. in linseed and tung oil. Efforts to prepare colourless oil-sol. products by the use of various adsorbents and reducing and oxidising agents failed. The  $\text{H}_2\text{O}$ -resistance of varnish films made from the resin is increased by pretreatment with NaOH.

S. M.

**Sorption of organic vapours by glyptal resins.** C. H. WINNING and J. W. WILLIAMS (J. Physical Chem., 1932, 36, 2915—2935).—Apparatus for measuring the sorption of vapours in a gas-free atm. is described. Curves obtained for the sorption and desorption of  $\text{CO}_2$  and MeOH vapours by powdered glyptal resins at  $35^\circ$  and  $50^\circ$  characterise the resin as a typical swelling gel. The first part of the sorption curve suggests a surface adsorption, whilst the last part suggests capillary condensation. The general behaviour agrees with the assumption of giant mols. as the fundamental structural units; a larger mol. size and a more firmly knit structure are indicated in the cured glyptal resins. The free-energy changes in the adsorption process have been calc. The tendency of a resin to become dispersed completely in a solvent decreases as the time of the curing process is increased. The resins may retain considerable amounts of adsorbed vapour.

E. S. H.

**Phenolphthalein as basis for a new class of artificial resins.** W. HERZOG (Farben-Chem., 1933, 4, 8—9).—The development of "Alkalit" resins (B., 1932, 562) is reviewed. Mol.-wt. determinations of phenolphthalein *o*-toluate (m.p.  $70$ — $85^\circ$ ) and *m*-toluate (m.p.  $67$ — $83^\circ$ ) in  $\text{C}_6\text{H}_6$  show that these resins are monomeric.

S. M.

## PATENTS.

**Anticorrosive paints or pigments.** F. RAHTJEN and M. RAGG (B.P. 385,310, 16.3.31).—The use of phosphates, nitrates, nitrophosphates, antimonates, ferro- and ferri-cyanides, manganates, and perman-

ganates of Pb is claimed. Particles of Pb or Pb alloy may be covered with a film of the Pb salt, and metals more resistant to oxidation than Fe may be added.

S. M.

**Manufacture of a red pigment of iron oxide.** F. KLEIN (B.P. 385,646, 9.7.31. Ger., 11.7.30).—Rust or Fe hydroxides of small  $\text{H}_2\text{O}$  content and of similar structure to rust, *e.g.*, FeS oxidised slowly in moist acid atm., are moistened with dil.  $\text{Fe}^{++}$  salt solutions, *e.g.*,  $\text{FeCl}_2$  (formed, if desired, in the reaction mixture) and heated under pressure, with no calcining process. The hue of the pigment is controlled by the degree of dilution of the solution and the duration of heating.

S. S. W.

**Manufacture of inorganic pigments.** I. G. FAR-BENIND. A.-G. (B.P. 385,773, 11.2.32. Ger., 11.2.31).— $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$  is intimately mixed with small quantities of the corresponding Cd, Mn, Mg, or Fe salt and the mixture is heated under non-oxidising conditions to expel  $\text{SO}_3$  and leave a solid solution of the monoxide of the added metal in ZnO having a characteristic, fast colour; *e.g.*, brown, red, or yellow pigments are obtained with varying quantities of MnO in the mixed oxides.

A. R. P.

**Decorating metal objects.** W. W. TRIGGS. From MEYERCORD Co. (B.P. 385,226, 29.6.32).—The metal is coated with a ground coat of varnish comprising a synthetic gum or resin ground in oil, with or without the addition of pigments, the ground coat is rendered tacky by moistening it with a mixture of (a) Et lactate, ethylene chlorohydrin, EtOH and  $\text{H}_2\text{O}$ , or (b) MeOH,  $\text{H}_2\text{O}$ , and the "Et ether of  $\text{C}_2\text{H}_4$ ," the design is applied as a printed or lithographic transfer, and the decorated article is then baked at  $120^\circ$ .

A. R. P.

**Phenol-aldehyde resin lacquers.** A. EHRENZWEIG (B.P. 385,730, 11.12.31. Switz., 6.3.31).—Lacquers containing hardenable PhOH-aldehyde resins in the resol (A) stage, tolyl phosphate, triacetin and/or dicrosylin, turpentine or glycerin, org. acids, 2—12% (on the resin) of cellulose ester, and, if desired, oil-sol. PhOH- $\text{CH}_2\text{O}$  condensation products, are claimed as wire enamels.

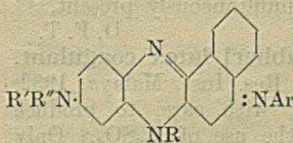
S. S. W.

**Manufacture of coloured lacquers and plastic masses.** SOC. CHEM. IND. IN BASLE (B.P. 385,409, 24.11.31. Switz., 25.11.30).—The lacquer or plastic is coloured violet to blue with a sulphonic acid (or alkali sulphionate) of a naphthaphenosafrafranine of annexed

formula. Examples are safranines from: phenyl- $\beta$ -naphthylamine-5-sulphonic acid and 4-amino- or 4-amino-2'- or -4'-methoxydiphenylamine-2-sulphonic acid, condensed with  $\text{NH}_2\text{Ph}$  (blue);  $\beta$ - $\text{C}_{10}\text{H}_7$ -NHPh and *p*-nitrososulphobenzylethylaniline, with 5-aminosalicylic acid (violet);  $\beta$ - $\text{C}_{10}\text{H}_7$ -NHPh and 4-aminodiethylaniline-3-sulphonic acid, with the corresponding 2-sulphonic acid (red-blue).

C. H.

**Manufacture of [resinous] condensation products.** I. G. FAR-BENIND. A.-G. (B.P. 383,764, 15.12.31. Ger., 27.12.30).—A cyclic ketone is condensed with a polyvinyl alcohol (which may be produced *in situ* from



an ester) in presence of an acid catalyst (dil.  $H_2SO_4$ ) to give resins sol. in org. solvents and in  $H_2O$ . Suitable ketones are cyclohexanone and its 2-Me- and 4-Cl-derivatives, and 1-ketotetrahydronaphthalene. C. H.

**Production of resin emulsions.** CHEM. U. SEIFEN-FABR. R. BAUMHEIER A.-G. (B.P. 385,344, 6.8.31. Ger., 6.8.30).—The resin after being melted with sufficient turpentine oil, fatty or mineral oil, alcohol or ester of high b.p., or wax of low b.p., to produce a liquid at  $100^\circ$ , is emulsified and homogenised. The product is suitable for dressing fabrics etc. [Stat. ref.] S. M.

