

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUG. 17, 1928.

I.—GENERAL; PLANT; MACHINERY.

Calorimeter for determination of heats of combustion. A. EUCKEN and L. MEYER (Chem. Fabr., 1928, 177—179, 195—196).—The apparatus comprises a heavy hollow copper cylinder (250 c.c.) with copper lid, carrying the usual ignition apparatus terminating in a thin nickel wire. The material to be burnt (30—40 mg.) is mixed with 6—8 mg. of kieselguhr and pressed into a small briquette which is placed in a conical silver wire cage in the calorimeter and burnt in oxygen under atmospheric pressure. The temperature is recorded to the nearest one thousandth of a degree every $\frac{1}{2}$ min. until constant. A series of results showing the range of utility of the apparatus is given.

A. R. POWELL.

Apparatus for sampling powdered solids. J. VON MEERSCHIEDT-HÜLLESSEM (Chem. Fabr., 1928, 357—358).—A four-sided pyramid of sheet iron standing upright is provided with wings on each angle so as to direct material sliding down each face into a separate container. A funnel-shaped container above the pyramid is held centrally above its axis by a wooden staff which also closes the bottom of the funnel, and the whole is encased in a cylinder. The funnel is filled with the sample to be reduced, and the wooden plug lifted when the sample is automatically "quartered."

C. IRWIN.

Wetting [of powders] and its measurement. E. F. GRIEG (Fuel, 1928, 7, 136—138).—The "wetting" of a powder is measured by the rate of rise of the liquid up a column of the powder in a tube, the lower end of which just dips into the liquid. The tube is suspended from one arm of a modified Odén continuous-weighing balance, and its increase in weight determined at suitable intervals. The resistances of the column of powder to air flow and to liquid flow are also determined.

A. B. MANNING.

Device for the preparation of solutions of constant density. J. HAMOUS (Z. Zuckerind. Czechoslov., 1928, 52, 448).—An apparatus for diluting a liquid to constant density consists of a cylindrical vessel containing a float carrying a graduated spindle, which, according to its rise or fall, actuates a mechanism controlling the entrance of the diluting liquid at the point where the denser liquid is admitted.

J. P. OGILVIE.

Rapid evaporation at room temperature. E. JANTZEN and H. SCHMALFUSS (Chem. Fabr., 1928, 373—375, 390—392).—The principles involved in the construction of a high-capacity laboratory evaporator (40 litres of water per hr. at 15°) are discussed in detail. To attempt to remove water vapour by simple pump action is very uneconomical. It is better to use a

water-jet pump with a condenser. The connexion from the pump to the apparatus must be as short and wide as possible. A multitubular copper condenser is far more efficient than one of glass. The connexion from the evaporator to the condenser should be as wide as possible without any baffling arrangement to prevent splashing, which is guarded against by the low gas velocity and slight pressure drop. The still should be of simple construction of steel, copper, or aluminium. An apparatus of 30 litres still capacity constructed on these lines is described.

C. IRWIN.

Viscosity measurements and viscosimeters. S. ERK (Petroleum, 1928, 24, 830—834).—The viscosimeters of Couette, Engler, Vogel and Ossag, and Lawaczek are described and illustrated.

W. S. NORRIS.

PATENTS.

Mixing or emulsifying apparatus. J. MCGOGAN and J. HUNTER (U.S.P. 1,671,868, 29.5.28. Appl., 26.6.26. U.K., 4.7.25).—The apparatus comprises a chamber containing two interacting toothed rollers which serve as emulsifiers, and a feed hopper connected by means of a pipe to the emulsifying chamber. The discharge pipe from the latter is provided with an auxiliary branch by means of which the emulsion may be returned to the hopper and thence back to the chamber for re-treatment if desired.

A. R. POWELL.

Hardening of binder materials. [Manufacture of brake linings etc.] A. E. WHITE. From RAYBESTOS Co. (B.P. 292,371, 20.7.27).—Tape woven, e.g., from brass wire covered with asbestos is passed successively through a solution of a binding material, e.g., a 30% solution of gilsonite in gas oil, and the solvent is expelled by passing the impregnated tape through a heated liquid which does not dissolve or combine with the binder. The tape is cooled in an atmosphere of steam.

L. A. COLES.

Distillation of liquid mixtures. J. P. FISHER, Assr. to HEAT-TREATING Co. (U.S.P. 1,672,978, 12.6.28. Appl., 7.10.22).—The liquid passes downwards through a series of zones of increasing temperature, disposed one above the other. In order to facilitate the removal of vapours, the liquid is spread over an extended surface in each zone, and heat is applied to the liquid as it passes in a confined stream to the next zone below. The vapours evolved from each zone are condensed, and the heat of vaporisation is utilised to heat the liquid entering the cooler zone immediately above.

F. G. CLARKE.

Use of atmospheric pressure for regularly and continuously supplying liquid to [distillation]

vessels. L. GOURSAT and F. MICHAUD (F.P. 623,636, 25.10.26).—A closed supply container above the distillation vessel is connected with the latter by a pipe which just dips below the surface of the liquid. As the latter distils over, air enters the supply vessel by means of this pipe and delivers a corresponding volume of fresh liquid to the distillation vessel. A. R. POWELL.

Fractional precipitation of the products of distillation from gases. LURGI APPARATEBAUGES.M.B.H. (G.P. 447,062, 17.9.24).—The hot gas mixture obtained from destructive distillation processes is fractionally cooled, and the resulting mist at every stage of the cooling is removed in an electrostatic precipitation apparatus, the purified gas from any one stage being enriched in constituents of lower b.p. by being passed back into an earlier stage of the process. A. R. POWELL.

Air separator. T. J. STURTEVANT, Assr. to STURTEVANT MILL Co. (U.S.P. 1,673,343, 12.6.28. Appl., 2.8.27).—Materials are graded in a separator comprising an inner and outer casing having a settling chamber between them. This chamber is situated in the inner casing, which consists of an upper and a lower shell separated by a series of spaced vanes. Material accumulating in the latter is removed pneumatically. F. G. CLARKE.

Crucible. E. L. HAUMAN (U.S.P. 1,673,115, 12.6.28. Appl., 13.11.26).—In the upper edge of the crucible is a semi-circular outlet, in line with which is arranged a trough-shaped lip, the bottom being slightly below the lower part of the outlet. H. ROYAL-DAWSON.

Hot filter funnel. P. SEGEBADE (G.P. 447,136, 19.4.25).—A Büchner funnel for hot filtration is totally enclosed within a container of similar shape through which heating liquid is passed. A. R. POWELL.

Protection of water pipes. C. BÜCHER (B.P. 265,932, 10.1.27. Ger., 12.2.26).—Suitable apparatus for carrying-out the process of B.P. 260,233 (B., 1927, 240) is described. F. G. CROSSE.

Reversible regenerative furnace. J. S. ATKINSON, Assr. to OPEN HEARTH COMBUSTION Co. (U.S.P. 1,675,588, 3.7.28. Appl., 8.8.21. U.K., 22.9.13).—See B.P. 22,678 of 1913; B., 1915, 875.

Gas liquefiers for refrigerating plants. SILLER & RODENKIRCHEN G.M.B.H., and W. SILLER (B.P. 292,402, 15.10.27).

Optical pyrometer (B.P. 291,971). Corrosion of metallic parts (U.S.P. 1,671,173).—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Replaceable bases in the roofs of bituminous coal seams of Cretaceous age. E. MCK. TAYLOR (Fuel, 1928, 7, 127—128; cf. B., 1928, 288).—Examination of the roofs of such bituminous coal seams shows them to have undergone base exchange with solutions of sodium salts, the sodium aluminosilicic complex having subsequently been hydrolysed in fresh water. A. B. MANNING.

Replaceable bases in the roofs of bituminous coal seams of Tertiary age. E. MCK. TAYLOR (Fuel, 1928, 7, 129—130; cf. preceding abstract).—A few

specimens of the roofs of bituminous coal seams of Tertiary age have been examined. They exhibit characteristics similar to those shown by the roofs of other bituminous coal seams. A. B. MANNING.

Conditions and geological evidence of base exchange between the roofs of bituminous coal seams and solutions of sodium chloride. E. MCK. TAYLOR (Fuel, 1928, 7, 130—134; cf. preceding abstracts).—From considerations based on the type of fossils found in the roofs of coal seams it is concluded that base exchange may have taken place in three ways: (a) by deposition of the roof in salt water, (b) by the submergence of a fresh-water roof in the sea, and (c) by capillary action from subsoil water containing sodium chloride. If it is accepted that, although the modes of accumulation of the plant residues may have been widely different, the roof conditions have been the final factor in their transformation into coal, it becomes possible to reconcile the opposing theories of the "drift" and "in situ" origin of coal. A. B. MANNING.

Brown coals of the Cheliaba region. G. L. STADNIKOV and N. F. PROSKURNINA (Trans. Karpov Inst. Chem., 1926, No. 5, 95—99).—Cheliaba coals, although externally similar to typical coals, are classed as brown coals; they contain C 71.51—76.05, H 4.40—4.97, N 1.32—2.00, S 0.84—3.53, O 16.49—20.61%, and yield moisture 16.9—19.0, ash 7.3—23.4, volatile matter 44.1—52.3, coke 47.7—55.9, S 0.75—2.52, resins 0.8—3.2%. CHEMICAL ABSTRACTS.

Resins of Wealden lignite. R. WIGGINTON (Fuel, 1928, 7, 118; cf. B., 1927, 3).—The ether-soluble resin of a sample of Wealden lignite, amounting to 1.5% of the original material, has been fractionated by extraction of the ethereal solution with dilute solutions of ammonium carbonate, sodium carbonate, and potassium hydroxide, in succession, followed by precipitation of part of the neutral resin by the addition of light petroleum. Each fraction has been analysed. A silver salt, prepared by the addition of silver nitrate to a neutral solution of the potassium salt of one of the resin acids, has also been analysed. A. B. MANNING.

Thermal insulation of retort settings. A. J. DALE and A. T. GREEN (Gas J., 1928, 183, 37—42).—Different methods of improving the thermal insulation of retort settings are critically discussed. Regarding the insulation of horizontal settings the guiding principles of design should be: (a) to use a diatomite type of brick, (b) to include wall insulation within the wall, away from external disruptive influences and sufficiently distant from the inner face to prevent undue shrinkage and loss of insulating power, (c) to ensure a rigid tying of the inner and outer firebrick walls, (d) to guard against overheating of metal fittings, mouthpieces, etc., (e) to use insulating plugs in the sight holes, and (f) to apply for top insulation a 4½-in. course, on the outside of the roof for horizontal settings, but preferably one course below the top course for vertical-retort settings. Tongued-and-grooved shapes should be used, so that if the joints open, direct radiation from the hot face is obviated. When the design involves external insulation it is advisable to build in the insulating brickwork after the

setting has attained working temperatures. A considerable decrease in the thermal efficiencies of plants fitted with waste-heat boilers or external producers can occur unless the gas mains also are efficiently lagged.

A. B. MANNING.

Relation between low- and high-temperature carbonisation of coal. A. SHIMOMURA (Fuel, 1928, 7, 119—127).—Seven Japanese coals of different types have been carbonised in the laboratory at 500°, and the semi-cokes then further carbonised at 900°. The volatile matter content of a coal is of little value for determining its behaviour on carbonisation. Water of constitution is driven off during both stages of the carbonisation, coals of lower rank yielding more water during the first than during the second stage, whilst the reverse is true of high-grade bituminous coals. All the tar is driven off below 500°. The yield of ammonia in the first stage is only 10—25% of that in the second, the total yield representing about 20% of the total nitrogen. The volume of gas evolved from 500° to 900° is 2—2.5 times that evolved up to 500°; the former consists largely of methane and hydrogen, contains more carbon monoxide than dioxide, and no unsaturated hydrocarbons.

A. B. MANNING.

Geipert's method for the determination of the gas yield in therms from gas coals. K. BUNTE and W. ZWIEG (Gas- u. Wasserfach, 1928, 71, 629—631; cf. B., 1926, 938).—The method yields concordant results in the hands of different investigators using either the same or different sets of apparatus. The results agree closely with those of gas-works' practice. Some of the experimental details which require careful control in order to ensure concordant results are discussed.

A. B. MANNING.

Thomas recording gas calorimeter (Dept. Sci. Ind. Res., Fuel Res., Tech. Paper No. 20, 1928, 42 pp.)—The results of experiments of a fundamental nature carried out on the earlier models indicate that (1) with gas of constant calorific value the record made by the calorimeter is correct within about 1%; (2) when the calorific value alters, there is a lag of about 30 min. before the change appears to its full extent on the record, small or temporary changes being ignored or under-estimated; over long periods the average of the record agrees satisfactorily with values obtained on a Simmance machine; (3) variations of gas pressure, voltage of electric supply, room temperature, and barometric pressure, within reasonable limits, do not upset the performance. After these tests the instrument was altered in the heat-absorbing air meter, where disturbance of the water level was lessened by changes in the construction of the vanes and of inlet and outlet ports, in the constant-level water feed where incoming water was made to splash on to the overflow weir, and in the burner which was constructed of silica instead of metal. Finally, changes were made in the recording mechanism by which it became more sensitive and less liable to lag and to irregular working. Tests on the modified apparatus indicate that (1) a steady reading can be obtained about 20 min. after lighting the burner, and if the calorific value of the gas is changed this will appear within about 4 min., but only to its full extent after

about 15—20 min.; at more frequent intervals than this changes are not recorded to their proper value, although the periods and the average will be correct; (2) fluctuations in the speed of the driving motor have small permanent disturbing effects, but may cause larger temporary errors; (3) changes in the level of the water in the tank must be avoided as far as possible, as a decrease of even 0.1 in. produces a rise of about 18 B.Th.U. on the chart; (4) the recording mechanism is satisfactory, and no troubles were encountered in other mechanical parts; the instrument is more accurate than water-flow or differential-expansion types, although its time lag is greater.

R. H. GRIFFITH.

Determination of dust and ash [fixed carbon] in tar. R. ARBEITER (Chem.-Ztg., 1928, 52, 529).—The tar sample is placed on a thick filter paper supported by a porcelain crucible (with no bottom) which rests on a glass tripod. The whole is placed in a large beaker containing carbon tetrachloride, and having resting on it a round-bottomed flask through which cold water flows. Heating is by an electric hot plate, and extraction is complete in 30—40 min. The filter is then dried, weighed, and, if desired, burnt.

C. IRWIN.

Liquid-phase cracking [of hydrocarbons]. A. DOBRJANSKI and A. MUREJEVA (Neftjanoe Chozjajstvo, 1927, 12, 411—413; Chem. Zentr., 1927, II, 1524).—Machine oil, cylinder oil, vaporising oil, and paraffin have been cracked in the liquid phase at 405—415°/atm. As the decomposition progresses, the quantity of ethylene and of heavier hydrocarbons decreases and that of saturated hydrocarbons increases. The density of the liquid distillate progressively increases, whilst the content of hydrocarbons of b.p. up to 150° decreases. Possibly during the earlier stages of decomposition the longest side-chains of the cyclic hydrocarbons are split off, this being followed by a different decomposition leading to the formation of saturated hydrocarbons. Pumice, coke, copper filings, and iron filings have no effect on the process of cracking.