**Continuous production of moulding mixtures from artificial resins.** A. NOWACK A.-G., and K. SPRENGER (B.P. 385,462, 3.2.32).—The intimately mixed materials are pressed between two moving bands, the upper band being pressed down by rollers and the lower supported by rollers or a platen; some or all of the rollers and the platen are heated. B. M. V.

**Acetylene polymerides. Ice colours.**—See III. **Green pigment.**—See IV. **Ti oxide.  $TiO_2$  hydrate.**—See VII. **Grinding bodies.**—See VIII. **Surface-marking wood.**—See IX.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Separation and identification of sol rubber hydrocarbons.** T. MIDDLEY, JUN., and A. L. HENNE (J. Physical Chem., 1932, 36, 2880—2884).—The hydrocarbon in natural rubber can be separated into two portions, "sol rubber" (I) and "gel rubber" (II), by means of their different solubilities in a  $C_6H_6$ -EtOH mixture. The sol rubbers have the same characteristics independently of their source; they are unstable at room temp. and are degraded slowly at the temp. of solid  $CO_2$ . No tendency to form (II) was observed. Extreme milling degrades both (I) and (II). Purified (I) can be vulcanised to yield a superior product. Types (I) and (II) are mutually sol. A method of determining the standard pptn. point of the rubber and a fractionation procedure are given. E. S. H.

**Quebrachitol and the lipin from *Hevea* latex.** R. M. WOODMAN and E. RHODES (J. Rubber Res. Inst. Malaya, 1932, 4, 153—155).—Consideration of these substances as to possible applications leads to the conclusion that they are of no val. for the production of emulsions for spraying purposes. With lipin it is possible to form oil emulsions in which oil-in-water and water-in-oil dispersions are simultaneously present. D. F. T.

**Sulphuric acid as a [rubber] latex coagulant.** J. L. WILTSHIRE (J. Rubber Res. Inst. Malaya, 1932, 4, 94—103).—It is possible, with care, to produce rubber of good quality by the use of  $H_2SO_4$ . Only very dil. acid must be added (e.g., 1 fluid oz. per gal. of  $H_2O$ ) and this should be used in min. quantity, e.g., 2.7 g. of pure acid per lb. of dry rubber; the sheet or crêpe should be washed thoroughly with  $H_2O$  during machining. The cost is approx.  $\frac{1}{2}$  that of  $HCO_3H$  and  $\frac{1}{3}$  that of AcOH. D. F. T.

**Effect of accelerators and antioxidants on electrical characteristics and water absorption of vulcanised rubber insulation.** J. H. INGMANSON,

C. W. SCHARF, and R. L. TAYLOR (Ind. Eng. Chem., 1933, 25, 83—87).—Examination of typical 30% vulcanised rubber insulating compounds, representing 10 well-known accelerators and 12 commercial antioxidants, as to  $H_2O$ -absorption, sp. resistivity, sp. a.-c. conductance, dielectric const., and power factor, in the dry condition and after 5 days in  $H_2O$  at  $70^\circ$ , indicates that the choice of accelerator or antioxidant is not crit. as regards  $H_2O$ -absorption, but may be somewhat so as regards electrical characteristics in high-grade soft rubber insulation. The relative ease of  $H_2O$  absorption with the various accelerators and antioxidants is not a satisfactory index to the electrical stability. Of the accelerators tested, highest resistivity vals. are obtained with the thiurams; also with these and 1.35% S the power factor increases with time of vulcanisation. The variations in electrical characteristics with various antioxidants cannot be attributed directly to differences in chemical structure of these. D. F. T.

**Development of organic accelerators for rubber vulcanisation.** G. OENSLAGER (Chem. and Ind., 1933, 91—95).—Perkin Medal address.

**Importance of accelerators in the modern rubber industry.** H. L. TRUMBULL (Chem. and Ind., 1933, 95—98).

**Retarders of vulcanisation [of rubber].** R. DITMAR (Caoutchouc et Gutta-Percha, 1933, 30, 16,264—16,266).—The knowledge of the use of retarders to prevent scorching or prevulcanisation of mixtures containing rapid accelerators is indicated by reference to various patents. The results of an experiment illustrating the use of a proprietary retarder are recorded. A good retarder should be effective up to about  $100^\circ$  and lose its activity above this temp. D. F. T.

**A century of technical progress in the rubber industry.** N. A. SHEPARD (Ind. Eng. Chem., 1933, 25, 35—41).—A review. D. F. T.

**Rubber resins in varnishes.**—See XIII. **Rubber plantations.**—See XVI.

#### PATENTS.

**Manufacture of articles of or containing rubber or like compositions.** DUNLOP RUBBER CO., LTD., and D. F. TWISS (B.P. 385,430, 1.1.32).—Articles vulcanised with  $S_2Cl_2$  are improved by treatment with an agent capable of replacing Cl by S, e.g., with a sulphide or a thiosulphate. D. F. T.

**Manufacture of [elastic] bricks, slabs, sheets, and the like [for roads etc.].** G. W. BELDAM (B.P. 385,001, 7.5., 10.7., and 30.9.31).—Resilient, moisture-repellent material for flooring or paving comprises compounded rubber (80 pts.) and disintegrated wood (40—120 pts.) which has been waterproofed. D. F. T.

**Products from asphalt emulsions.**—See II.

#### XV.—LEATHER; GLUE.

##### PATENTS.

**Fatty acid amides.**—See III. **Azo dyes for leather.**—See IV.

## XVI.—AGRICULTURE.

**Organic matter and its condition in soil.** U. SPRINGER (Soil Res., 1932, 3, 39—70).—The formation, isolation, and properties of humic matter in soils is discussed. A. G. P.

**Method of fractionating the mineral matter of soil.** W. SIBIRSKY (Soil Res., 1932, 3, 77—90).—The soil sample is leached with  $\text{NH}_4\text{Cl}$  solution to remove the greater part of the adsorbed basis, filtered, washed with  $\text{EtOH}$  (or  $\text{MeOH}$ ) and then with  $\text{H}_2\text{O}$  to remove free  $\text{NH}_4\text{Cl}$ . The residue is washed through a 0.25-mm. sieve,  $\text{NaOH}$  is added, and, after cooling ( $5^\circ$ ),  $\text{Cl}_2$  is passed through the suspension. The chlorination is repeated and the mixture heated on a  $\text{H}_2\text{O}$ -bath and finally treated with a small amount of  $\text{H}_2\text{O}_2$  to remove any remaining  $\text{Cl}_2$ . The soil residue is separated by decantation, treated with  $\text{HCl}$ , filtered, washed, and oxidation is completed with the aid of  $\text{H}_2\text{O}_2$  and  $\text{K}_2\text{Cr}_2\text{O}_7$ . The filtered and washed residue is dispersed by careful addition of  $\text{NaOH}$ , and mechanically analysed by the pipette or the decantation method. The amount of mineral matter dissolved in the chlorination process from a no. of typical soil minerals was  $> 2$ —3%. A. G. P.

**Aggregate analysis as an aid in the study of soil structure relationships.** L. D. BAVER and H. F. RHOADES (J. Amer. Soc. Agron., 1932, 24, 920—930).—An elutriation method is described. The interpretation of results and the relationships between stability of structure and state of aggregation in soils are discussed. A. G. P.

**Soil algæ.** R. GISTL (Arch. Mikrobiol., 1932, 3, 634—649).—The algal flora of soils is controlled by physico-chemical soil conditions and depends largely on the relative ion concns. in the nutrient medium. Addition of  $(\text{NH}_4)_2\text{SO}_4$  to soils favours the development of *Diatomacea*. A. G. P.

**Microbiological basis of variations in soil acidity.** D. FÉHER with Z. KISZELY (Arch. Mikrobiol., 1932, 3, 609—633).—Variations in  $p_{\text{H}}$ ,  $\text{H}_2\text{O}$  content, and bacterial nos. in soils are recorded. The  $\text{H}_2\text{O}$  content is an important controlling factor. The practical difficulty of differentiating between direct and indirect relationships among the three factors is emphasised. A. G. P.

**Mobility of phosphoric acid in soil.** S. GERICKE (Z. Pflanz. Düng., 1932, 27A, 144—162).—The solubility of natural and fertiliser phosphates in soils is dependent on soil type and very largely on soil reaction. The distribution of fertiliser phosphate in the surface layer is rapid, but depth penetration is very slow. Superphosphate is more rapidly "fixed" by soils than are the less sol. basic slag and Rhenania phosphate. A. G. P.

**Utilisation of phosphorite and leucite by soils.** C. ANTONIANI and M. NICCOLINI (Giorn. Chim. Ind. Appl., 1932, 14, 490—491).—When 0.1—2% of phosphorite is mixed with neutral ( $p_{\text{H}}$  7.0—7.5) soil containing 30% of the saturation quantity of  $\text{H}_2\text{O}$ , about 2 mg. of assimilable  $\text{P}_2\text{O}_5$  per 100 g. of soil are liberated within 5—6 months; with a more acid soil ( $p_{\text{H}}$  6.3) the quantity liberated is about 3.5 mg. within 4 months and 4.5 mg. after 7 months. Leucite (0.2—4%) yields

about 5 mg. per 100 g. of assimilable  $\text{K}_2\text{O}$  within 2 months and 10 mg. after 6—7 months, these quantities being practically independent of the  $p_{\text{H}}$  of the soil.

H. F. G.

**Correlations between crop yields and the readily available phosphorus in soils as determined by Truog's method.** F. L. DAVIS and G. D. SCARSETH (J. Amer. Soc. Agron., 1932, 24, 908—920).—Statistical data are recorded. The necessity of correlating experimental and field yields prior to the application of laboratory methods for determining available P is emphasised. The process applied to Truog's method is recorded. A. G. P.

**Theoretical relationships between crop yields and manuring, and its experimental proof.** M. J. VAN UVEN (Z. Pflanz. Düng., 1932, 27A, 162—193).—A mathematical discussion. A. G. P.

**Possibility of making brown coal available for plant nutrition.** J. GEILER (Braunkohlenarch., 1932, No. 36, 43—55; Chem. Zentr., 1932, ii, 2356).—Bacterial elimination of  $\text{CO}_2$  from brown coal is possible, but not in quantity sufficient for the purpose. A. A. E.

**Grassland management.** E. G. DOERELL (Superphosphat, 1932, 8, 155—157).—Application of superphosphate to pastures treated with liquid manure considerably increased the protein content of the herbage as shown by the ninhydrin test on aq. plant extracts. A. G. P.

**Effects of fertilisers on seasonal production of pastures.** B. A. BROWN (J. Amer. Soc. Agron., 1932, 24, 898—908).—The total productivity of pastures (in food units) is markedly increased by suitable manuring, but the % total feed produced in any particular part of the season is not greatly altered. Notable differences in this latter respect are produced by varying the period of grazing. A. G. P.

**Influence of potassium nitrate on nodule formation and nitrogen fixation by clover.** E. W. HOPKINS, P. W. WILSON, and W. H. PETERSON (Plant Physiol., 1932, 7, 597—611).—Addition of  $\text{NO}_3^-$  to agar media decreased the no. and size of nodules developing on clover roots, and also induced nodulation on secondary roots but not on main tap roots near the crown. Where the  $\text{NO}_3^-$  supplied was  $<$  the  $\text{NO}_3^-$  requirement of the plants the balance was provided by fixation. With excessive  $\text{NO}_3^-$  supplies, fixation of N ceased although nodules actually formed. A. G. P.

**Response of iris to soil reaction.** J. H. GOURLEY (Plant Physiol., 1932, 7, 739—742).—*Iris germanica* grows best on neutral or alkaline soil. With increasing acidity growth diminishes and the proportion of tip-burn in leaves increases. A. G. P.

**Relation of nitrogen, phosphorus, and potassium to the fruiting of cotton.** M. NELSON and J. O. WARE (Arkansas Agric. Exp. Sta. Bull., 1932, No. 273, 75 pp.).—Fertiliser trials are recorded and the effects of the individual nutrients on growth and productivity discussed. A. G. P.

**Cotton wilt. IV. Effect of fertilisers.** V. H. YOUNG, G. JANSSEN, and J. O. WARE (Arkansas Agric.

Exp. Sta. Bull., 1932, No. 272, 27 pp.).—Heavy applications of K fertilisers either alone or in conjunction with N and P markedly decreased cotton wilt and rust and stimulated vegetative growth and seed production.