W. S. NORRIS.

Cracking [of mazout] under low pressure. K. KOSTRIN (Neftjanoe Chozjajstvo, 1927, 12, 39—45; Chem. Zentr., 1927, II, 1523—1524).—Paraffin base mazout from Surachany has been cracked at 480—490° and 2.5—3.0 atm. in a tube furnace, which also served for the subsequent distillation, during which 33.5% of distillate was collected. The residue, 59.2%, had a setting point 12° lower than that of the original mazout, and was suitable for use as fuel oil. The distillate had initial b.p. 90°, and 39% was volatile up to 150°. The gasoline obtained therefrom contained 13% of unsaturated hydrocarbons; this value was halved and the boiling range raised by treatment with Florida earth, which polymerised the unsaturated constituents. Sulphuric acid produced a similar but even greater effect, and could not be used as a refining agent.

W. S. NORRIS.

Refining of mineral oils. F. SCHWARZ (Petroleum, 1928, 24, 803—810).—In place of solutions of alkali naphthenates, which are capable of dissolving easily emulsifiable and/or gum-forming hydrocarbon constituents of light lubricating oils, the author makes use of solid soaps upon which gum-forming unsaturated

constituents are adsorbed (cf. G.P. 403,134—5; B., 1925, 64). The use of this reagent may supplant or supplement refining by means of sulphuric acid, or it may replace the usual treatment (following acid) with alkali hydroxide solution or with dry adsorbents. Treatment with precipitated soaps, if used in conjunction with acid refining, preferably follows the latter, although the soap treatment can be given first. W. S. NORRIS.

Manufacture of "contact substance" [from petroleum distillates]. B. VLASSENKO (Neftjanoe Chozjajstvo, 1927, 12, 542—549; Chem. Zentr., 1927, II, 1525).—A report dealing with the large-scale preparation of "contact substance," consisting of about 40% of naphthenesulphonic acids, 2% of sulphuric acid, and 15% of oil and water, obtained as a by-product in the refining of petroleum distillates with fuming sulphuric acid. W. S. NORRIS.

Transformer oils and their brown deposit. G. L. STADNIKOV and Z. I. VOZZINSKAJA (Trans. Karpov Inst. Chem., 1926, No. 5, 109—126).—The brown deposit in used transformer oils is formed by condensation of the resinous with organic compounds formed by oxidation of the oil. Sulphonic acids remaining in insufficiently washed refined oils catalyse the condensation. The behaviour on oxidation of oils treated with 90% and fuming sulphuric acid in the presence or absence of naphthalenesulphonic acids supported this view.

CHEMICAL ABSTRACTS.

Valenta's reaction in relation to its use with gasolines and benzene-benzene mixtures. R. WEL-
LER (Petroleum, 1928, 24, 768—773).—The solubility of benzene, amylene, cyclohexane, and octane in 1.5 vols. of methyl sulphate at 20°, respectively, is 100, 72, 13.5, and 5.5%. The soluble (9%) and insoluble portions of Kahlbaum's "normalbenzin" are practically identical with the original material. Between —20° and +60° the variation in solubility of a commercial gasoline is linear with the temperature. Experiments are recorded which show that treatment of methyl sulphate under a variety of conditions will effect only a partial separation into soluble and insoluble portions. Methyl sulphate is therefore useless as a means of investigating mixtures, or of determining the content of aromatic hydrocarbons or other anti-knock constituents of a commercial gasoline. W. S. NORRIS.

Viscosity-gravity constant of petroleum lubricating oils. J. B. HILL and H. B. COATS (Ind. Eng. Chem., 1928, 20, 641—644).—From data previously published (cf. Hill and Ferris; B., 1926, 84) on the physical properties of the viscous fractions from various types of crude petroleum, a mathematical relation has been worked out between Saybolt viscosity and sp. gr. which gives a constant which has a calculable value for any oil and is an index of its chemical composition. As the value is higher or lower so the character of the oil becomes more naphthenic or more paraffinic, respectively. Furthermore, the higher the constant, the greater is the viscosity change with temperature. The relations hold for distillates of fairly narrow boiling range and for blended oils from components of widely different viscosities, but fractions from a mixture of several crudes may give abnormal results.

H. S. GARLICK.

Apparatus for determining the setting point of mineral oils. ANON. (Chem.-Ztg., 1928, 52, 529).—A Dewar flask containing ether is fitted with a tube in which the oil sample is placed and having the necessary thermometers and air connexions. The inlet air is dried with calcium chloride and connected to a reservoir of ether, and the outlet to a filter pump. The temperature difference between the ether and oil should not exceed 5°. Temperatures are easily controlled, and can be obtained as low as —40°. C. IRWIN.

Oxidation of "vaseline" oil. G. S. PETROV, A. J. DANILOVICH, and A. Y. RABINOVICH (Trans. Karpov Inst. Chem., 1926, No. 5, 81—89).—Purified (sulphuric acid) "vaseline" oil, when heated and refined with alkalis, is not oxidised when exposed to a current of air for 49 hrs. at 100° in presence or absence of zinc or iron. In presence of lead, oxy-acids insoluble in benzene are formed. Commercial "vaseline," when heated for 10 hrs. at 250—270°, shows signs of oxidation, but distilled oils (although not purified with sulphuric acid) do not. Oxidation of the oils, yielding acids soluble in benzene, is best effected below 100° in presence of cobalt naphthenate. CHEMICAL ABSTRACTS.

Determination of ceresin in ozokerite [mineral wax] and paraffin goudron. W. TOKMANOV (Neftjanoe Chozjajstvo, 1927, 12, 558—561; Chem. Zentr., 1927, II, 1525).—The ozokerite, dissolved in benzene, is treated for 1.5 hrs. at 60—65° with sulphosil (B., 1928, 602); the portion not absorbed is ceresin. This procedure gives higher values for ceresin, when applied to crude ozokerite, than the method of Lach ("Die Ceresinfabrikation," 1911, p. 60), but gives somewhat lower figures when applied to ozokerite previously freed from liquid constituents. In Lach's method, which entails heating at 200°, volatile hydrocarbons are lost which are not absorbed by the sulphosil; on the other hand, the separation of resin from ozokerite by sulphosil is more complete. Silica gel or Florida earth adsorbs resin already present, but, unlike sulphosil, does not polymerise and completely remove unsaturated, resin-forming constituents. If ozokerite is treated first with silica gel and then with sulphosil, separation into resin, resinifying constituents, and ceresin is effected.

W. S. NORRIS.

Solid-solution formation in mixtures of paraffin waxes. L. D. MEYERS and G. STEGEMAN (Ind. Eng. Chem., 1928, 20, 638—641).—Equilibria existing between mixtures of waxes separated from one another by a series of fractional distillations were determined from the temperatures at which crystals first appeared when melts were slowly cooled. The crystals varied in composition according to the original mixture from which they separated—a characteristic of solid solutions. The purest wax obtainable possessed no definite m.p. Results suggest that wax mixtures form a complete series of solid solutions the freezing points of which lie between those of the components. Intersolubility of wax and oil is low up to about 10° below the m.p. of the wax. Increased solubility at high temperatures accounts for the great loss of wax in the later stages of the sweating process. Purification of crystals separating from fused wax-oil mixtures by a series of pressings was resorted to. Since a wax containing 2.5% of oil produced an oil spot on

filter paper, whilst a wax to which 2% of oil had been added could be subjected to 3000 lb./in.² pressure without doing so, it is concluded that the oil is retained in the form of a solid solution. H. S. GARLICK.

Calorimeter. EUCKEN and MEYER.—See I. **Activated silica gel.** TYTSCHININ and TOKMANOV.—See VII. **Preservation of wood.** VORONOV.—See IX.

PATENTS.

Treatment of coals, ores, etc. mainly composed of particles of small sizes. L. HOYOIS (B.P. 279,447, 18.10.27. Belg., 19.10.26).—The material is separated into sharply distinct grades by an apparatus comprising trough washers and a specially designed upward-current washer, the general principle of the method of separation being first to divide the material into two portions of less and greater density, respectively, than the average, and then to subdivide these, separating from the former all particles of density greater than the permissible limit for the light particles, and from the latter all particles of density less than the permissible limit for the heavy particles. These two portions are then combined to form a middle portion. A. B. MANNING.

Production of stable suspensions or pastes of coal. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 292,217, 11.3.27).—The finely-ground coal is mechanically dispersed in an alcohol, *e.g.*, crude butyl alcohol, with the addition of a basic substance (sodium hydroxide, pyridine, aniline) as stabiliser. The stability of the suspension may be further improved by the addition of a small quantity of mineral oil. A. B. MANNING.

Preparation of solid fuels for burning in pulverised form. L. B. GREEN, Assr. to BORDEN CO. (U.S.P. 1,673,114, 12.6.28. Appl., 20.11.26).—The fuel is pulverised, then briquetted, and the surfaces are freed from loose fuel before delivery to a burner. H. ROYAL-DAWSON.

Production of briquettes. W. T. MILLER, Assr. to U.S. COAL MANUF. CO. (U.S.P. 1,670,865, 22.5.28. Appl., 10.10.23).—Finely-divided carbonaceous material is mixed with a binding and waterproofing composition, consisting of a mixed solution of a soluble silicate and glucose or dextrin, the latter ingredient having been partially precipitated by means of alcohol. Sodium chloride or other volatilisable salt may also be present. The plastic mass is moulded under pressure and baked. F. G. CLARKE.

Manufacture of fuel briquettes. H. H. HANSARD (U.S.P. 1,675,266, 26.6.28. Appl., 23.6.24).—A mixture of pulverised coal and a liquid hydrocarbon is heated to remove the constituents of low b.p. of the latter, and the resulting mass is briquetted. H. ROYAL-DAWSON.

Composition fuel [briquettes]. H. V. T. MILLS and V. D'O. NOBLE (B.P. 291,958, 8.8.27).—A mixture of small coal, cement, sawdust, and creosote is used. F. G. CROSSE.

Active carbon. SOC. ANON. DES ENGRAIS ET NOIR ANIMAL (F.P. 623,455, 18.2.26).—Carbonised wood turnings are heated at 300–500° with a mixture of sulphuric and phosphoric acids. A. R. POWELL.

Active carbon. GES. F. CHEM. PROD. M.B.H. (Austr. P. 106,603, 29.9.22).—In the preparation of alkali sulphides from alkali sulphates a large excess of organic reducing substance is employed in the fusion, and the carbon resulting therefrom is obtained as a sludge after leaching out the soluble salts from the fused sulphide mass. A. R. POWELL.

Manufacture of activated charcoal or carbon. METALLBANK & METALLURGISCHE GES. A.-G., and E. SCHELLER (B.P. 292,039, 4.1.28).—Wood, peat, sugar, inactive charcoal, or other carbonaceous material is heated with sodium monoxide. The initial reaction proceeds automatically after being started by local heating; when the reaction has subsided the product is heated at 500–700°. The sodium monoxide may be used alone or in conjunction with other substances such as sodium chloride, hydroxide, or carbonate, the salts remaining at the end of the reaction being removed by washing. A. B. MANNING.

Distillation of coal and other solid carbonaceous materials. T. A. GOSKAR (B.P. 291,836, 1.3.27 and 29.6.27).—Bituminous coal (20–40 pts.) and a binding agent (8–15 pts.) consisting of a solid hydrocarbon of the paraffin or terpene series, *e.g.*, bitumen or resin, are reduced to a fine state of division and intimately mixed. Finely-ground coal waste, anthracite duff, or coke breeze, etc. (70–50 pts.) is then added, the mixture briquetted, and distilled in a retort internally heated by the passage of hot producer- or water-gas, with or without superheated steam. A. B. MANNING.

Distillation of solid carbonaceous materials. H. NIELSEN and B. LAING (B.P. 292,060, 4.12.26. Cf. B.P. 276,407, 287,037, and 287,381; B., 1927, 867; 1928, 356, 395).—Coal is subjected to the action of hot gases containing 2–8% of oxygen in order to modify its coking properties, and is then distilled in a current of an inert, gaseous, heating medium. The distillation may be carried out in two stages, first at a temperature not exceeding 600° and then at a high temperature (up to 1200°). The solid residue is finally gasified in the presence of steam. A. B. MANNING.

Distillation of bituminous substances, fuels, etc. R. HADDAN. From KOHLENVEREDLUNG A.-G. (B.P. 291,481, 20.1.27).—The finely-divided raw material is maintained in suspension in a stream of heating gas which is either itself a hydrogenating agent or into which hydrogen or other hydrogenating agent is introduced. In one method of working, steam is added to the heating gas, the temperature being such that the fuel interacts with the steam to produce water-gas. Pressures higher than atmospheric may be used to aid hydrogenation. A. B. MANNING.

Distillation of solid fuels. C. STILL (G.P. 446,173, 14.5.25).—The material is heated in long vertical retorts, and the volatile products formed are removed from the lowest part of the retort. The retort is heated in such a way that, in the region of the outlet pipe for the gases, a premature progress of the coking from the walls towards the centre is avoided. A. R. POWELL.

Retort for the distillation of bituminous solid fuels, in particular bituminous shale, coal, peat,

etc. G. MENELL (B.P. 291,550, 17.3.27).—A horizontal rotary retort is fitted throughout its length with a spiral web, made of suitably bent semi-circular sheet-iron vanes bolted or riveted together. Longitudinal plates are fitted between the spiral threads of the web, extending from the inner surface of the retort towards the centre. The retort is particularly adapted for the treatment of shale which has a tendency to cake.

A. B. MANNING.

Carbonisation of coal. S. DUNLOP. From M. J. McQUADE (B.P. 291,962, 14.6.27).—Carbonisation is carried out at 350—450° in a horizontal cylindrical retort along which the coal is alternately advanced a certain distance and retracted a shorter distance by means of a screw conveyor. A space is left above the coal, and the volatile products which collect therein are drawn off through suitably placed offtake pipes. A. B. MANNING.

Treatment of gases arising from the distillation of coal etc. C. COOPER (B.P. 291,832, 11.2.27).—The gases are first submitted to a process for reducing their moisture content (cf. B.P. 248,841; B., 1926, 428) and are then oil scrubbed for the removal of naphthalene. The degree of dehydration may be merely sufficient to secure the maximum efficiency of the subsequent oil scrubbing, or, if desired, may be carried a stage further in order to prevent condensation of water in the distributing mains.

A. B. MANNING.

Apparatus for enriching with oil the gas obtained by the complete gasification of carbonaceous fuel. REGENERATIVE COAL GASIFICATION SYSTEM, LTD., and M. W. TRAVERS (B.P. 292,410, 11.11.27).—The apparatus described in B.P. 210,356 (B., 1924, 244) is modified by placing the regenerating and carburetting chambers in series and passing the whole of the blow gas through them in succession. If desired, the carburettor may be divided into two chambers, the carburettor proper being followed by a superheater.

A. B. MANNING.

Gas generators. H. S. and W. S. MOORE (B.P. 292,408, 10.11.27).—A plant for the production of coal gas, water-gas, mixed gas, carburetted water-gas, or low-grade gas, according to the method of operation, consists of a vertical retort below which and in open communication with which is a water-gas generating chamber. The fuel is either partially or totally gasified; in the former case the coke is discharged through a semi-rotary extractor into a chamber in which it is quenched. The retort is heated by the blow gases which are burnt with secondary air and then pass through a series of annular superposed chequered recuperative heating chambers surrounding the retort. The water-gas produced during steaming passes up through the retort, aiding the carbonisation process and mixing intimately with the coal gas evolved. One or more of the recuperative heating chambers can be utilised as carburetting chambers if it is desired to enrich the gas.

A. B. MANNING.

Production of labile bitumen emulsions. RÜTGERSWERKE A.-G., and L. KAHL (B.P. 275,928, 18.3.27. Ger., 13.8.26).—Emulsions for tarring roads are produced by mixing a basic magnesium salt to a paste with water and then stirring into this the liquid tar or asphalt.