A. G. P.

**Fertiliser requirements of sugar cane on Yazoo, very fine sandy loam in Louisiana.** A. M. O'NEAL, L. A. HURST, and S. J. BREAUX, JUN. (J. Amer. Soc. Agron., 1932, 24, 888—898).—The length of millable cane increased with the amount of N applied in mixed fertilisers up to a limiting val. Excessive N caused weakening and lodging of the cane.

A. G. P.

**Growth of tree seedlings in relation to light intensity and concentration of nutrient solution.** G. P. STEINBAUER (Plant Physiol., 1932, 7, 742—745).—The min. light requirement of plants cannot be lowered by increasing the concn. of the nutrient above the normal requirement. With high light intensity the response of plants to increased nutrient concn. was more marked. High concns. tended to produce greater root length. Lateral branching of roots was more evident in the less conc. media.

A. G. P.

**Effect of potassium nitrate on vigour and productivity of healthy and leaf-roll Green Mountain potato plants and their progenies.** O. BUTLER and H. L. MURRAY (J. Amer. Soc. Agron., 1932, 24, 881—887).—KNO<sub>3</sub> reduced the viability and productivity of potatoes infected with leaf roll, the effect being more marked with growth temp. of 20° than at 15°.

A. G. P.

**Incidence and control of apple scab (*Venturia inaequalis*) and apple mildew (*Podosphaera leucotricha*) at East Malling.** M. H. MOORE (J. Pomology, 1932, 10, 271—294; cf. B., 1931, 216).—Bordeaux mixture gave good control of scab on Cox's Orange Pippin, but was ineffective against mildew. Scab, mildew, and red spider were effectively controlled by 1 pre-blossom and 2 part-blossom applications of CaO-S. Best yields of fruit followed pre-blossom use of CaO-S even when subsequent treatments were omitted.

A. G. P.

**Control of fire blight by treatment of cankers.** R. C. THOMAS (Ohio Agric. Exp. Sta. Bimo. Bull., 1932, No. 159, 195—197).—Cankers are painted with a solution prepared by dissolving 9 lb. of ZnCl<sub>2</sub> in 1 qt. H<sub>2</sub>O, adding 3 oz. conc. HCl, and, when cool, pouring into 7 pints of methylated spirit.

A. G. P.

**Distribution and control of the great stinging nettle.** G. H. BATES (J. Min. Agric., 1932, 39, 912—922).—The development of colonies of nettles (*Urtica dioica*, L.) is favoured more by the presence of a loose soil covering than by a high N content of the soil. Control measures are described.

A. G. P.

**Sulphur-dusting experiments [on rubber plantations].** F. BEELEY (J. Rubber Res. Inst. Malaya, 1932, 4, 115—122).—The procedure adopted and the difficulties experienced in dusting operations, using 2 varieties of S dust, to combat the fungus *Oidium Hevea* are indicated.

D. F. T.

**Carbon and nitrogen contents of some natural covers [on rubber plantations].** C. G. AKHURST

(J. Rubber Res. Inst. Malaya, 1932, 4, 131—139).—The C and N contents of various cultivated and natural cover plants for growth between rubber trees are discussed with a view of estimating the ultimate relative benefit produced in the soil by such covers.

D. F. T.

**Quebrachitol and the lipin from *Hevea latex*.**—See XIV.

PATENTS.

**Manufacture of a field weed killer.** L. and F. MEYER (CHEM. FABR. L. MEYER) (B.P. 385,860, 10.6.32. Ger., 10.6.31).—A mixture of cryst. CuSO<sub>4</sub> and NaCl in proportions to form CuCl<sub>2</sub> is ground together at 100° for 3 hr. in a ball mill which can be heated. Alternatively, the mixed salts may be dried at 50° in vac. until a greenish-yellow product is formed, which is afterwards ground.

H. R.-D.

**Insecticides.**—See XX. **Insecticide.**—See XXIII.

## XVII.—SUGARS; STARCHES; GUMS.

**Theory and control of crystallisation [in sugar factories].** D. TEATINI (Bull. Assoc. Chim. Sucr., 1932, 49, 287—293, 297—328, 345—364).—An exposition of fundamental principles, with numerous graphs, and with special reference to beet-sugar boiling. For the control of supersaturation throughout the boiling process a new apparatus to be fitted to vac. pans is described, which records on a revolving drum the temp. of the boiling massecuite and that of ebullition of pure H<sub>2</sub>O under the same vac. From the difference between these temp. at any moment the Brix of the mother-liquor in the massecuite can be ascertained without calculation by means of a transparent chart which moves over the surface of the drum in accordance with the movements of the pencil recording the temp. of ebullition of pure H<sub>2</sub>O. The reading of the chart requires an approx. knowledge of the purity of the mother-liquor, such as an operator could estimate from the stage of boiling at which an observation is made. If boiling is carried out at const. temp. the chart can be graduated specially to indicate directly the coeff. of supersaturation of the mother-liquor in the massecuite.

J. H. L.

**Cane wax in raw and refined sugars.** C. F. BARDORF and J. A. B. BALL (J. Physical Chem., 1932, 36, 2940—2947).—The content of non-dialysable matter in the sugars investigated was 0.15—0.38%. This is classified roughly as (a) COMe<sub>2</sub>-sol., soft wax (m.p. 52°); (b) EtOH-sol., brittle wax (m.p. 82°); (c) H<sub>2</sub>O-sol., not fusible; (d) insol., infusible, containing N compounds. The wax is adsorbed by filter cloth, diatomite, and paper pulp. Wax (a) is present in greater quantity in refined than in raw sugar and appears to be the chief colloidal constituent of granulated sugar.

E. S. H.

**Acid inversion of sucrose in presence of neutral salts.** H. COLIN and A. CHAUDUN (Bull. Assoc. Chim. Sucr., 1932, 49, 365—370).—Inversion of 5% sucrose solutions by 0.1N-HCl is accelerated by KCl at concns. above 0.1N, but retarded at concns. below 0.05N. Increase in the concn. of sugar lessens the retardation. The inverting action of HCO<sub>2</sub>H or AcOH is retarded by salts of other weak acids and also by sulphates, but

nitrate and chlorides have little effect. The retarding influence of oxalates or acetates on inversion by HCl is  $>$  corresponds with the concomitant reduction in  $[H^+]$ . (Cf. A., 1928, 249, 1100; 1931, 802.) J. H. L.