Such an emulsion can be diluted with water and applied to the road surface irrespective of temperature or weather.

A. B. MANNING.

Treatment of bitumen and other materials adaptable for insulating or dielectric purposes. D. ANDERSON & SON, LTD., and R. O. CHILD (B.P. 291,858, 16.8.27).—In order to free bitumen, tar, etc. from suspended matter and thereby improve its insulating properties, it is treated with a colloid, e.g., casein, in a partially coagulated state, and the mixture centrifuged. If necessary the material is heated during the process.

A. B. MANNING.

Manufacture of asphalt. E. A. RUDIGIER, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,673,533, 12.6.28. Appl., 19.8.22).—Hot petroleum residuum at about 216° is transferred to a heat-insulated chamber and air-blown without further application of heat.

C. O. HARVEY.

Decolorisation of acetone oils and wood spirit distillates. ÉTABL. LAMBIOTTE FRÈRES (F.P. 619,857, 14.12.25).—The coloured oils are heated under reflux with a metal chloride (chloride of calcium, magnesium, zinc, aluminium, iron, tin, or antimony) at ordinary or increased pressure; the coloured constituents are converted into resinous or tarry products, and the oil is obtained colourless by distillation. Examples are: heavy acetone oil (b.p. 125—200°) with anhydrous magnesium chloride, and a wood spirit fraction (b.p. 125—195°) with anhydrous ferric chloride. C. HOLLINS.

Catalytic cracking of heavy hydrocarbons, mineral oils, oil residue tars, and the like. E. ERLÉNACH, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,671,573, 29.5.28. Appl., 15.8.22. Ger., 22.9.21).—The formation of coke during catalytic cracking is prevented by circulating the heated oil repeatedly through a deep layer of the catalyst resting on a perforated support within the still. The vapours pass off through a dephlegmator, and a viscous residue is removed from the still.

C. HOLLINS.

Cracking of mineral crude oils. H. BLUMENBERG, JUN., Assr. to A. M. BULEY (U.S.P. 1,673,491, 12.6.28. Appl., 18.5.27).—Aluminium chlorosulphate is added to the oil, the mixture is heated to cracking temperature and distilled, and the distillate condensed.

H. ROYAL-DAWSON.

Production of olefines. A. W. NASH, A. R. BOWEN, and O. C. ELVINS (B.P. 291,867, 9.3.27).—Mixtures consisting substantially of olefines and paraffins are produced from gases containing carbon monoxide and hydrogen, in which the proportion by vol. of the former exceeds that of the latter, by passage at atmospheric pressure and at 280—300° over a granular catalyst consisting of a reduced mixture of the oxides of cobalt, copper, and manganese. The catalyst is prepared by precipitation of the oxides or ignition of the nitrates etc., followed by reduction with hydrogen at 400—420°, and may be revived after use by treatment with steam or by oxidation and subsequent reduction. E.g., a suitable catalyst is produced by the reduction of a mixture containing copper oxide, cobalt oxide, and manganese oxide in the proportions by wt.: 30:60:45. If the gases are in equal proportion (ordinary water-gas), or if

the hydrogen is in excess, the products are mainly paraffins. C. O. HARVEY.

Production of nitrogenous-base oils from hydrocarbon materials. H. K. IHRIG, Assr. to S. E. CAMPBELL and ASSOCIATED OIL Co. (U.S.P. 1,671,721, 29.5.28. Appl., 8.11.26).—The sulphur dioxide extract of mineral oil (especially Californian petroleum) contains most of the nitrogenous bases (pyridine, piperidine, quinoline) originally present. These bases are removed with dilute sulphuric acid and recovered. C. HOLLINS.

Separation of hydrocarbons. C. B. WATSON, Assr. to PURE OIL Co. (U.S.P. 1,673,854, 19.6.28. Appl., 26.12.25).—Cracking residues are forced by means of an inert gas through a conduit and atomised in an enlarged chamber, into which passes a further quantity of heated inert gas to maintain the temperature and promote turbulence in the material. The vapours are withdrawn and the unvaporised residue is recirculated, a portion of it being continuously consumed by a number of burners. C. O. HARVEY.

Refining of hydrocarbon oils. P. McMICHAEL, Assr. to HYDROCARBON REFINING PROCESS Co. (U.S.P. 1,669,944, 15.5.28. Appl., 12.1.25).—Petroleum oils and distillates are agitated with 10% of their volume of a solution of sodium hydroxide (*d* 1.1) saturated with sodium hyposulphite, then with 10% of their volume of a 10% solution of sodium chromate containing 1% of sodium hydroxide, and finally with a 1% solution of hydrochloric acid. Instead of the first-named solution the liquid obtained by treating with lime the solution produced by reducing sodium hydrogen sulphite with zinc dust may be used. A. R. POWELL.

Purification of mineral and naphtha oils. G. PETROFF (B.P. 291,823, 1.2.27).—In the production of light-coloured odourless oils, such as transformer or turbine oils, by treatment with sulphonating agents, more complete removal of unsaturated and resinous substances and greater ease of separation of the sulphonic acids from the diluted acid tars and of the alkaline liquor during subsequent alkali treatment are attained by mixing fatty acids, *e.g.*, oleic or stearic acid, with the oil to be refined. C. O. HARVEY.

Manufacture of fuel briquettes [from peat]. L. RUDEMAN (U.S.P. 1,674,179, 19.6.28. Appl., 30.4.27).—See B.P. 236,366; B., 1925, 699.

Dewatering peat. G. A. SCHROTER (B.P. 292,661, 18.3.27).—See U.S.P. 1,625,058; B., 1927, 577.

Absorbent catalyst. A. GODEL, Assr. to SOC. DE RECHERCHES ET D'EXPLOIT. PÉTROLIFÈRES (U.S.P. 1,674,897, 26.6.28. Appl., 8.12.25. Fr., 9.12.24).—See B.P. 244,461; B., 1927, 133.

Production of carbon electrodes. I. SZARVASY (U.S.P. 1,675,674, 3.7.28. Appl., 16.3.27. Ger., 15.11.26).—See B.P. 284,818; B., 1928, 252.

Manufacture of gas from coal. M. W. TRAVERS and F. W. CLARK, Assrs. to TRAVERS & CLARK, LTD. (U.S.P. 1,674,000, 19.6.28. Appl., 5.3.23. U.K., 9.3.22).—See B.P. 198,777; B., 1923, 759 A.

Treatment of oils [hydrocarbons] with liquid sulphur dioxide. W. HESS, Assr. to ALLGEM. GES. F.

CHEM. IND. M.B.H. (U.S.P. 1,674,676, 26.6.28. Appl., 28.10.26. Ger., 29.3.26).—See B.P. 268,726; B., 1928, 181.

Separating from fluid hydrocarbons other hydrocarbons which precipitate at low temperature. N. O. BACKLUND, Assr. to BERGEDORFER EISENWERK A.-G. (U.S.P. 1,676,069, 3.7.28. Appl., 28.1.27. Ger., 16.8.26).—See B.P. 267,038; B., 1927, 357.

Separating the neutral oils in tars, tar oils, and pitches from acid constituents. H. WITTEK (U.S.P. 1,674,710, 26.6.28. Appl., 14.4.27. Ger., 12.8.25).—See B.P. 256,933; B., 1927, 901.

Pulverised fuel burners. H. CAMBERS (B.P. 291,842, 3.3.27).

Apparatus for generation of acetylene gas. J. HAWORTH (B.P. 291,872, 10.3.27).

Smoke-treating apparatus. C. J. SCHOBRONE (B.P. 292,380, 15.8.27).

Apparatus for generation of acetylene gas under low or high pressure. J. HAWORTH (B.P. 291,897, 17.3.27).

Gaseous mixtures (B.P. 281,675).—See VII. **Waterproof cement** (B.P. 291,988).—See IX. **Purification of mineral oils** (B.P. 291,817).—See XII.

III.—ORGANIC INTERMEDIATES.

Absolute alcohol in industry. J. L. GENDRE (Chim. et Ind., 1928, 19, 396—401; cf. B., 1925, 185, 563, 646).—Processes depending on the use of lime for the rectification of alcohol have now been abandoned, the method being retained only in the treatment of fermented bisulphite lyes in some paper factories. Even in this case losses of alcohol are excessive and there is risk of explosion. Lorette's process, using alcohol vapour and powdered lime, gives a turbid alcohol which must be freed from lime. Dehydration with a glycerol solution of potassium carbonate has been tried at Montières-les-Amiens, but the process requires the regeneration of anhydrous or nearly anhydrous glycerol—an operation involving the use of vacuum and live steam at 145—150°. Processes depending on the distillation of azeotropic mixtures are discussed and compared. Comparative costings show that rectification is practically as costly a process as the manufacture of absolute alcohol from phlegms, and, accordingly, methods based on rectification are probably destined to be displaced by the processes starting from phlegms, especially as the rectification stills are readily convertible into plant suitable for dehydration by the azeotropic methods.

R. BRIGHTMAN.

General process of sulphonation. L. GAY, M. AUMÉRAS, and P. MION (Chim. et Ind., 1928, 19, 387—395).—Sulphonation of naphthalene with sulphuric acid of any concentration is readily effected by using an "auxiliary" liquid, sufficiently volatile, *e.g.*, carbon tetrachloride or ligroin, which is also of low miscibility with water, to eliminate the excess of water and prevent the concentration of the sulphuric acid falling below the minimal concentration (π) (cf. A., 1926, 605). Alternatively, a current of chemically inert gas may be used.

In the former case the liquid distilling from the sulphonation vessel is returned continuously after separation of the water eliminated. With equimolecular quantities of naphthalene and 94% sulphuric acid, a 91–94% yield of naphthalenemonosulphonic acids is obtained in 3–5 hrs. at 140–150°, using carbon tetrachloride as auxiliary liquid. About 6–7% of sulphones are formed and the reaction product contains no free sulphuric acid. Excess of naphthalene does not appreciably increase the formation of sulphones, the velocity of the reaction, $C_{10}H_7 \cdot SO_3H + C_{10}H_8 = (C_{10}H_7)_2SO_2 + H_2O$ being relatively small compared with that of sulphonation. Mercuric sulphate does not catalyse the formation of sulphone but catalyses the side-reaction between water and carbon tetrachloride, affording hydrogen chloride which is always formed to some extent, especially if the temperature exceeds 150°. Excess of sulphuric acid also promotes the formation of hydrochloric acid, but no sulphones are found in the reaction product, which consists of a mixture of mono- and di-sulphonic acids, together with some free sulphuric acid. Thus, with 2 mols. of sulphuric acid (94%) and 1 mol. of naphthalene, the reaction product after 3 hrs. (or 9½ hrs.) at 140–150°, contains 18.5% (9.3%) of free sulphuric acid, 39.5% (19.9%) of monosulphonic acids, and 33.5% (67.6%) of disulphonic acids. Similar results are obtained with ligroin or carbon dioxide as the auxiliary. In the latter case the elimination of water is practically complete in 12 hrs. Use of an inert gas in place of an auxiliary liquid offers advantages in the avoidance of side-reactions and in heat economy, even if the thermal efficiency of the apparatus is low. The proportions of isomeric naphthalene-mono- and di-sulphonic acids formed have not been determined, but experiments indicate that the isomerides are obtained at corresponding temperatures in approximately the same proportions as in ordinary sulphonation. Unsuccessful attempts have been made to adapt the methods of Guyot (B., 1919, 811 A) and of Ambler and Gibb (B., 1919, 405 A) to the sulphonation of naphthalene.

R. BRIGHTMAN.

Industrial preparation of perylene. A. CORBELLINI and G. AYMAR (*Giorn. Chim. Ind. Appl.*, 1928, 10, 196–199).—Numerous experiments are described on the industrial preparation of perylene by the condensation of β -naphthol by known methods. The maximum yield of perylene, 19.3%, was given by the Hansgirk process (cf. B., 1921, 790), and the minimum, 13%, by the Marschalk process (cf. B., 1926, 869). No sensible improvement in the yield is obtained by the addition of sodium carbonate in the Pereira process. Contrary to the statement by Weitzenböck and Seer (A., 1913, i, 847), the condensation of β -dinaphthol to dihydroxyperylenes takes place in presence of aluminium chloride alone. The scanty yields obtained render it unlikely that the processes tested are capable of industrial application.

T. H. POPE.

“Contact substance” from petroleum distillates. VLASSENKO.—See II.

PATENTS.

Apparatus for the treatment of hydrocarbon compounds. E. R. HAMILTON (U.S.P. 1,671,423,

29.5.28; Appl., 17.11.24).—A closed container divided into concentric cells is used. The hydrocarbon enters the central compartment and flows alternately over and under transverse divisions in each cell in a more or less spiral course to the outermost cell, from which it is drawn off.

C. HOLLINS.

Manufacture of methyl alcohol. SOC. NAT. DE RECHERCHES POUR LE TRAITEMENT DES COMBUSTIBLES (F.P. 613,896, 31.7.25).—Carbon monoxide and hydrogen are passed at 160–180° and 150 atm. pressure over a catalyst consisting of precipitated copper hydroxide reduced in hydrogen at ordinary pressure; reduction begins at 140°, and the temperature must not exceed 450–500°.

C. HOLLINS.

Use of wood charcoal as catalyst in preparation of methyl alcohol from carbon monoxide and hydrogen. J. CAMPARDOU and J. VERGUES (F.P. 613,470, 3.3.25).—The gases are heated in a closed vessel at 300–400° in presence of wood charcoal.

C. HOLLINS.

Manufacture of methylene dichloride. I. G. FARBENIND. A.-G. (B.P. 283,119, 3.1.28; Ger., 3.1.27).—A mixture of methyl chloride (3 vols.) and chlorine (1 vol.) is passed at 120 litres per hr. through a tube (750–800 c.c.) at 360–380°. The product contains 92% of methylene dichloride, only 3% of chloroform, and traces of carbon tetrachloride, whereas methane yields greater proportions of the tri- and tetra-chloro-compounds. A catalyst may be used.

C. HOLLINS.

Manufacture of monocarboxylic acids. I. G. FARBENIND. A.-G. (B.P. 262,101 and Addn. B.P. 291,326, 22.11.26; Ger., [A] 27.11.25).—(A) Vapours of dicarboxylic acids with or without water vapour, or the corresponding anhydrides with water vapour, are passed over decarboxylating catalysts at 250–500°. Diluent moist gases may be added, and the gas mixture from the hydrogenation of unsaturated dicarboxylic anhydrides or from the oxidation of hydrocarbons etc. may be employed. Examples are: benzoic acid from phthalic anhydride; acrylic acid from maleic anhydride. (B) Oxides of zinc, cadmium, lead, bismuth, or mixtures containing one or more of these are selected as catalysts.

C. HOLLINS.

Manufacture of formic acid. A. SCHLOSS (G.P. 445,644, 18.9.24).—The kneading together of formate and acid and the distillation of the formic acid are combined in a single operation, under vacuum if desired, by use of a plant consisting of a two- (or more) chambered kneading machine with arms moving in opposite directions and sweeping the walls of the apparatus. The mass is thus evenly mixed and evenly heated, and the process occupies only one third of the usual time.

C. HOLLINS.

Purification of lactic acid. W. KLAPPROTH (B.P. 280,969, 22.11.27).—Crude 80% lactic acid (1000 pts.) is mixed with concentrated sulphuric acid (80 pts.) and anhydrous sodium sulphate (120 pts.) before extraction of the pure lactic acid content with ether.

C. HOLLINS.

Preparation of acetic anhydride. CONSORT. F. ELEKTROCHEM. IND., G.M.B.H., Assees. of R. MEINGAST and M. MUGDAN (G.P. 442,256, 25.6.25).—Acetic acid

vapour is heated to high temperatures, e.g., 600°, in containers of graphite or calcined carbon preferably in presence of a catalyst (fused phosphates). Ordinary carbon has a destructive action on the acetic acid.

C. HOLLINS.

Manufacture of esters. I. G. FARBENIND. A.-G. (B.P. 265,233 and Addn. B.P. 292,059, 31.1.27. [A] Ger., 29.1.26).— β -Hydroxyethyl esters are obtained by heating ethylene oxide etc. with organic acids in presence of (A) an alkali salt of the acid or (B) a mineral acid, a mineral acid salt, or a small quantity of an alkali salt of another organic acid.

C. HOLLINS.

Manufacture of secondary amines. W. SCHILT (Swiss P. 117,162, 29.6.23).—Halogen acid salts of trialkylamines or arylalkylamines are heated with acylating agents until no more alkyl halide is split off, and the resulting acylated secondary amine is then hydrolysed. E.g., dimethylaniline hydrochloride is heated, with gradual addition of acetic anhydride, to 170–180°. At about 155° methyl chloride begins to be evolved and acetic acid distils; after the reaction is complete, acetmethylanilide crystallises out on cooling, and may be recrystallised and hydrolysed. Hydrogen chloride, as gas or in acetic acid solution, may be added during the dealkylation.

C. HOLLINS.

Manufacture of $\gamma\delta\delta$ -tetramethylpentan- β -one [methyl $\beta\beta$ -dimethyl-tert.-amyl ketone]. E. R. LOCQUIN (F.P. 592,216, 29.3.24).— $\beta\gamma\delta\delta$ -Tetramethylamylene $\beta\gamma$ -glycol [$\beta\gamma$ -dihydroxy- $\beta\gamma\delta\delta$ -tetramethylpentane] is isomerised by concentrated sulphuric acid at 0° or by heating in alcohol with dilute 20% phosphoric, sulphuric, or oxalic acid under pressure at 150–160°, giving methyl $\beta\beta$ -dimethyl-tert.-amyl ketone, $\text{CMe}_3\cdot\text{CMe}_2\cdot\text{COMe}$, m.p. 63–64°, b.p. 167–168° (semi-carbazone, m.p. 207–208°), a substitute for camphor as a plasticiser especially for cellulose acetate.

C. HOLLINS.

Manufacture of acyl peroxides. N. V. INTERNAT. OXYGENIUM MAATSCHAPPIJ "NOVADEL," Assees. of T. KROEBER (G.P. 441,808, 7.4.25).—The acyl peroxide (e.g., benzoyl peroxide) is prepared in presence of finely-divided, preferably crystalline, substances, e.g., quartz, silica gel, calcium hydroxide or carbonate, insoluble phosphates, oxides, carbonates, or silicates, or other microcrystalline compounds. Thus benzoyl chloride is added to a well-stirred mixture of hydrogen peroxide with 33% of sodium hydroxide and powdered quartz or with iron-free calcium hydroxide. The fine division of the product makes it very suitable for cleaning solids.

C. HOLLINS.

Separation of 2 : 4-dinitrophenol from admixture with picric acid. SOC. CHIM. DE LA GRANDE PAROISSE (F.P. 575,302, 7.1.24).—The mixed nitro-compounds are heated with water and iron turnings, whereby the picric acid dissolves as ferrous picrate and the dinitrophenol melts and may be removed as a cake after cooling. Picric acid is recovered by acidifying with sulphuric acid the ferrous picrate solution, and the dinitrophenol is purified by dissolving it in alkali hydroxide, filtering, and adding sulphuric acid.

A. R. POWELL.

Manufacture of naphthalene derivatives. O. Y. IMRAY. FROM I. G. FARBENIND. A.-G. (B.P. 291,965, 16.6.27).—Sulphonation of 6-hydroxy-2-naphthoic acid with concentrated sulphuric acid at 50–80° gives a separable mixture of 7-sulpho- and 4-sulpho-derivatives. With oleum at 25–50° the 4 : 7-disulphonic acid is obtained, which may be hydrolysed by heating with diluted sulphuric acid to give 6-hydroxy-7-sulpho-2-naphthoic acid.

C. HOLLINS.

Manufacture of derivatives of 2-[hydr]oxy-naphthalene-3-carboxylic acid amide [2 : 3-hydroxynaphthamide]. I. G. FARBENIND. A.-G. (B.P. 289,037, 4.1.28. Ger., 22.4.27).—2 : 3-Hydroxynaphthoic acid reacts with thiocarbimides to give *N*-substituted 2 : 3-hydroxynaphthamides (cf. Kay, A., 1894, i, 76); e.g., phenylthiocarbimide gives the anilide in quantitative yield.

C. HOLLINS.

Production of wax-like polychloronaphthalenes. BRIT. DYESTUFFS CORP., LTD., J. B. PAYMAN, and W. GIBSON (B.P. 291,849, 292,056, and 292,058, 4.3.27).—(A) Chlorine is passed into molten naphthalene at 130–160° in presence of ferric chloride until 2 mols. have been absorbed, and the product is distilled at 230–260°/12–14 mm. The distillate is a hard, pale yellow, non-inflammable, insulating, wax-like substance, m.p. 131°, containing about 52.5% Cl, free from objectionable physical properties. (B) The naphthalene may be chlorinated in a solvent (carbon tetrachloride), and (c) the product may be mixed with α -chloronaphthalene in suitable proportions (4 : 1) to form a soft, crumbling solid which gives a good polish on wood, linoleum, etc.

C. HOLLINS.

Manufacture of wax-like mixtures of substances. BRIT. DYESTUFFS CORP., LTD., J. B. PAYMAN, and W. GIBSON (B.P. 292,057, 4.3.27).—The wax-like substances of B.P. 291,849 or 292,056 (preceding) are melted with sulphur to give a harder, whiter, and more porcelain-like mixture, m.p. about 111°, which adheres firmly to metallic and other surfaces.

C. HOLLINS.

Manufacture of side-chain-bearing polynuclear aromatic compounds or their sulphonic acids. I. G. FARBENIND. A.-G. (B.P. 267,132, 3.3.27. Ger., 3.3.26).—Polynuclear aromatic compounds (naphthalene) are heated at 60–95° with hydrogen sulphates of alkyl, aralkyl, or alicyclyl radicals, containing 3 or more carbon atoms, in presence or absence of condensing agents. Thus *n*-butyl alcohol is treated with chlorosulphonic acid to form *n*-butyl hydrogen sulphate, naphthalene is added, and the mixture is heated at 60–70° for condensation and finally at 95° for sulphonation.

C. HOLLINS.

Manufacture of α -naphthylamine-4 : 6 : 8-trisulphonic acid. E. HERTEL (Swiss P. 118,720, 12.12.25).—The reduction of 1-nitronaphthalene-4 : 6 : 8-trisulphonic acid with iron and acid gives improved yields in presence of copper, or salts of copper or nickel, or when a copper stirrer is used.

C. HOLLINS.

Manufacture of organic compounds containing a 1 : 3 : 5-triazine residue. GES. F. CHEM. IND. IN BASEL (Swiss P. 118,602–5, 7.10.24. Addns. to Swiss P. 103,430).—1 : 3 : 5-Triazine-2 : 4 : 6-tricarboxylic

chloride is condensed with 1 mol. of (A) 2:4-tolylene-diamine, (B) 2-amino-5-naphthol-7-sulphonic acid, (C) 1-amino-8-naphthol-3:6-disulphonic acid, or (D) *p*-phenylenediaminesulphonic acid. C. HOLLINS.

Nitration of anthrone and preparation of 2:7-dinitroanthraquinone. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 291,886, 11.3.27).—Anthrone is nitrated, mainly in the β -positions, with 95% nitric acid at 5°. The product, when boiled with acetic acid, nitrobenzene, dichlorobenzene, etc. until no more nitrous fumes are evolved, gives 2:7-dinitroanthraquinone.

C. HOLLINS.

Manufacture of benzanthrone derivatives. I. G. FARBENIND. A.-G. (B.P. 275,927, 7.3.27. Ger., 10.8.26).—Benzantrones are hydroxylated by oxidation (*e.g.*, with manganese dioxide) in 65–85% sulphuric acid (instead of the concentrated acid employed in the production of 3:3'-dibenzanthronyls). Hydroxylation of benzanthrone and of 4-chloro- and 4-methyl-benzantrones is described.

C. HOLLINS.

Production of benzanthrone derivatives. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 291,131, 21.2.27).—3-Methoxybenzantrones are obtained by heating a 3-nitrobenzanthrone with methyl alcohol and alkali, urea being added to destroy the nitrite formed;

derivatives containing other replaceable groups may be used in place of the nitro-compounds. The following benzantrones are described (numbering as in annexed formula):—3-methoxy-, m.p. 173°; 9-chloro-3-nitro-, m.p. 277°; 9-chloro-3-methoxy-, m.p. 262–263°; 3:9-dinitro-, m.p. 266°; 9-nitro-3-methoxy-, m.p. 318°;

3-nitro-9-methyl-, m.p. 280°; 3-methoxy-9-methyl-, m.p. 218°; 11-chloro-3-nitro-, m.p. 222°; 11-chloro-3-methoxy-, m.p. 225–226°; 3:9-dibromo-, m.p. 253°; 9-bromo-3-methoxy-, m.p. 275°; 3-chloro-9-bromo-, m.p. 252°; 9-chloro-3-bromo-, m.p. 262°; 3-chloro-9-nitro-, m.p. 280–281°.

C. HOLLINS.

Production of anthraquinone derivatives. I. G. FARBENIND. A.-G. (B.P. 272,469, 19.5.27. Ger., 8.6.26).—An amino-group is introduced in the 4-position into 1-amino-2-alkoxyanthraquinones by nitration and reduction (the 1-amino-group being protected by acylation if necessary) or by halogenation followed by amination. 1:4-Diamino-2-methoxyanthraquinone, m.p. 234–236°, is described.

C. HOLLINS.

Manufacture of hydrocarbons or oxygen derivatives thereof. I. G. FARBENIND. A.-G. (B.P. 268,775, 28.3.27. Ger., 3.4.26).—Alternate catalytic oxidation and decarboxylation are applied to the step-wise degradation of hydrocarbons or their oxygenated derivatives. Naphthalene and air, for example, are passed over vanadium pentoxide at 400°, then over cadmium pentoxide at 400°, and then over cadmium and aluminium oxides at 380° to give benzene; air may then be added and the mixture passed again over vanadic acid at 410° to form maleic anhydride and benzoquinone.

C. HOLLINS.

Manufacture of methylolureas [hydroxymethyl-carbamides]. G. WALTER (B.P. 284,272, 29.11.26.

Austr., 28.11.25).—Carbamides or thiocarbamides are condensed with formaldehyde (paraformaldehyde) in an organic solvent (alcohol) at 15° or slightly above in presence of caustic alkali.

C. HOLLINS.

Methylol [hydroxymethyl] compounds of amides. G. WALTER (B.P. 291,712, 29.11.26. Addn. to B.P. 284,272; preceding).—Acid amides (acetamide) are condensed with formaldehyde in an organic solvent (alcohol) at about 15° in presence of caustic alkali.

C. HOLLINS.

Apparatus for transformation of ethylsulphuric acid into alcohol or ethers. M. DUCHANGE, Assr. to COMP. DE BÉTHUNE (U.S.P. 1,674,891, 26.6.28. Appl., 19.1.25. Fr., 11.2.24).—See B.P. 229,272; B., 1925, 899.

Continuous purification of raw carbon disulphide. E. LEGELER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,672,948, 12.6.28. Appl., 14.4.25. Ger., 3.9.24).—See B.P. 238,489; B., 1925, 803.

Manufacture of oxygenated products from hydrocarbons or oxidisable derivatives of hydrocarbons. W. A. CASPARI (U.S.P. 1,674,589, 19.6.28. Appl., 6.11.26. U.K., 11.11.25).—See B.P. 263,201; B., 1926, 378.

Preparation of carbocyclic ketones with more than nine ring members. L. RUZICKA, Assr. to M. NAEF & Co. (U.S.P. 1,673,093, 12.6.28. Appl., 9.6.25. Switz., 16.6.24).—See B.P. 235,540; B., 1925, 738.

Acetone oils and wood spirit (F.P. 619,857).—See II.

IV.—DYESTUFFS.

PATENTS.

Manufacture of vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 291,546, 14.3., 5.9., and 11.10.27).—1:8-Dihydroxy- $\alpha\beta$ -dinaphthazine (N:N = 7:14), or its lower halogenation products (*cf.* B.P. 241,270; B., 1925, 954), or its acyl derivatives are vigorously chlorinated to give red vat dyes which may be purified by means of 96% sulphuric acid; they contain 6–10 atoms of chlorine. The chlorination is effected preferably in a diluent (acetic anhydride, nitrobenzene, trichlorobenzene, dichloroacetic acid) and in presence of a catalyst (iodine). At high temperatures the red vat dyes are produced directly; low-temperature products are treated with reducing agents, sulphuric acid, or potassium acetate, or sodium acetate and carbolic oil.

C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 291,878, 10.3.27).—1-Amino-2-aldehydoanthraquinone is condensed with a halogenated anthraquinone in presence of sodium acetate and copper powder. The primary condensation products are converted into vat dyes (preferably after purification by precipitation from sulphuric acid) by boiling with dilute acid or better with alkali. From 1-amino-2-aldehydoanthraquinone and α -chloroanthraquinone an orange vat dye, identical with the acidone, $C_{14}H_6O_2 \left\langle \begin{array}{c} NH \\ CO \end{array} \right\rangle C_{14}H_6O_2$ of G.P. 268,219, is obtained; 1-chloro-5-benzamidoanthraquinone

(brownish-orange) and 1-chloro-4-benzamidoanthraquinone (dark blue-violet) give similar products.

C. HOLLINS.

Manufacture of new [thioindigoid] vat dyes containing sulphur. W. CARPMAEL. From I. G. FARBENIND A.-G. (B.P. 291,825, 3.2.27).—An isatin α -chloride or α -anil is condensed with a 4-alkyl-, aralkyl, or arylthiol derivative of α -naphthol to give vat dyes. Examples are: 5:7-dibromoisatin α -chloride with 4-hydroxy-1-naphthyl methyl or *p*-tolyl sulphide (greenish-blue).

C. HOLLINS.

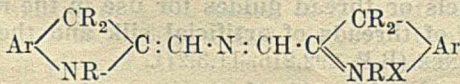
Preparation of soluble derivatives of indigoid vat dyes. I. G. FARBENIND. A.-G., Asses. of E. MÜNCH (G.P. 445,566, 14.3.25).—*N*-Acylated indigos etc. combine with sulphurous acid or sulphites to give water-soluble products which regenerate the starting materials by the action of cold ammonia, or are immediately hydrolysed to indigos by sodium hydroxide or hot ammonia. *E.g.*, *NN'*-diacetylindigo is heated for 30–60 min. under reflux with 40% sodium bisulphite solution and alcohol, more alcohol is added, and the yellow filtrate is evaporated in a vacuum to give a separable mixture of the white bisulphite compound of diacetylindigo and a deep yellow additive product. The pyridine salt of the bisulphite compound is formed when sulphur dioxide is passed into a suspension of *NN'*-diacetylindigo in aqueous pyridine; it may be converted into the sodium salt by action of sodium acetate and alcohol. Similar products are obtained from *NN'*-dibenzoylindigo, *NN'*-dicarbethoxyindigo, *N*-acetylrubrin, *N*-acetyl-3-indole-2'-thionaphthene-indigo (from *N*-acetylisanin and thioindoxyl), 4:4'-dichloro-1:1'-diacetylindigo, *NN*-diacetylisoindigo (from isoindigo, acetic anhydride, and a little β -chloroethyl *p*-toluenesulphonate). The bisulphite compounds may be applied to wool from an acetic acid bath, and developed with ammonia.