**Standardisation of Fehling's solution.** A. E. CASE and W. J. PRICE (J. Inst. Brew., 1933, 39, 36—37).—Standard invert solutions, prepared by Lane and Eynon's and by Ling's methods, under varying temp. conditions and times of inversion, are equally accurate for the standardisation of Fehling's solution. Ling's method is the more rapid and is recommended. In the determination of sugars by Fehling's solution the Lane-Eynon "open-flask" method gives a higher titre than the "closed flask" method of Ling (B., 1931, 216).

R. H. H.

**Sugar cane.**—See XVI. **Lactic and bacteria and sugar fermentation.**—See XIX.

## PATENTS.

**Filters [for saccharine liquids].**—See I. **Glutamic acid.**—See III.

## XVIII.—FERMENTATION INDUSTRIES.

[Drying of hops.] Institute of Brewing Research Scheme. Report on the tenth and eleventh seasons' work at the experimental oast, 1930 and 1931. A. H. BURGESS (J. Inst. Brew., 1933, 38, 8—15; cf. B., 1932, 699).—Formulae are given for the time of drying of hops in relation to depth of loading, air speed, temp., and humidity. Hops dried at low temp. produce a mild-flavoured beer, at moderate temp. an aromatic flavour, and at higher temp. bitterness. The best hop colour is given by slowly raising the temp. of drying, and by using 1 oz. of  $SO_2$  per 1000 cu. ft. during the first hr. Dried hops, covered with cloths and stored, remain sound, and moisture penetrates in the first week only to a depth of 1 ft. R. H. H.

**Preservative principles of hops.** XIV. Development of the antiseptic constituents of hops and of tannin during ripening. T. K. WALKER and J. J. H. HASTINGS (J. Inst. Brew., 1933, 39, 15—27; cf. B., 1932, 746).—During early ripening the rate of formation of  $\alpha$ -resin exceeded that of  $\beta$ -resin, but this order was later reversed. The aroma was most fragrant a week before picking. On September 8th total resin per cone had increased to four times that present on August 16th in "Early Bird" and to  $3\frac{1}{2}$  times in the case of "Canterbury Golding." Preservative val.  $10(\alpha + \beta/3)$  was maximal at the time of picking (Sept. 12th—Sept. 20th). Tannin increased to a const. val., at a rate relatively  $<$  that of increase in wt. of the cone. R. H. H.

**Comparison of moisture determinations in malt.** A. E. CASE (J. Inst. Brew., 1933, 39, 37).—A malt sample dried at  $99^\circ$  for 3 hr. in an electric oven shows a moisture content of 2.3% compared with 2.1% (means of 25 results) found by similar treatment in "protected"  $H_2O$ -ovens. R. H. H.

**Determination of nitrogen in yeast.** A. E. CASE and W. J. PRICE (J. Inst. Brew., 1933, 39, 35—36).—The Kjeldahl-Gunning-Arnold method gives lower results

than Thorne's modification of the Christensen-Fulmer method (B., 1932, 203). The amount of N found by the latter method varies with the amount of  $H_2O_2$  used and the age of the sample. The use of 100-vol.  $H_2O_2$  is recommended. The difficulty of the representative sampling of pressed yeast was overcome by grinding with anhyd.  $CaSO_4$ . R. H. H.

**High-temperature fermentation.** E. M. HESS and F. BRETSCHNEIDER (Brauer Hopfen-Ztg. Gembrinus, 1932, 59, 126; Chem. Zentr., 1932, ii, 2382).—Certain yeasts gave at  $10^\circ$  and  $13^\circ$  beers which in taste and odour were better than those obtained at  $7^\circ$ . A. A. E.

**Rôle of salicylic acid in fermentation.** G. DENYS (Bull. Assoc. Chim. Sucr., 1932, 49, 279—285).—Addition of small amounts of salicylic acid, e.g., 1 pt. to 40,000 or 50,000 pts. of must or wort, in accordance with F.P. 328,704, stimulates the activity of yeast and represses that of foreign organisms, particularly acetic bacteria. It has been found specially beneficial for distillery fermentations in tropical countries, increasing the yield of EtOH by 20—40%. J. H. L.

**Effect of filtration on results of examination of distillery mash.** W. KAMIENOBRODZKI (Przemysł Chem., 1932, 16, 253—256).—The  $d$  of fermented potato mash increases, and the EtOH content diminishes, with the content of particles in suspension; it is therefore essential that the fermented mash be thoroughly filtered before making tests for efficiency of fermentation. R. T.

**Acidity of fermenting distillery mash.** W. KAMIENOBRODZKI (Przemysł Chem., 1932, 16, 256—257).—The acidity attains a max. val. at the height of fermentation, corresponding with max.  $CO_2$  content. R. T.

**Determination of sulphurous acid in musts, red and sweet wines.** P. PHOTIADIS (Z. anal. Chem., 1932, 91, 181—182).—The method recommended is to distil the wine with 5% of  $H_3PO_4$  ( $d$  1.30) in a current of  $CO_2$  until half the liquid is distilled off, the distillate being absorbed in  $H_2SO_4$ -free  $H_2O_2$ . The  $H_2SO_4$  produced is neutralised with aq.  $NH_3$ , the excess  $H_2O_2$  removed by boiling, and  $SO_4^{2-}$  determined volumetrically (cf. A., 1933, 136). J. W. S.

**Determining MeOH.**—See III. **Fermentation of cellulose.**—See V. **Standard Fehling's solution.**—See XVII. **N requirements of lactic acid bacteria.**—See XIX.

## PATENT.

**Sterilisation [of beers etc.].**—See III.

## XIX.—FOODS.

**Determination of benzoyl peroxide in flour.** J. R. NICHOLLS (Analyst, 1933, 58, 4—7).—The flour is steam-distilled in the presence of HCl and  $CaCl_2$  and the distillate is saturated with NaCl and extracted with  $Et_2O$ . The  $Et_2O$  is removed in an air current at  $30^\circ$  and the residue is dissolved in  $COMe_2$  and oxidised by aq.  $KMnO_4$  in the presence of aq. NaOH. After evaporation of the  $COMe_2$  the solution is acidified with  $H_2SO_4$  and decolorised by  $H_2C_2O_4$ . Insol. fatty acids are removed by filtration, and BzOH is extracted with

Et<sub>2</sub>O and light petroleum and determined by oxidation with H<sub>2</sub>O<sub>2</sub> to salicylic acid (cf. A., 1928, 313).