C. HOLLINS.

Manufacture of new triphenylmethane dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 291,887, 11.3.27).—An *o*-hydroxy-acid (salicylic acid, hydroxytoluic acid) is condensed with 4-benzylamino-benzaldehyde-3-sulphonic acid in sulphuric acid at 30–35°, and the product oxidised in the usual manner to give a chrome-violet of good light-fastness.

C. HOLLINS.

Manufacture of new dyes from indoline bases. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 291,888, 11.3.27).—By the action of a nitrite or nitrous ester on 2-methylene-1:3:3-trialkylindoline or its salts (2-methyl-1:3:3-trialkylindoleninium salts) in presence of acetic anhydride, yellow to orange-red basic dyes, probably of the annexed formula, are obtained. Thus

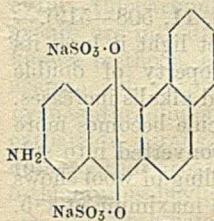


from 2-methylene-1:3:3-trimethylindoline, sodium nitrite, and acetic anhydride a light-fast, greenish-yellow dye for natural or artificial silk or tannin-mordanted cotton is prepared.

C. HOLLINS.

Manufacture of dyes of the anthracene series. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P.

291,851, 7.3.27).—Dyes of the indanthrone type are obtained by oxidation of a sulphuric ester of a leuco- β -amino- (or alkylamino-)anthraquinone having a free α -position *ortho* to the amino-group. The alkali salts of 2-amino-9:10-disulphatoanthracene, prepared by the action of chlorosulphonic acid and a tertiary base on leuco- β -acetamidoanthraquinone with subsequent hydrolysis, are oxidised with ferric chloride, or with ferric and ammonium or sodium sulphates, to indanthrone. Substituted indanthrones are similarly obtained from 6(7)-chloro-, 6:7-dichloro-, and 6(7)-methyl derivatives of leuco- β -aminoanthraquinone, and from leuco- β -methylaminoanthraquinone. The leuco-ester salt of 9-amino- α -naphthanthraquinone (annexed formula) gives a green vat dye of similar type.



C. HOLLINS.

Manufacture of anthraquinone derivatives. BRIT. DYESTUFFS CORP., LTD., A. SHEPHERDSON, W. W. TATUM, and F. LODGE (B.P. 291,814, 13.12.26).—One or both alkyl groups are removed from 1:4-dialkyldiaminoanthraquinones by heating with 100% sulphuric acid at 180–220°, or with 100% sulphuric acid and boric acid at 130°. From 1:4-dimethyldiaminoanthraquinone a bluish-violet dye for acetate silk is obtained.

C. HOLLINS.

Manufacture of derivatives of *N*-dihydro-1:2:1':2'-anthraquinoneazine fast to chlorine. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 291,552, 18.3.27).—Indanthrone is chlorinated in 85–95% sulphuric acid containing a catalyst (nitrogen oxides) at about 50°, and the resultant azines are reduced to chlorinated indanthrones. The products may be fractionally precipitated by dilution. Dichloroindanthrone, prepared thus, is completely fast to chlorine.

C. HOLLINS.

Azo dyes. J. HALLER, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,671,422, 29.5.28. Appl. 23.6.27. Ger., 28.6.26).—Clear violet shades are obtained by coupling in substance or on the fibre a 2:3-hydroxynaphthoic arylamide (β -naphthylamide, 2:5-dimethoxyanilide) with a diazotised arylamine \rightarrow aminonaphthol methyl ether, *e.g.*, 1:7- or 1:8-.

C. HOLLINS.

Treatment of dyes. D. GARDNER (B.P. 291,827, 5.2.27).—Insoluble dyes, particularly vat dyes, and also soluble dyes such as purpurin, rhodamines, etc., are treated with 5 or 6 pts. of tetrachloride or tetrabromide of tin or titanium, with or without addition of acetic acid or other organic solvent soluble in water, at 15° or higher temperatures (60–75°), with the formation of water-soluble products which can be used for direct dyeing.

C. HOLLINS.

Manufacture of thiocarbazides and carbazides of the naphthalene series. G. M. DYSON, F. A. MASON, and A. RENSHAW, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,673,498–9, 12.6.28. Appl., 12.5.27. U.K., 25.5.26).—See B.P. 278,037; B., 1927, 902.

Production of benzanthrone derivatives. K. ZAHN and W. ECKERT, Assrs. to GRASSELLI DYESTUFF

CORP. (U.S.P. 1,674,351, 19.6.28. Appl., 27.5.26. Ger., 27.6.25).—See B.P. 254,294; B., 1927, 837.

Anthraquinone derivatives (B.P. 272,469).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Properties of wool. L. MEUNIER and G. REY (J. Soc. Leather Trades' Chem., 1927, 11, 508—519).—When wool is subjected to ultra-violet light it loses its elasticity, fluorescence, and its property of double refraction. Its solubility in water and alkalis increases. Part of the sulphur in the wool molecule becomes more labile, and the sulphur liberated is converted into sulphurous acid. The curve for the swelling of wool shows minima at p_H 0.1 and 4.0—4.5 and a maximum at 1.5. Neither deaminated wool nor wool treated with quinone swells to the same degree as untreated wool. The second minimum is shifted towards p_H 5.0 by subjecting wool to ultra-violet light. Wool swells about equally in hydrogen sulphide and ammonia solutions, and to a lesser degree than in distilled water; the swelling is quite different from that produced by solutions of lime, baryta, or the strong alkalis, which seems to confirm Merrill's hypothesis (B., 1925, 18, 327). The primary amino-nitrogen content in wool is 0.57% as compared with 0.40% in silk fibroin, which shows less swelling at equal p_H values. D. WOODROFFE.

Hygroscopicity of cellulose esters. A. CAILLE (Chim. et Ind., 1928, 19, 402—406).—The relation between the hygroscopicity of nitrocellulose and its total sulphuric acid content is closely analogous to that previously observed between the sulphuric acid content and the absorption of basic dyes (B., 1926, 355). The hygroscopicity increases with the total sulphuric acid content, and samples containing the same amount of total sulphuric acid but different amounts of combined acid have practically the same hygroscopicity. Similar results were obtained with parchment paper which had been sulphurised by treatment with sulphuric acid, d 1.68, for 20 sec. (cf. B., 1927, 475). Examination of samples of cellulose acetate prepared as previously described (B., 1924, 937) shows that the fraction insoluble in acetone always contains less sulphuric acid than the original ester. Other things being equal, samples with the largest ratio of combined to total sulphuric acid are the most soluble in acetone and give the most viscous solutions. For a given sample of cellulose acetate, the viscosity of its solution in acetone decreases with the proportion of combined sulphuric acid in the sample. On fractional precipitation from acetone with aqueous acetone or water, cellulose acetate affords fractions differing widely in their sulphuric acid content, and experiments prove the existence of cellulose acetates soluble in 50% aqueous acetone. The sulphuric acid is therefore regarded as functioning otherwise than as a depolymerising agent, and in consequence of the dispersion in acetone the cellulose acetate is obtained in micelles of different sizes and varying sulphuric acid content. Bréguet's view that the largest micelles contain the highest proportion of combined sulphuric acid requires confirmation by ultrafiltration experiments. R. BRIGHTMAN.

Formation of poisonous gases in the manufacture of viscose artificial silk, and their removal. J. EGGERT (Chem.-Ztg., 1928, 52, 505—506).—Hydrogen sulphide is produced chiefly in the spinning process, during regeneration of the cellulose in the precipitating bath, and diffuses into the air. The production of only 1000 kg. of silk per day may cause the liberation of about 200 m.³ or 300 kg. in the air of the spinning room. Satisfactory designing of the spinning machines to prevent escape of the gas, admission of pure air from above, and removal of impure air from below the machines are recommended. Again, chlorine is liberated by acid-washing the yarn after bleaching it with hypochlorite liquors; this should be drawn off from the lowest part of the floor of the bleaching room and pure air admitted from above. B. P. RIDGE.

Saccharification of cellulose. LEONE and NOERA.—See XVII.

PATENTS.

Removal of fat from raw sheep's wool. I. G. FARBENIND. A.-G. (F.P. 623,103, 13.10.26).—The wool is treated with esters of carbonic acid either alone or admixed with other solvents for fats. A. R. POWELL.

Manufacture of hollow viscose fibres. N. V. NEDERLANDSCHE KUNSTZIJDEFABR. (G.P. 623,148, 14.10.26).—The viscose solution is spun directly into a bath containing 10% of ammonium sulphate and 12% of sodium carbonate, whereby part of the latter is absorbed by the fibres formed. The fibres then pass through a bath containing 15% of magnesium sulphate, 8% of sodium sulphate, 1% of zinc sulphate, and 9% of sulphuric acid. For a smooth fibre the material is sprayed with a 15% magnesium sulphate solution during its passage from the first to the second bath. A. R. POWELL.

Colloid product [from viscose]. S. A. NEIDICH (U.S.P. 1,670,162, 15.5.28. Appl., 3.3.26).—An extruded colloid product comprising a solid core filament and an outer imperforate tubular sheath having a crinkled highly reflecting surface is produced by passing viscose filaments from spinnerets into a coagulating bath containing 16.5% of sulphuric acid and 0.8% of sodium phosphate. The bath effects almost complete removal of sulphur from the viscose and causes rapid coagulation of the outer skin and retarded coagulation of the interior. A. R. POWELL.

Manufacture of a cellulose fibre product. I. F. LAUCKS, INC., ASSEES. OF G. DAVIDSON, H. F. RIPPEY, C. N. CONE, I. F. LAUCKS, and H. P. BANKS (B.P. 270,335, 30.4.27. U.S., 3.5.26).—See U.S.P. 1,622,496; B., 1927, 438.

Funnels or thread guides for use in the manufacture of threads of artificial silk and the like. E. WAGNER (B.P. 292,216, 11.3.27).

Cleansing of funnels of cuprammonium artificial silk spinning machines. O. VON KOHORN [F. O. KOHORN & Co.], and A. PERL (B.P. 290,560, 28.1.28. Austr., 14.5.27).

Machine for beating, bleaching, refining, etc. of paper and like stock. U. KIRCHNER (B.P. 271,457, 12.5.27. Ger., 22.5.26).

Manufacture of figured paper, cardboard, etc. L. CLEMENS (B.P. 279,805, 27.9.27. Ger., 28.10.26).

[Conical-type] refining engines for use in pulp refining. W. G. FRASER (B.P. 292,228, 14.3.27).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Effect of oxycellulose on tests for mercerisation. E. RISTENPART (Textilber., 1928, 9, 577—579).—Two samples each of mercerised and non-mercerised cotton yarns containing relatively small and large amounts of oxycellulose were prepared by immersing suitable yarns for $\frac{1}{2}$ and 3 hrs. respectively in solutions of bleaching powder (d 1.007) at 50°, and their behaviour to several reagents commonly used in testing cellulosic materials were compared. Mercerised yarn suffers a greater loss of strength, has a greater solubility in caustic soda and a greater affinity for methylene-blue, and attains a purer white than unmercerised yarn when overbleached. Although the original mercerised and unmercerised cotton yarns had nearly the same copper value, the copper values of the resulting overbleached mercerised yarns were 50—60% higher than those of the overbleached unmercerised yarns. In application of the Lange test to the unmercerised (original, slightly bleached, and overbleached) and the corresponding mercerised yarns, the resulting blue (iodine) colorations of the yarns resisted discharge by washing for 3, 2.3, 2, 7.5, 6.5, and 3.5 min. respectively. Mercerisation of overbleached cotton can be detected by microscopical examination of the fibres, but if oxycellulose is present the Lange test cannot be used for determining the degree of mercerisation.

A. J. HALL.

Organic peroxides as bleaching agents. HOOFT.—See XII.

PATENTS.

Dyeing with vat dyes. D. GARDNER (B.P. 291,828, 5.2.27).—Textile fibres, particularly wool and silk, are dyed in neutral or slightly acid solutions prepared by dissolving vat dyes in water-soluble organic solvents (*e.g.*, acetic acid, alcohols, ketones, esters, aldehydes, etc.), reducing the dyes by the addition of titanous chloride, and then suitably diluting with water. Alternatively, the dye liquor is prepared by the addition of titanous chloride to the product obtained by treating the vat dyes with anhydrous tetrachlorides or tetrabromides of metals of the fourth group in the periodic system, *e.g.*, titanium tetrachloride. It is preferable to dye artificial silk and vegetable fibres from a neutral bath, the organic solvent being replaced by an aqueous solution of a carbohydrate such as dextrose.

A. J. HALL.

Development of vat dyes [indigosols] on the fibre. I. G. FARBENIND. A.-G., Assees. of K. JELLINEK (G.P. 445,251, 23.1.26. Addn. to G.P. 441,984).—Oxalic acid is used, in place of the formic acid of the prior patent, to liberate nitrous acid for development of indigosol prints.

C. HOLLINS.

Dyeing of acetate silk. I. G. FARBENIND. A.-G. (B.P. 274,823, 24.5.27. Ger., 24.7.26).—Acetate silk is dyed with unsulphonated azo dyes containing a di-(hydroxyethyl)amino-group. Thus di-(β -hydroxyethyl)-

aniline is coupled with diazotised *p*-nitroaniline (yellow) or 3 : 4 : 5-trichloroaniline (orange); di-(β -hydroxyethyl)-*m*-toluidine, m.p. 71—72°, with diazotised *p*-nitroaniline (dark yellowish-red) or 2 : 4-dinitroaniline (bluish-bordeaux).

C. HOLLINS.

Dyeing, printing, or stencilling of materials made of or containing cellulose derivatives. H. DREYFUS (B.P. 291,118, 23.11.26).—One or more thiocyanato-, cyano-, thiocarbimido-, carbimido-, ureido-, or thioureido-groups are introduced into dyes or dye components by known methods, and the products are used for dyeing acetate silk etc. Examples are: 1-acetamido-4-thiocyananthraquinone (yellow), 1-anthraquinonylthiocarbamide (orange), *N*-ethyl- or *N*-allyl-*N'*-1-anthraquinonylthiocarbamide (orange); 2 : 4-dinitroaniline \rightarrow 5-thiocyanosalicylic acid (brownish-yellow); aniline \rightarrow α -naphthylamine \rightarrow *o*-hydroxyphenylcarbamide (golden-yellow); *m*-aminophenylcarbamide \rightarrow aniline, coupled on the fibre with *m*-hydroxyphenylcarbamide (reddish-yellow); 2 : 2'-dinitro-4 : 4'-dithiocyanodiphenylamine (yellow); 3 : 3'-dinitro-4 : 4'-diureidodiphenyl (lemon-yellow), and 3 : 3'-dinitro-4 : 4'-diureidodiphenylmethane (greenish-yellow). The usual methods of dyeing are employed.

C. HOLLINS.

Manufacture of colour-printed fabrics. J. MORTON (B.P. 292,415, 22.11.27).—Colour-printed patterned fabrics are prepared by applying colours by means of plain (not engraved) printing rollers or blocks to fabric having figures or designs produced therein by jacquard mechanism, such figures containing dyed yarns or yarns pretreated so that they are capable of resisting or modifying the colour printed upon them.