T. McL.

**F.p. of pasteurised and sterilised milks.** G. D. ELSDON and J. R. STUBBS (Analyst, 1933, 58, 7—10).—There is a general tendency for the f.p. depression to be slightly decreased when milk is heated, but the change is so slight that it does not interfere with the val. of the test. The f.p. of a sample of milk heated at 110° for 30 min. was unaffected.

T. McL.

**[Use of the] Hortvet cryoscope [in milk analysis].** G. D. ELSDON and J. R. STUBBS (Analyst, 1933, 58, 27—28; cf. B., 1933, 40).—A lens, which is in focus about 4.3 or 4.4 cm. from the thermometer, is most satisfactory for observing the temp., otherwise there should be no deviation from the standard apparatus.

T. McL.

**Triacetin as adulterant in butter.** A. NEMBROT and M. CEOLA (Giorn. Chim. Ind. Appl., 1932, 14, 497—498).—Triacetin may be used to restore the Reichert val. and Zeiss refractometric index of adulterated butter to vals. falling within the legal limit. Fincke's method will indicate the presence of only considerable quantities of triacetin. In the method recommended, the sample is refluxed with about 50% EtOH for 1 hr., and the cooled solution is treated with an excess of KOH and conc. on the H<sub>2</sub>O-bath to ensure complete hydrolysis of the triacetin. The acidified solution is then steam-distilled and the AcOH in the distillate is titrated with 0.1N-NaOH. The results are about 5% low, but the method may be employed to determine 0.4% of triacetin in butter.

H. F. G.

**Cheese-ripening studies. Nitrogen requirements of lactic acid bacteria. I. Fractional analysis of various nitrogen sources used for determination of sugar-fermenting abilities of lactic acid bacteria.** B. A. EAGLES and W. SADLER. **II. Influence of defined nitrogen sources on sugar-fermenting abilities of lactic acid bacteria.** W. SADLER, B. A. EAGLES, and G. PENDRAY (Canad. J. Res., 1932, 7, 364—369, 370—377).—I. The prep. of 43 N sources is described, and their N distributions are determined. The N of peptic caseinogen digest broth consists of 55—63% proteose-, 19—25% peptone-, and 14—17% sub-peptone-N. These vals. are lower with smaller quantities of caseinogen or shorter periods of digestion. In tryptic caseinogen digest broth the sub-peptone and peptone fractions are, respectively, 70 and 28% of the total N.

II. The influence of 36 of the above N sources on the sugar-fermenting abilities of lactic acid bacteria is determined by measuring the titratable acidity after incubation with glucose, mannose, and lactose. The recorded data lead to previously reported conclusions (cf. A. 1932, 1169; B., 1932, 1052).

A. C.

**Cheese-ripening studies. II. Nitrogen requirements of the lactic acid bacteria.** W. SADLER, B. A. EAGLES, and G. PENDRAY (Biochem. J., 1932, 26, 1532—1535).—The influence of peptic caseinogen digest, proteose peptone (Difco), hydrolysed caseinogen (Difco), and "Bacto yeast extract" as N sources on the sugar-fermenting ability of 33 strains of lactic acid strepto-

cocci has been studied. When provided with yeast extract many of the streptococci produce a small amount of acidity from most of the carbohydrates employed. In order to bring about active and vigorous fermentation of fructose, glucose, mannose, galactose, and lactose these streptococci require a N source containing an appreciable amount of proteose-, peptone-, and sub-peptone-N. These requirements are best met by using peptic caseinogen digest.

S. S. Z.

**Determination of dry matter in tomato pulp.** K. TAXNER (Konserven-Ind., 1932, 19, 483—484; Chem. Zentr., 1932, ii, 2387).—Refractometric and direct determinations differed by 0.1—0.4%, and very seldom by 1% or more.

A. A. E.

**Pectin production.** W. R. BUFTON (Food, 1933, 2, 142—143).—The extraction of pectin from apple pulp is described. The optimum gelling power is obtained at  $p_H$  3.1—3.5.

T. McL.

**Some recent developments in low-temperature research.** (SIR) W. HARDY (Chem. and Ind., 1933, 45—49).—A lecture dealing mainly with the respiration of apples and the changes undergone by muscle during cold storage.

J. W. B.

**Evaluation of potatoes by feeding tests on cattle.** K. G. SCHULZ (Z. Spiritusind., 1933, 56, 6).—The relative merits of feeding with dry and wet potatoes and of the various auxiliary constituents of the diet used in such tests are discussed, with special reference to cows, horses, and pigs. The basal diet recommended includes herring meal, soya-bean meal, and washed chalk for pigs; this is supplemented with hay, summer straw, and palm-kernel cakes for cows.

J. G.

#### PATENTS.

**Apparatus for kneading of margarine and other edible fatty substances.** A. GERSTENBERG (B.P. 384,969, 11.10.32. Denm., 15.10.31).—The apparatus comprises a no. of units of different kneading devices clamped together in line and easily changed so that the best types for any purpose may be used. In any event the first section is a feeder that delivers at a regulable const. rate irrespective of any excess in the hopper.

B. M. V.

**Manufacture of tea.** (SIR) W. G. MCKERCHER (B.P. 385,493, 31.3.32. Ceylon, 9.12.31).—More rapid and better controlled fermentation is obtained by treatment in a machine which crushes, tears, and curls the withered leaf in one operation (cf. B.P. 385,443) with the production of less dust etc.

E. B. H.

**Glutamic acid. Sterilisation [of drinks, foods, etc.].—See III.**

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

**Rapid testing of ointments and chemicals for mercury.** E. TORNOW (Angew. Chem., 1932, 45, 707—708).—2 c.c. of 5% KOH solution and 2 c.c. of 25% Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution are added to 0.1 g. of ointment, the mixture is boiled, and a strip of Al foil dipped therein for ¼ min., washed, and wiped. If Hg is present the Al becomes coated with oxide.

A. G.

**Determination of moisture in tobacco.** C. J. RZYMOWSKA (Rocz. Chem., 1932, 12, 943—951).—

Schlöpfer's method of distillation with PhMe is preferred. R. T.