A. J. HALL.

Printing pastes for textile materials. R. GAUNT, C. L. WALL, and BLEACHERS' ASSOC., LTD. (B.P. 292,441, 27.2.28).—Glycols having not less than three and not more than five carbon atoms are particularly suitable for addition to printing pastes containing basic dyes, since they are excellent solvents for the latter, *e.g.*, Acridine Orange DH and New Blue B.

A. J. HALL.

Weighting of silk. E. ELÖD, Assr. to R. KOEPP & Co. (U.S.P. 1,674,356, 19.6.28. Appl., 2.11.22. Ger., 14.11.21).—See G.P. 389,813; B., 1924, 708.

Preparation of [immunised] cotton fibres. P. KARRER, Assr. to CHEM. WORKS FORMERLY SANDOZ (U.S.P. 1,673,627, 12.6.28. Appl., 14.7.27. Switz., 11.3.25).—See B.P. 249,842; B., 1927, 71.

[Machine for elastic] mercerisation of fabrics. F. THIES (B.P. 291,130, 18.2.27).

Wet treatment of hanks of textile material. O. VON KOHORN and A. PERL (B.P. 274,885, 20.7.27. Ger., 20.7.26).

[Continuous] washing of silk piece goods. Soc. CLERTIN (B.P. 284,674, 3.2.28. Fr., 3.2.27).

Coloured nitrocellulose (B.P. 291,539).—See XIII.

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

Production of concentrated nitric acid by oxidation of ammonia under pressure. G. FAUSER (Giorn. Chim. Ind. Appl., 1928, 10, 183—195).—The

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results of experiments on the oxidation of ammonia in presence of platinum as catalyst, under various pressures and with different velocities of, and different proportions of air or oxygen in, the mixed gases, show that the use of increased pressure is attended by considerable advantages. The velocity of oxidation of the nitrous vapours increases rapidly as the pressure is raised, and the absorption occurs far more readily, so that much smaller towers may be used. The practical difficulties prove by no means insurmountable. Joints present little trouble, since at 5 atm. pressure the cross-section of the piping is reduced to one sixth, and chromium-steels appear to be suitable construction materials. The small amount of extra energy required when pressure is used is largely compensated for by the diminished cost of the plant and the greater concentration of the resulting acid. A saving may be effected of 1 ton of steam, required for pre-concentration, per ton of acid produced, and the final concentration is simplified. If the production of nitrogen is not desired, and if a sufficient amount of by-product oxygen is available, the residual gases may be sent directly, without re-compression, to the gas producers. In this way the alkaline absorption is avoided and it is necessary only to compress the ammonia-oxygen mixture in the closed circuit; the cost of compression is about one third of that involved in compressing ammonia-air mixtures. In an industrial plant with four converters, each having a capacity of 2500 kg. of 100% nitric acid per day, acid of d 1.436 has been obtained. This acid could be concentrated by distillation through a rectifying column, or, if dehydration is effected by distillation with sulphuric acid, only one fourth as much sulphuric is required as is necessary when acid of d 1.33 is treated. T. H. POPE.

Activated silica gel (sulphosil). B. TYTSCHININ and W. TOKMANOV (Neftjanoe Chozjajstvo, 1927, 12, 414—415; Chem. Zentr., 1927, II, 1524).—Instead of treating petroleum products etc. with concentrated or fuming sulphuric acid, and then with an adsorbent, the oil is treated with silica gel moistened with sulphuric acid (sulphosil). Silica gel is dried at 300° and, while still hot, mixed with concentrated sulphuric acid at 200—250° or with fuming acid at about 50°. The gel can take up 2—4 pts. of the concentrated acid and 0.6 pt. of sulphur trioxide; usually, gel containing only 0.2 pt. of oleum is used. Sulphosil is hygroscopic and loses its activity in contact with moist air; it also reacts with alcohol, benzene, or chloroform, but not with benzene. W. S. NORRIS.

Ammonia-soda process: physicochemical action of the carbon dioxide tower. E. I. ORLOV (Ukraine Chem. J., 1928, 3 [Tech.], 1—29).—In the upper part of the carbon dioxide tower used in the Solvay process, conditions are such that sodium bicarbonate and carbonate may crystallise out. This is shown experimentally by saturating an appropriate sodium chloride-ammonia solution with carbon dioxide and analysing the solution and salts crystallising out. Means of avoiding this side reaction are suggested, and, on the basis of a study of phase conditions, certain faults present in the Donetz and Berezniki factories are pointed out and the necessary alterations suggested. R. TRUSZKOWSKI.

Determination of barium hydroxide. E. VON DRATHEN (Chem.-Ztg., 1928, 52, 518).—Titration of barium hydroxide with hydrochloric acid gives results in close agreement with those obtained gravimetrically. Lower values, which may show a deficiency in BaO of as much as 1%, are obtained when using sulphuric or oxalic acid, owing to the absorption of baryta solution by the precipitated sulphate or oxalate. F. R. ENNOS.

Titration of bleaching powder with nitrite solution. Z. KERTÉSZ (Z. anal. Chem., 1928, 74, 105—108).—In the titration of bleaching powder, or of the bleach liquors obtained from it, by the nitrite method, an excess of sodium hydrogen carbonate should be added if much free lime is present. In the titration of bleach liquors containing free sodium or potassium hydroxide, boric acid must be added in excess of that required to saturate the alkali. A. R. POWELL.

Toxicities of arsenicals and fluorine compounds. MARCOVITCH.—See XXIII.

PATENTS.

Purification of silica gel. SILESIA VER. CHEM. FABR., and P. SCHLÖSSER (G.P. 447,139, 16.4.26).—The silica gel obtained by passing into water silicon fluoride obtained in the manufacture of superphosphates is purified by heating it in a current of steam to expel organic vapours, boiling an aqueous suspension of the product with potassium permanganate until the solution remains pink, and, finally, digesting with sulphur dioxide. A. R. POWELL.

Purification of sodium sulphate. M. A. PURDY, ASSR. TO PACIFIC DISTRIBUTING CORP. (U.S.P. 1,673,471, 12.6.28. Appl., 7.2.23).—Anhydrous sodium sulphate is crushed wet in a saturated solution of sodium sulphate, and the crushed salt is passed backwards through a continuous flow of sodium sulphate solution of gradually decreasing strength. A. R. POWELL.

Manufacture of globular sodium hydrogen sulphate. C. P. LINVILLE and C. E. MENSING, ASSRS. TO CALCO CHEM. CO. (U.S.P. 1,671,866, 29.5.28. Appl., 18.12.24. Renewed 17.9.27).—Molten sodium hydrogen sulphate is fed into the centre of a flat pan with outwardly sloping sides while the pan is rotated at a high speed within a deep chamber, so that the droplets of liquid which are thrown out by centrifugal force solidify in globules before they reach the floor of the chamber. A. R. POWELL.

Apparatus for recovering salts from hot solutions, e.g., potassium chloride liquors. F. KRUPP GRUSONWERK A.-G. (G.P. 447,059, 12.10.22).—The hot liquor is projected through a centrally disposed horizontal spray into a flat circular vessel provided with a series of revolving rakes which continuously move the deposited salt towards a central discharge opening in the vessel. A. R. POWELL.

Manufacture of solid tripotassium phosphate. I. G. FARBENIND. A.-G., ASSEES. OF E. JÄNECKE (G.P. 446,110, 21.5.26).—A concentrated solution containing potassium and phosphate ions in the correct proportions is saturated with ammonia; the tribasic potassium salt is precipitated in the form of a crystalline meal. A. R. POWELL.

Manufacture of potassium sulphate and ammonium chloride. N. SEVRIN (F.P. 622,684, 8.2.26).—A mixture of potassium chloride and ammonium sulphate in stoichiometrical proportions is added to such a quantity of water below 35° that the potassium sulphate formed by double decomposition is almost completely precipitated while the ammonium chloride is retained in solution. A. R. POWELL.

Manufacture of ammonium nitrate and blanc fixe. WOLFF & Co., and F. FROWEIN (G.P. 445,668, 26.5.25).—A mixture of ammonium sulphate and barium carbonate is stirred into a solution containing more than 15% of nitric acid, the resulting barium sulphate is separated, and the solution evaporated to obtain ammonium nitrate. A. R. POWELL.

Preparation of antimony [penta]sulphide. H. NUHN and L. H. BLOOD, Assrs. to ANTIMONY PRODUCTS CORP. (U.S.P. 1,671,203, 29.5.28. Appl., 27.7.22).—Finely-ground stibnite is digested with the requisite quantities of sodium sulphide and sulphur in hot water to obtain a solution of sodium thioantimoniate. This solution is diluted to *d* 1.13 and sprayed into a chamber through which sulphur dioxide is passed. The resulting mixture of antimony pentasulphide and sulphur is separated from the sodium thiosulphate solution formed at the same time, and, after drying, may be used directly for vulcanising rubber. By treating the thiosulphate solution with antimony trichloride "antimony crimson" is obtained, with the regeneration of sulphur dioxide for the first operation. A. R. POWELL.

Manufacture of calcium cyanamide. G. E. COX, Assr. to AMER. CYANAMID Co. (U.S.P. 1,674,466, 19.6.28. Appl., 17.6.25).—Ground calcium carbide is covered with a layer of comminuted kieselguhr and caused to react with nitrogen. H. ROYAL-DAWSON.

Preparation of alumina [from bauxite, clays, etc.]. J. C. SEAILLES (B.P. 283,509, 11.1.28. Fr., 11.1.27. Addn. to B.P. 277,697; B., 1928, 230).—Calcium aluminate is made by adding at least 4 mols. of lime for each mol. of alumina in the ore, and, optionally, 2–3 mols. of lime for each mol. of silica. The mixture is then ground with water in a colloid or other mill, and subjected to prolonged treatment in an autoclave to fix the silica. The calcium aluminate is washed to remove lime, treated with sodium carbonate solution or with caustic soda solution in presence of carbon dioxide, and the aluminium hydroxide is recovered from the sodium aluminate formed. W. G. CAREY.

Manufacture of anhydrous aluminium chloride. N. C. CHRISTENSEN (U.S.P. 1,673,495, 12.6.28. Appl., 7.6.23).—Aluminium is heated, in the presence of the chloride of a metal having less affinity for chlorine than aluminium at high temperatures, to a reacting temperature, the aluminium chloride being then separated from the residue. H. ROYAL-DAWSON.

Crystallisation of chrome alum. KÖNIGSBERGER ZELLSTOFF-FABR. U. CHEM. WERKE KOHOLYT A.-G., and E. SCHLUMBERGER (G.P. 447,070, 21.4.26).—A solution of chromic and potassium sulphates in the correct proportions is acidified with nitric acid and treated with formic acid or a formate to promote more rapid crystallisation of the alum. A. R. POWELL.

[Separation of] asbestos [from rock]. H. SIMON, LTD., and F. R. JOLLEY (B.P. 291,864, 8.3.27).—The crushed and screened material is fed into a washing trough containing a worm-type conveyor and having a slot through which heavy material falls, a further separation being effected through a grid or screen in the channel along which the rock travels.

W. G. CAREY.

Manufacture of magnesium phosphate. VER. F. CHEM. U. MET. PROD. (G.P. 447,393, 13.3.25).—Tricalcium phosphate is boiled with magnesium chloride and hydrochloric acid until complete conversion into magnesium phosphate takes place. A. R. POWELL.

Manufacture of phosphotungstomolybdenum compounds and of lakes therefrom. W. CAREMAEL, From I. G. FARBENIND. A.-G. (B.P. 292,253, 17.3.27).—Compounds obtained by the action of reducing agents, e.g., sulphur dioxide, sodium bisulphite or hyposulphite, or glucose, on complex acids having the formula $(a\text{WO}_3 + b\text{MoO}_3) \cdot \text{P}_2\text{O}_5 \cdot 3\text{H}_2\text{O}$, where $a + b = 24$ or 18 (cf. G.P. 445,151; B., 1928, 123), are used in the production of lakes from basic dyes or acid dyes containing free or substituted amino-groups in addition to sulpho-groups; e.g., Rhodamine B yields a red lake fast to light.

L. A. COLES.

Base-exchange bodies. SELDEN Co., Asses. of A. O. JAEGER (B.P. 279,466, 19.10.27. U.S., 19.10.26).—Base-exchange materials for use as catalysts are prepared by treating a solution of an alkali silicate with the salt of a metal capable of becoming part of the non-exchangeable nucleus, then with a salt of a metal acid, e.g., vanadic, molybdic, tungstic acid, etc. The gelatinous mass is stirred, filtered, and dried; it may then be subjected to a base-exchange process or heated in a suitable oxidising or reducing atmosphere to induce the desired catalytic properties. If desired, the zeolite may be incorporated with inert porous material either during or after formation. A. R. POWELL.

Catalytic process for the manufacture of hydrogen. R. J. A. GRENIER (B.P. 271,523, 23.5.27. Belg., 22.5.26).—Carbon monoxide and steam or water-gas and steam are passed over wood charcoal at 375°, yielding a mixture of hydrogen and carbon dioxide with a trace only of carbon monoxide.

W. G. CAREY.

Production of hydrogen-nitrogen mixtures. M. CASALE-SACCHI (B.P. 292,342, 7.6.27).—Liquid air is distilled to give pure nitrogen, and the oxygen (containing 2–3% N) is used for the incomplete combustion of hydrocarbons in the presence of steam. The carbon monoxide formed is catalytically converted into carbon dioxide and is removed, and the water is separated from the hydrogen by cooling; the pure nitrogen is then added to the hydrogen in suitable amounts, having regard to the nitrogen content of the oxygen used, for the synthesis of ammonia. W. G. CAREY.

Liquefying and separating the constituents of gaseous mixtures at low temperatures. L'AIR LIQUIDE SOC. ANON. POUR L'ÉTUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 281,675, 30.11.27. Fr., 4.12.26).—Oxides of nitrogen are removed from gas mixtures to be used in the manufacture of synthetic ammonia by

passing them prior to compression through a red-hot tube so as to decompose the nitrogen oxides into the constituent gases. Sulphuric acid used in the purification of gas mixtures is freed from oxides of nitrogen by treating it with a current of crude coal gas or with gases containing hydrogen sulphide. The presence of nitrogen oxides in gases to be liquefied may cause explosions if acetylene or other hydrocarbons are also present. A. R. POWELL.

Isolation of hydrogen cyanide from gaseous mixtures. I. G. FARBENIND. A.-G., Asses. of A. MITTASCH and W. MICHAEL (G.P. 444,502, 2.11.22).—Carbon dioxide is removed as ammonium carbamate etc. by addition of ammonia, or sufficient ammonia is added to remove hydrogen cyanide also and the mixture is fractionated. In the synthesis from carbon monoxide and ammonia, excess of the latter sufficient to combine with the carbon dioxide may be used. C. HOLLINS.

Stabilisation of liquid hydrogen cyanide. DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT, VORM. ROESSLER (G.P. 443,741, 3.7.25).—Those metals (or their alloys or compounds) which form complex compounds with ammonia or amines stabilise liquid hydrogen cyanide. Preferably they are used in thin plates, gauze, or powder (e.g., nickelled or coppered sheets etc.). Traces of organic or inorganic acids may be added. C. HOLLINS.