**Identification of phthalic esters in essential oils.** T. PAVOLINI (Riv. Ital. Ess. Prof., 1932, 14, 250—252; Chem. Zentr., 1932, ii, 2250).—The perfume is evaporated on the  $H_2O$ -bath until odourless, and the  $d$ ,  $n_D$ , solubility, and ester val. of the residue are determined. Phthalic and succinic esters react with  $KHSO_4$  with sublimation of the anhydride, but are distinguished by interaction of the former with phenols to give coloured substances. Other identification reactions are described.

A. A. E.

**Determining MeOH in mixtures.**—See III.

## PATENTS.

**Manufacture of disinfecting, antiparasitic, and insecticidal agents.** SOC. ANON. DES MINES D'ORBAGNOUX (B.P. 384,151, 16.3.32. Fr., 13.6.31).—Sulphonated mineral oils, particularly those containing S, are mixed with a hydroxy-acid (citric, tartaric) or a polyhydric alcohol (glycerol, sucrose, glucose) and a quantity of an insecticidal metal salt ( $CuSO_4$ ,  $PbSO_4$ ) equiv. to the sulphonic acid present. The OH-compound should be just sufficient to prevent pptn. and the mixture should be neutral or feebly alkaline.

C. H.

**Production of antiscorbutic preparations.** H. D. ELKINGTON. From NYEGAARD & Co. A./S. (B.P. 383,746, 17.11.31).—The Me groups of the meconine residue in narcotine are replaced by H (with or without further demethylation), e.g., by enzyme action or with 50%  $H_2SO_4$  at  $100^\circ$ , or by exposure to ultra-violet rays, or by oxidation with  $H_2O_2$  at  $15^\circ$ . Alternatively, cotarnine or demethylated cotarnine is condensed with normeconine. The products show antiscorbutic properties.

C. H.

**[Manufacture of] extracts obtainable from mammalian suprarenal glands.** PARKE, DAVIS & Co., Assees. of W. W. SWINGLE and J. J. PFIFFNER (B.P. 383,992, 28.8.31. U.S., 28.8.30).—The fresh suprarenal cortex is extracted with cold 95% and 80% EtOH, and the extract is freed from proteins, inert matter, and adrenaline with  $C_6H_6$ , from phospholipins with  $COMe_2$ , and from cholesterol and neutral fat with 70% aq. EtOH in presence of light petroleum. The 70% EtOH solution is conc. to two thirds vol., diluted with  $H_2O$ , and centrifuged. Adrenaline and other org. bases may be removed by concentrating the 70% EtOH solution, with addition of EtOH, to 95% EtOH and filtering through a zeolite; or by evaporating the 70% EtOH solution, dissolving the residue in  $Et_2O$ , and washing with enough 0.1N-NaOH to combine only with the adrenaline.

C. H.

**Preparation of vitamin-D.** N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 385,626, 20.4.31. Holl., 25.4.30).—Ergosterol is irradiated with ultra-violet light after passage through filters which strongly absorb rays of wave-length 313.2 m $\mu$  and/or 312.6 m $\mu$ .

E. H. S.

**Manufacture of stable barbituric acid compounds.** I. G. FARBENIND. A.-G., Assees. of (MRS.) F. FORBING (B.P. 384,176, 29.4.32. U.S., 29.4.31).—An alkali-metal (5 : 5-disubstituted) barbiturate is dissolved

in an anhyd. glycol; the solution may be sterilised by heating or a germicide may be added. Examples are Na 5-phenyl-5-ethyl-, 5 : 5-diethyl-, 5-cyclohexyl-5-ethyl-, or 5 : 5-diallyl-barbiturates in ethylene or propylene glycol.

C. H.

**Manufacture of stable [organic] antimony compounds.** CHEM. FABR. VORM. SANDOZ (B.P. 384,687, 26.6.31. Addn. to B.P. 343,898; B., 1931, 654).—Hydrated Sb oxides are dissolved in solutions of alkaloid salts of polyhydroxy-acids derived from aldoses, e.g., gluconates of emetine and quinine. (Cf. B.P. 372,143; B., 1932, 818.)

C. H.

**Fatty acid amides.** Sterilisation [of tobacco etc.].—See III. **Plaster bandages.**—See IX. **Oils for cosmetics.**—See XII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

**Azoimide anti-fogging substances.** A. SEYEWETZ (Brit. J. Phot., 1933, 80, 17).—Good substitutes for KBr in developers are aromatic compounds containing the  $N_3H$  radical, e.g.,  $C_6H_4.N_3H$ ,  $C_{10}H_6.N_3H$ . Thus  $p$ - $NO_2.C_6H_3.N_3H$  suppresses entirely the fog caused by a quinol developer containing NaOH. Much smaller amounts are required of these substances than of KBr. Ag images of a bluish-black colour are obtained. Neither  $N_3H$  nor a derivative thereof (where H is replaced by an aromatic residue) has any effect.

J. L.

**Perpetual-life film tank developer.** W. G. BARKER (Brit. J. Phot., 1933, 80, 16).—Formulae for a developer and strengthener (both metol-quinol) are given. After a preliminary use the standard of development is maintained by keeping the vol. const. by additions of the strengthener. Sludge is removed every 2 or 3 months.

J. L.

## PATENTS.

**Photographic emulsions.** I. G. FARBENIND. A.-G. (B.P. 385,545, 4.7.32. Ger., 4.7.31).—Panchromatic emulsions are much increased in sensitivity, without causing fog or instability, by incorporating small amounts of Ag salts of acids with anions containing a heavy metal, e.g.,  $Ag_2MoO_4$ , or  $Ag_2WO_4$ , together with a little aq.  $NH_3$ . Usual hypersensitisation processes with ammoniacal Ag or  $H_2O_2$  will cause fogging in such treated emulsions.

J. L.

**Manufacture of anti-halation layers for photographic materials.** I. G. FARBENIND. A.-G. (B.P. 385,832, 5.5.32. Ger., 8.5.31).—The layer consists of colloidal Ag, made by adding a reducing agent (e.g.,  $N_2H_4.H_2SO_4$ ) to aq.  $AgNO_3$  solution containing gelatin and  $NH_3$ . The proportions are such that the resultant colloidal Ag has a particle size giving the layer a spectral permeability between 550 and 700 m $\mu$ . The emulsion support is dyed blue or lavender, with e.g., Light Blue and Rhodamine 3B, to cover the absorption gap of the anti-halation layer. The dye is permanent, and of a colour suitable in projecting the finished film. The process is particularly applicable to films for the reversal process, the colloidal Ag being dissolved in the reversal bath.

J. L.

**Manufacture of light-sensitive films.** TECHNICAL COLOR MOTION PICTURE CORP., Assees. of L. T. TROLAND

(B.P. 385,293, 14.5.31. U.S., 23.5.30. Cf. B.P. 263,331 and 263,650; B., 1927, 157, 174).—Positive film is rendered especially suitable for cinematographic reproduction by the imbibition process, by sensitising it to a wide spectral range (comprising 3 zones), and incorporating 3 or more dyes selectively absorptive in those 3 zones (preferably not overlapping). The light-absorption is made greatest in the blue-green and least in the yellow zone. Then on exposure to white light 3 successive depths of image will be obtained, corresponding to regions of low, medium, and high density; the gammas in these regions are controlled by the proportions of the dyes. Thus an emulsion may be sensitised with erythrosin and/or Rose Bengal, and the filter dyes tartrazine, Rose Bengal, and magenta added. Emulsions having nearly equal contrast throughout the density range are obtained. J. L.

**Production of photographs on aluminium.** (MRS.) J. E. MOSSOR (B.P. 385,609, 19.6.31).—In processes in which images are obtained in dichromated gelatin films on Al supports, the images being dyed after development, the adhesion of the film to the Al and the deposition of the dye are improved by abrading the surface of the Al and etching it lightly with hot aq. NaOH, before it is coated with the emulsion. The emulsion is improved by the incorporation of egg-albumin. [Stat. ref.] J. L.

**Production of photographic prints.** W. P. LEUCH, and S. C. & P. HARDING, LTD. (B.P. 385,522, 28.5.32).—Blue prints are obtained with deeper blues and clearer whites by treating them with an oxidising agent after development. This is attained in one step by coating the back of the paper with the oxidising agent, which then soaks through the paper on development. Gelatin may be incorporated to check premature action. Suitable oxidants are  $H_2O_2$  (stabilised by  $H_2C_2O_4$ ,  $CaCl_2$ , or  $Na_4P_2O_7$ ),  $(NH_4)_2S_2O_8$ ,  $NaBO_3$ , chloramine-T, or  $BaO_2$  (if the emulsion contains a little  $H_2C_2O_4$ ). J. L.

**Cyanine dyes and emulsions.**—See IV. **Surface-marking wood.**—See IX.

### XXIII.—SANITATION; WATER PURIFICATION.

**Mode of action of gas-mask filters.** H. ENGELHARD and K. PÜTTER (Z. Elektrochem., 1932, 38, 906—911).—A comparison has been made between the efficiency of an active C gas-mask filter when the air was passed through it in one direction only (breathing in) and when the expired air was also passed through the filter. Mixtures of  $COCl_2$  and air were employed. The expired air was found to effect considerable regeneration of the C by removing  $COCl_2$ , but the  $CO_2$  in the expired air was partly adsorbed, so that the air breathed in was richer in  $CO_2$  than in the case of one-way filters. Data are given for various commercial filters which contain KOH as well as C. D. R. D.

**Refuse disposal.** E. W. SMITH (J. Inst. Fuel, 1932, 6, 88—103).—The possibility of using town's refuse as a fuel to raise steam for the generation of electricity is discussed and illustrated by descriptions of the destructor installations at Huddersfield and Gjentofte (Denmark). A. B. M.

**British practice in sewage disposal.** H. C. WHITEHEAD and F. R. O'SHAUGHNESSY (Inst. Chem. Eng., Jan. 25, 1933, 21 pp.).—The development of the various systems of sewage disposal is reviewed, with particular reference to the percolating filter, the activated-sludge process, and sludge digestion. A plea is made for the prevention of pollution by the formation of Rivers Boards for each watershed which could provide general expert supervision over existing small works and thus avoid the necessity of concentrating the work of sewage purification at a large main-outfall works. C. J.

**Sewage treatment in America.** L. R. HOWSON (Inst. Chem. Eng., Jan. 25, 1933, 16 pp.).—The present tendency is to supersede fine screens by a short period of sedimentation. The deposits are removed mechanically, the grit being washed and freed from the bulk of the org. matter which, together with skimmings of grease, is incinerated. The activated-sludge process is being largely adopted, large cities installing the spiral-flow air-diffusion system and smaller places the mechanical means of inducing surface aeration. Two-storey settling tanks are being replaced by plain tanks, with mechanical sludge-removal devices, and separate digestion chambers in which the raw sludge and surplus activated sludge can be successfully treated and subsequently dried on sand beds. C. J.

**German practice in sewage disposal.** K. IMHOFF (Inst. Chem. Eng., Jan. 25, 1933, 2 pp.).—Existing sewage farms to guard against overloading are installing artificial pretreatment plants. Screens are uncommon and grit chambers narrow and small in cross-section. Two-storey tanks are common, but are being replaced by separate tanks in order to facilitate sludge heating. Fish ponds for the final effluent serve a population of  $10^6$ . In the Ruhr district artificial impounding reservoirs covering 1400 acres contribute largely to the self-purification of the river without cost, as the interest on capital is met by the income derived from developed water power. C. J.

**Sewage plant rebuilt to treat rayon wastes.** U. F. TURPIN (Eng. News-Rec., 1932, 109, 780—782).—The original plant (Imhoff tank and contact beds) at Sparta, Ill., was grossly overloaded with wastes from a creamery and a rayon mill and has been replaced by a diffused-air activated-sludge installation. It has been found that relatively small amounts of casein make it impossible to maintain an activated sludge, but rayon wastes can be treated in conjunction with domestic sewage, the factor governing the relative proportions of each being the grease content of the waste. In this plant, the max. amount which can be treated successfully is 160 p.p.m. C. J.

**Detecting metal in dust.**—See X.

### PATENTS.

**Disinfectant and insecticide.** J. W. J. STEAD (B.P. 385,709, 11.11.31).—A mixture of liquid carbolic acid 5,  $CCl_4$  5, eucalyptus oil 10, aq.  $NH_3$  5, turpentine 25, and kerosene 50 pts., is claimed. H. R.-D.

**Clarification of water.**—See I.