Manufacture of hydrogen sulphide or sulphides from sulphur. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 292,186, 10.1.27).—Sulphur or inorganic compounds yielding it, e.g., polysulphides, are treated under pressure above 100° but below dull redness with hydrogen, carbon monoxide, formic acid, or formates, preferably in the presence of catalysts, e.g., silica gel, alumina, active charcoal, or metals of the eighth group of the periodic system or their oxides, hydroxides, sulphides, or carbonates. L. A. COLES.

Recovery of sulphur from iron sulphides. R. F. BACON (U.S.P. 1,672,924, 12.6.28. Appl., 13.4.27).—Iron sulphides consisting mainly of the monosulphide are treated with sulphur dioxide so as to liberate sulphur and produce iron oxide. F. G. CLARKE.

Removal of free chlorine and bromine from fluid mixtures. BRIT. DYESTUFFS CORP., LTD., J. B. PAYMAN, and H. A. PIGGOTT (B.P. 292,307, 27.4. and 4.5.27).—The free halogen is removed from hydrogen chloride or bromide, either gaseous or in solution, by treatment with naphthalene, anthracene, solid olefines, or other solid hydrocarbon capable of forming additive compounds with the halogen. C. HOLLINS.

Extraction of the constituents of marine algæ. G. J. B. CHAMAGNE (B.P. 275,999, 11.8.27. Fr., 11.8.26).—The algæ are carbonised, finely ground, and burned (with the addition of ordinary powdered fuel, if necessary) in suitable plant provided with heat regenerators. The volatilised salts are precipitated electrically, dissolved in water, and the chlorides crystallised out from the cooled solution. The iodides and bromides are recovered from the mother-liquor by known methods. W. G. CAREY.

Concentration and distillation of solutions of hydrogen peroxide. I. G. FARBENIND. A.-G. (B.P.

264,535, 17.1.27. Ger., 18.1.26).—A dilute solution of hydrogen peroxide may be concentrated, or a concentrated solution purified, by passage in a finely-divided state or in thin layers through tubes or down a tower filled with non-catalytic fillers, e.g., glass or porcelain balls, while a strong current of gases travels in counter-current to the liquid. External heating may be applied or dried and preheated gases may be employed, using them again after drying for further treatment of the peroxide. W. G. CAREY.

Apparatus for making leaden powder [litharge]. C. A. HALL (U.S.P. 1,675,345, 3.7.28. Appl., 29.3.24. Renewed 2.12.27).—See B.P. 251,449; B., 1926, 584.

Separation of ingredients from an alkaline mixture containing oxy salt of arsenic and/or oxy salt of tin. H. HARRIS (U.S.P. 1,674,642, 26.6.28. Appl., 22.11.23. U.K., 4.12.22).—See B.P. 213,626; B., 1924, 522.

Purification of barium carbonate. J. E. MARWEDEL and J. LOOSER, Asses. to RHEINANIA-KUNHEIM VER. CHEM. FABR. A.-G. (U.S.P. 1,673,985, 19.6.28. Appl., 2.12.26. Ger., 4.1.22).—See G.P. 427,223; B., 1926, 630.

Production of [soluble] carbonates. M. BUCHNER, Assr. to A. F. MEYERHOFER (U.S.P. 1,675,786, 3.7.28. Appl., 18.6.25. Ger., 14.6.24).—See B.P. 235,588; B., 1925, 714.

Production of hydrogen. F. G. LILJENROTH and M. LARSSON, Assrs. to PHOSPHORUS-HYDROGEN Co. (U.S.P. 1,673,691, 12.6.28. Appl., 26.5.25. Swed., 29.5.23).—See G.P. 409,344; B., 1925, 448.

Acid-proof structures (B.P. 292,334).—See IX. **Complex mercury compounds** (B.P. 292,245).—See XXIII.

VIII.—GLASS; CERAMICS.

Silica bricks made without added bond. W. HUGILL and W. J. REES (Trans. Ceram. Soc., 1928, 27, 97—103).—Bricks were made from ganister and quartzite without the addition of a bond, and also, for purposes of comparison, with 2% of lime as bond. They were fired in an industrial down-draught kiln to cone 16—17. The percentage linear expansion in the kiln, compressive strength, powder and bulk density, and volume porosity were determined on the fired bricks. In the bricks without bond the degree of quartz conversion was greater than in the lime-bonded bricks, but the inversion was almost entirely to cristobalite. The addition of the bond facilitated tridymite formation, but did not accelerate the rate of quartz inversion. F. SALT.

Use of manganese in the manufacture of face bricks. G. E. SEIL and H. A. HEILIGMAN (J. Amer. Ceram. Soc., 1928, 11, 241—248).—Manganese dioxide is used either to change the background colour of a clay brick or to produce a speckled material. A grey colour can be produced without a colorant by soaking in a reducing atmosphere. 0.5% of air-floated manganese ore will deepen the colour under the same firing conditions. In order to obtain the greatest effect of the manganese ore in the manufacture of speckled bricks

it is essential to have a reducing atmosphere during the later stages of firing. Overfiring causes the manganese spots to pit the brick. In the case of clays maturing below 1000° the addition of a colorant has no effect, since the temperature never reaches the point at which the colorant fuses with the clay. The tinctorial effect of any addition is proportional to its exposed surface. It is essential to control the particle size of the colorant.

A. T. GREEN.

Influence of different felspars on the "freezing" behaviour of cones. E. ORTON, JUN., and J. F. KREHBIEL (*J. Amer. Ceram. Soc.*, 1928, 11, 215—223).—The "freezing" of a ceramic body is defined as its increased resistance to fusion consequent on an interruption of its heat-treatment in the vitrification zone, usually by chilling. Divergency in the behaviour of German and certain American pyrometric cones, which give identical values under normal firing conditions, is exhibited when they are subjected to a freezing treatment. Thus, a "frozen" American cone gave a value of 1295°, a raw cone from the same batch 1251°, whereas two comparable German cones each gave 1260°. It is shown that this effect occurs more seriously with the use of Hybla felspar instead of Swedish spar in the cone mixture. The kaolin used also has some influence. No evidence of crystallisation in the "frozen" cones could be found. The observed differences, in the main, related to the degree of completeness with which the felspar particles had dissolved in the glass. No explanation of the effect is put forward.

A. T. GREEN.

Effect of heat on the crystalline break-up of kaolin. J. F. HYSLOP and H. P. ROOKSBY (*Trans. Ceram. Soc.*, 1928, 27, 93—96).—Samples of kaolin were heated in an expansion furnace at the rate of 12° per min. to various temperatures between 600° and 1300°. The fired specimens were powdered and their X-ray patterns examined. The results indicated that kaolin breaks down at 550° to a crystalline phase (α), which is stable to 870°. At 870° the α -phase breaks down and two phases, β and either sillimanite or mullite, appear. The β -phase disappears at 1060°, mullite and silica remaining. Further heating eliminates the β -phase and mullite becomes well defined. The α - and β -patterns have not yet been identified.

F. SALT.

Terra-cotta slips. E. C. HILL (*J. Amer. Ceram. Soc.*, 1928, 11, 260—263).—The original body used contained 60% of clay and 40% of grog through 16-mesh. 70% of the clay content was made up of New Jersey tight-firing clay. No satisfactory slips for this body were obtained. The felspar-clay slips had a tendency to crack; slips with 50% of clay or less crazed, whilst others gave an unsatisfactory colour. Crazing decreases with increase in the content of clay or of Cornish stone. A similar body containing 60% of the New Jersey tight-firing clay was made up. A number of slips satisfactory for this body were obtained. The slip containing 43.7% of clay, with equal amounts of felspar and Cornish stone, is probably the safest slip developed, since it is equally removed from crazing and cracking on the one hand and from immaturity on the other.

A. T. GREEN.

Gas-producer operation. WINDETT.—See II.

PATENTS.

Manufacture of glass opaque to X-rays. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNY, ET CIREY (B.P. 284,648, 26.1.28. Fr., 2.2.27).—A glass effective for use as a protective screen from X-rays contains the oxides of lead and barium in the proportion given by the formula $B \times 1.005(P + 350) = 110$, B being the percentage of barium oxide and P that of lead oxide in the glass.

A. COUSEN.

Manufacture of a readily-fusible glaze or enamel. PATENT TREUHAND GES. F. ELEKTR. GLÜHLAMPEN, Assees. of H. NACHOD (G.P. 445,736, 5.12.24).—To the usual enamel mixture of boric acid, silica, zinc oxide, and 40—60% of lead oxide is added 8—16% of an alkali silicofluoride consisting wholly or partly of the lithium salt. The coefficient of thermal expansion of the product is lower than that of the lithium-free glazes.

A. R. POWELL.

Tunnel kilns. L. MELLERSH-JACKSON. From AMER. ENCAUSTIC TILING Co., LTD. (B.P. 291,504, 28.2.27).—The firing chamber of a tunnel kiln is provided with a stationary floor, the under side of which is exposed directly to the hot gases. This floor forms part of a muffle, which extends the full length of the tunnel and is heated at the sides, top, and bottom. The ware to be fired is placed on slabs of refractory material, e.g., carborundum; each loaded slab is placed on an exterior extension of the muffle floor, and is pushed into the kiln by means of a plunger. All the slabs are in contact, edge to edge, and also in direct contact with the floor of the muffle. Alternatively, a thin layer of sand may be placed between the stationary floor and the slabs.

F. SALT.

Kilns for burning clay products, glassware, and pottery. H. WEBSTER, SEN. (B.P. 291,482, 27.1.27).—A kiln is provided with two series of fire grates, one of which is arranged to fire with an up-draught, and the other with a down-draught. The whole of the contents of the kiln are fired with the draught first in one direction and then in the other, only one series of grates being in operation at a time. In a series of such kilns waste heat from either series of grates in any one kiln can be passed by a system of flues to any other kiln of the series with an up-draught or down-draught, or to dryers.

F. SALT.

Drying kilns for ceramic ware. BOMKESSEL & Co. M.B.H. (G.P. 445,817, 13.6.23).—The units of a series of drying kilns are coupled to one another by a series of flues closed by valves which permit of the hot air and flue gases being led as required from one chamber to another or to the stack. The air used for drying is preheated.

A. R. POWELL.

Process of forming ceramic articles and the like. Manufacture of ceramic products. Apparatus for making ceramic articles. T. S. CURTIS (B.P. 291,523—5, 3.3.27).—(A) A ceramic body is prepared in a "soft mud" consistency, a small percentage of an electrolyte (sodium silicate) is added, and the mixture thoroughly mixed in a blunger until the body has reached the consistency of a casting slip, such consistency being maintained as long as the mixture is agitated. When

agitation ceases, the slip consistency disappears. The body is cast while in the slip condition, and means are provided for maintaining the agitation while the slip is in the mould or being conveyed thereto. When agitation is discontinued, the body hardens quickly and can be removed promptly from the mould. Non-plastic, refractory bodies can be cast by this method. Air may be removed from the body mixture by treatment under a vacuum, and the casting operation may be carried out under air pressure. (B) A ceramic body formed of fibrous mullite crystals and a binder is obtained as the product of firing purified natural cyanite with additional alumina in splintery form. The latter substance is in the form of artificial corundum, obtained from alunite, and the binder may be a siliceous material, or magnesia and lime. The body mixture is practically free from clay, and articles are formed therefrom by the process outlined above. Maximum strength etc. is obtained by firing the ware to Orton cone 32. (C) Suitable apparatus is described. F. SALT.

Production of shaped metal-containing ceramic and like articles. M. HAUSER (B.P. 270,300, 26.4.27. Switz., 28.4.26).—Mixtures of ceramic materials with one or more metals or alloys, *e.g.*, iron, copper, nickel, ferromanganese, ferrosilicon, etc., are shaped and fired under such conditions that the free metal is present in the finished article. Oxidation of the metal or alloy is prevented by the use of a composition which sinters at a relatively low temperature, by covering the article with a readily fusible glaze, or by firing in a non-oxidising atmosphere. Metals of high m.p., *e.g.*, tungsten and tantalum, are used in the production of refractory products containing magnesia and/or zirconia.

L. A. COLES.

Refractory material. J. G. DONALDSON and H. L. COLES, Assrs. to GUARDIAN METALS Co. (U.S.P. 1,673,106—7, 12.6.28. Appl., [A] 16.1.25, [B] 3.7.26).—(A) The material consists of at least 70% of zirconia, less than 30% of zirconium silicate, and carbon, the last-named being dissolved in the silicate. (B) The refractory contains more than 85% of zirconia, less than 15% of silica, and less than 10% of carbon, the carbon being dissolved in the silicate formed during fusion of the oxides. H. ROYAL-DAWSON.

Manufacture of refractory articles. G. S. DIAMOND, Assr. to ELECTRIC REFRACTORIES CORP. (U.S.P. 1,674,961, 26.6.28. Appl., 23.9.26).—A mixture of magnesium oxide, crystalline graphite, a carbon binder, and a glaze is formed into the desired shape and baked; the product is again coated with glaze and rebaked. H. ROYAL-DAWSON.

Manufacture of abrasives. H. BERCKEMEYER (G.P. 447,051, 18.2.26).—Loam is mixed with a small quantity of a flux, *e.g.*, sodium chloride, and the mixture is heated in a gas-fired furnace with a regulated air supply until it begins to melt. The product is cooled and pulverised. A. R. POWELL.

Manufacture of abrasives. W. MARTINOV and H. BERCKEMEYER (G.P. 447,391, 28.2.25).—Loam which has been clinkered at 1500° is ground with sand and a flux, *e.g.*, sodium chloride, and the mixture is fused, cooled, and pulverised. A. R. POWELL.

Oven for burning refractories etc. H. KOPPERS, Assr. to KOPPERS DEVELOPMENT CORP. (U.S.P. 1,674,985, 26.6.28. Appl., 31.8.21. Ger., 8.3.18. Renewed 21.9.27).—See G.P. 347,672; B., 1922, 548 A.

Melting glass etc. R. L. FRINK (B.P. 291,883, 11.3.27).

Manufacture of sheet glass. R. L. FRINK (B.P. 292,448, 11.3.27).

IX.—BUILDING MATERIALS.

Application of Grosny paraffin mazout, mixed with creosote, to the preservation of wood. A. VORONOV (Neftjanoe Chozjajstvo, 1927, 12, 550—553; Chem. Zentr., 1927, II, 1526).—When creosote and petroleum products are mixed, resinous precipitates are often formed which inhibit the entrance of the preservative into the wood. Less precipitate is formed the lighter is the creosote (which must boil below 350°) and the heavier and more free from resin the petroleum product. A mixture of equal parts of creosote and paraffin mazout impregnated the wood satisfactorily. W. S. NORRIS.

PATENTS.

Manufacture of waterproof plastic Portland cement. E. C. R. MARKS. From INTERNAT. PRECIPITATION Co. (B.P. 291,988, 15.8.27).—Portland cement or clinker (10—20 pts.) is ground with 1 pt. of a waterproofing and plasticising agent consisting of shale and/or diatomaceous earth containing not less than 5% and preferably 10—50% of oil or bituminous material in a form which can be extracted with solvents. When the proportion of soluble oil is less than 10%, oil is added to the shale in such quantity that the total soluble oil exceeds 10%. Other substances, *e.g.*, magnesium, calcium, or zinc chloride, sulphate, silicate, or fluosilicate, etc., may also be added. L. A. COLES.

Preparation of cement. ELEKTROSCHMELZE G.M.B.H. (G.P. 436,052, 25.6.25).—Aluminous material is melted together with a highly conducting substance in an electric furnace. A. R. POWELL.

Hardening and ageing calcined gypsum products. J. H. COLTON, Assr. to PACIFIC PORTLAND CEMENT Co., CONSOL. (U.S.P. 1,659,971, 21.2.28. Appl., 7.9.26).—Casting plaster is produced by calcining calcium sulphate at the required temperature, adding raw gypsum to the product, and further calcining the mixture to reduce its water content to 4—6%. H. ROYAL-DAWSON.

Manufacture of bricks, tiles, etc. H. and T. PARDOE and H. HILL (B.P. 291,960, 14.6.27).—Mixtures of slate debris or colliery shale with ferric oxide ores or siliceous iron oxide ores, finely ground with the addition of water, if necessary, until they bind together when squeezed in the hand, are pressed or moulded into shape, dried, and baked. L. A. COLES.

Production of acid-proof structures. J. K. WIRTH (B.P. 292,334, 26.5.27).—Acid-proof bricks are cemented together and metal surfaces etc., after application of an intermediate acid-proof layer, are coated with unhardened phenol-aldehyde condensation products, and these are subsequently hardened by treatment with acids or acid salts at the ordinary or at a slightly

raised temperature. Alternatively, uncondensed phenol-aldehyde mixtures are used and condensation and hardening are effected in one operation after application.

L. A. COLES.

Manufacture of marble substitutes. K. STICHA, J. HUBÁČEK, and J. KNEIFL (B.P. 292,388, 5.9.27).—A glazing composition of sodium sulphate, calcium chloride, black and red colouring matters, water-glass, ammonium chloride, and water is applied at about -2° to a smoothly-ground backing, and is then dried and polished.

W. G. CAREY.

Manufacture of a porous building material. J. A. ERIKSSON (G.P. 447,194, 12.6.24. Swed., 17.3.24).—A dry mixture of hydraulic material (burnt shale or the like), slaked lime, and a metal powder which evolves gas in contact with water is ground, mixed with cement and water, and formed into shapes.

A. R. POWELL.

Preservation of wood. L. P. CURTIN (B.P. 291,857, 7.3.27).—Wood is protected from attack by wood-rotting fungi, e.g., *Fomes annosus*, by impregnation with barium hydroxide solution, or an emulsion of it with petroleum oil, the hydroxide being rapidly converted into the carbonate by the carbon dioxide in the air. Sodium carbonate solution may be used when it is not likely to be leached out of the wood.

L. A. COLES.

Production of a continuous floor for linoleum, trioline, etc. L. FRIESER and O. SEEWALD (U.S.P. 1,659,867, 21.2.28. Appl., 23.7.25. Ger., 24.10.24).—To peat or peat flour previously desiccated is added coal tar to render it waterproof, and the product hardened by treatment with magnesium chloride and magnesite.

H. ROYAL-DAWSON.

Agitators [for cement slurry]. R. E. MINOGUE (B.P. 292,714, 2.4.27).

Bitumen emulsions (B.P. 275,928).—See II. Rotary kiln (U.S.P. 1,673,051).—See X.

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

Alloys of iron [steels] with a low percentage of titanium. H. MATHESIUS (Stahl u. Eisen, 1928, 48, 853—858).—Addition of a small percentage of aluminothermic ferrotitanium to mild steels in the ladle immediately before pouring removes practically the whole of the sulphur content, increases the yield point to about 80% of the tensile strength, instead of 50—60%, prevents segregation, produces an even structure, and generally toughens the steel. A steel with 0.15% C and 0.23% Ti had an average tensile strength of 63 kg./mm.², an elongation of 15%, and a reduction of area of 42%. A hard-drawn wire of this steel 0.5 mm. in diam. had a tensile strength of 115 kg./mm.² A. R. POWELL.

[Copper-chromium] constructional steel. E. H. SCHULZ (Stahl u. Eisen, 1928, 48, 849—853).—A steel containing 0.5—0.8% Cu, about 0.4% Cr, 0.15% C, 0.25% Si, and 0.8% Mn has all the valuable properties, for constructional purposes, of a 1% silicon steel, but its preparation is unattended by the difficulties met with in melting and casting the silicon alloy. The tensile strength is 53—58 kg./mm.², yield point 37—43 kg./mm.², elongation 22—24%, and reduction in area 47—57%.

The new steel is readily welded and is more highly resistant to corrosion than plain carbon steel containing a small percentage of copper.

A. R. POWELL.

X-Ray investigation of the structure of quenched steels. N. J. SELJAKOV, G. V. KURDUMOV, and N. T. GOODTZOZ (Rev. Mét., 1928, 25, 99—104, 222—230).—Carbon steels containing 0.84, 1.02, and 1.18% C were quenched from different temperatures, and their crystal structures, before and after tempering at increasing temperatures, were determined by the Debye-Scherrer method. The quenched steels gave diffraction patterns corresponding to (1) a body-centred tetragonal structure with nearly equal axes, and (2) some unchanged γ -iron (austenite). The parameter of the body-centred tetragonal structure was almost constant at 2.85 Å., and the ratio of the axes a/c varied from 1.0 to 1.06, increasing with the carbon content and with the temperature of quenching. The body-centred tetragonal structure is thus very similar to the body-centred cubic structure of α -iron, and is looked on as a distorted form of the latter in which the carbon atoms have not combined to form cementite; the mechanism of the change from γ - to α -iron with carbon atoms occupying interstices in the iron lattice is discussed in detail on this basis. The quenched steels give diffused diffraction lines owing to the smallness of the micro-crystals, the presence of internal strains, and to heterogeneous effects due to the irregular distribution of the carbon atoms, and to the fact that the speed of quenching is not the same throughout the specimen.

W. HUME-ROTHERY.

Critical points and the martensitic tempering of nickel and nickel-chromium castings. L. GUILLET, GALIBOURG, and BALLAY (Compt. rend., 1928, 187, 14—16).—Martensitic (or secondary) tempering trials made with castings containing 3.2—3.4% C (total), 0.9% P, and varying amounts of other elements, by heating to above the transformation point and cooling rapidly, showed that silicon raises the transformation temperature, and therefore increases the critical rate of tempering, but diminishes the tendency both for primary and secondary tempering. Manganese (0.20—0.65%), nickel, and chromium have the reverse effect on the secondary tempering, whilst the tendency for primary tempering is unaffected by manganese and diminished by nickel. Such castings may be classed as (a) easily workable after moulding and hardened by cooling in air after heating to 900°, and (b) hardened after pouring but easily workable if reheated for a short period at 650°, and subsequently hardened by heating to 900° and cooling in air.

J. GRANT.

Reduction of oxide ores. B. BOGICH (Rev. Mét., 1928, 25, 247—261).—The reduction of oxide ores of copper, nickel, cobalt, and iron by means of carbon monoxide, wood charcoal, and coal has been investigated. The degree of reduction is independent of the quantity of reducing agent present, if the temperature is high enough for the slag to separate. Rapid fusion of oxide ores of copper, nickel, and cobalt generally prevents equilibrium between the ore and the reducing agent from being reached. The addition of fluxes increases the velocity of the reaction. Their effect is specific, and depends also on the composition of the ore. The

optimum proportion of flux can be determined only by experiment, a method for which is described.

C. W. GIBBY.

Copper-antimony alloys. NEUMARK.—See XI.
Protective coatings for metals or alloys. GARDNER.—See XIII.

PATENTS.

Rotary kiln for burning, roasting, and sintering of mineral materials. F. LUTHER, Assr. to G. POLYSIUS (U.S.P. 1,673,051, 12.6.28. Appl., 4.3.27. Ger., 9.9.26).—The material falls from the kiln into a cooling drum which has a hood over its delivery end. Air is injected into the drum in such a manner that air is also sucked into it, the amount of which can be controlled.

F. G. CLARKE.

Metallurgical furnace. E. H. BUNCE and G. T. MAHLER, Assrs. to NEW JERSEY ZINC CO. (U.S.P. 1,674,947, 26.6.28. Appl., 10.12.25).—Spaced resistances having spiral convolutions providing a circuitous path for the passage of electric current extend into a molten bath contained in the furnace chamber.

J. S. G. THOMAS.

Regenerative open-hearth furnace. S. J. CORT, T. BURNS, R. S. A. DOUGHERTY, and C. E. LEHR, Assrs. to BETHLEHEM STEEL CO. (U.S.P. 1,671,100, 29.5.28. Appl., 21.12.23).—The furnace is provided with a damper-controlled throat, the lining of which is either made of heat-resistant material or provided with water-cooling means, and the dampers to which work in slots adapted to reduce leakage of gases from the furnace.

A. R. POWELL.

Annealing furnaces. BRIT. FURNACES, LTD., and E. W. SMITH (B.P. 291,909, 23.3.27).—A furnace for annealing coils of wire.

A. R. POWELL.

Annealing of metal. H. W. BROWNSDON and KYNOCH, LTD. (B.P. 292,690, 25.3.27).—Loss of heat from hot annealed metal sheets or strips is reduced by bringing the hot metal from the furnace into close contact with cold sheet about to enter the furnace chamber, the heat interchange being effected mostly by direct conduction.

C. A. KING.

Prevention of oxidation during the annealing of metal [iron or steel] bands or wires. O. H. DÖHNER (G.P. 447,143, 21.4.26).—The material to be annealed is passed between two movable plates of refractory material during its passage through the muffle.

A. R. POWELL.

Reduction of [iron] ores. H. G. C. FAIRWEATHER. From NYBERGS GRUFKÄMBOLAG (B.P. 291,822, 31.1.27).—Iron ore contained in carriages is passed through a long channel furnace divided into three zones, in the first of which it is subjected to a high temperature in an oxidising atmosphere to remove sulphur and roast the ore, in the second it is partially reduced by a current of reducing gases, and in the third it is completely reduced by means of carbon monoxide, which is continually regenerated by the circulation of the gas from this zone through a mass of glowing carbonaceous material. The excess gas produced in the continuous reduction process is used to effect the preliminary reduction in the second zone, and the gases from this zone, together with preheated air obtained from the cooling

chambers, are used for providing the heat necessary for roasting the ore. Water is used for quenching the partially air-cooled product from the furnace, and the steam generated passes to the recarbonising furnace for the production of water-gas, which may be used in the reduction zone.

A. R. POWELL.

Rust-proofing of iron, steel, and other metals. W. H. COLE (B.P. 292,666, 19.3.27).—Metal articles are heated in a revolving container in contact with a dry mixture of emery, copper-zinc precipitate, tin, and aluminium, to which may be added cobaltous chloride, ferrous chloride, nickel sulphate, antimony trichloride, etc.

C. A. KING.

Hardening of iron or steel articles. DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT, VORM. ROESSLER (B.P. 277,030, 5.9.27. Ger., 6.9.26).—A carburising bath for hardening purposes comprises a mixture of 40% of alkali cyanide with sodium carbonate and chloride together with 3–97% of carbon, preferably wood or animal charcoal, or with substances, e.g., anthracene or naphthalene, which liberate carbon at high temperatures. Alternatively, a mixture of 85% of sodium cyanide, 12% of sodium cyanamide, and 3% of finely-ground wood charcoal may be used. The baths may be operated at temperatures up to 950° without frothing.

A. R. POWELL.

Production of hard, wear-resisting printing surfaces. K. W. SCHWARTZ, Assr. to UNITED CHROMIUM, INC. (U.S.P. 1,673,779, 12.6.28. Appl., 5.8.25).—An unhardened steel body bearing the design to be printed has a coating of electrolytic chromium on its printing surface.

H. ROYAL-DAWSON.

Etching of [aluminium] printing plates. F. GUTZSCHKE (U.S.P. 1,660,366, 28.2.28. Appl., 17.2.27. Ter., 22.2.26).—A dilute solution of calcium nitrate and potassium dihydrogen phosphate is claimed.

H. ROYAL-DAWSON.

Ascertaining the degree of absolute or relative resistance to rusting shown by iron, steel, or iron alloys. C. MAULER (B.P. 292,794, 28.7.27).—Specimens of iron or steel alloys, particularly rustless steel, are immersed in a boiling solution containing 60–70 kg. of sodium hydroxide and 8–15 kg. of sodium nitrate in 20–40 litres of water, a little of the used solution also being added. As the solution evaporates and the b.p. rises, the specimens are withdrawn and examined with respect to the degree of rusting, which is classified in relation to the b.p. of the solution at which rusting commences. Alkalinity of the solution is maintained by adding caustic lime at intervals.

C. A. KING.

Treatment of manganese steel. V. C. MEKEEL, Assr. to TAYLOR-WHARTON IRON & STEEL CO. (U.S.P. 1,671,870, 29.5.28. Appl., 27.2.26).—In order to impart greater strength and wear-resisting properties to manganese steel without impairing its toughness or ductility the metal is case-hardened, during the regular heat-treatment, in a closed vessel packed with barium carbonate and charcoal or with boron trioxide and a reducing agent, and subsequently quenched out of contact with the air.

A. R. POWELL.

Raising the yield point of steel alloys. F. KRUPP A.-G. (B.P. 282,015, 21.10.27. Ger., 7.12.26).—A hollow

rotary body of nickel-manganese steel is rotated in a centrifugal pit at a high speed so that it undergoes a stress exceeding its yield point. The deformed rotary body is then given its final shape and will have a yield point beyond that required in service.

A. R. POWELL.

Concentration of oxidised ores. T. S. CARNAHAN, Assr. to UNION MINIERE DU HAUT-KATANGA (U.S.P. 1,671,698, 29.5.28. Appl., 23.5.27. Belg., 28.5.26).—A warmed mixture of partly rancid palm oil (1–4 pts.) and oleic acid (1 pt.) gives a better and more efficient foam than oleic acid alone when added to the ore pulp.

C. HOLLINS.

Froth-flotation concentration of ores. C. H. KELLER, Assr. to MINERALS SEPARATION NORTH AMER. CORP. (U.S.P. 1,671,590, 29.5.28. Appl., 24.3.27).—One of the usual frothing agents is used in conjunction with an impure metal xanthate, *e.g.*, the calcium xanthate obtained from lime, carbon disulphide, water, and alcohol. The process is especially useful for obtaining copper, lead, and zinc concentrates low in iron.

C. HOLLINS.

Preparation of flotation concentrates. H. J. STEHLI (U.S.P. 1,673,891, 19.6.28. Appl., 30.7.26).—In preparing plastic ore fines for sintering, a mass of wet fines is divided into lumps, and these are coated with powdered material to prevent them from adhering to one another and to allow for air passages in the mass.

H. ROYAL-DAWSON.

Roasting and/or sintering of fine [zinc] ore or other fine material. COMP. DES METAUX OVERPELT-LOMMEL (B.P. 284,248, 9.3.27. Belg., 26.1.27).—The ore is roasted until the sulphur content is reduced to 5–6% of sulphide sulphur. The product is mixed with 1–5% of ferrous sulphate or sulphuric acid and with sufficient water to form a plastic mass. This mass is fed into a machine which compresses it and extrudes it as friable cylindrical lumps which readily break up into coarse sand amenable to blast roasting or sintering in the Dwight-Lloyd or Schlippenbach apparatus.

A. R. POWELL.

Smelting of zinc ores. H. SEIDLER (G.P. 445,689, 16.1.25).—The roasted ore is smelted with a deficiency of carbon so that only about half of the zinc is recovered as metal. The sintered residue in the retorts is freed from any powdery carbonaceous material it contains, mixed with an excess of fuel or reducing agent, and smelted in blast or revolving furnaces to obtain zinc oxide for use as a pigment, and to recover any other easily reducible metal contained in the residue.

A. R. POWELL.

Bath for galvanising. N. K. TURNBULL (B.P. 291,961, 14.6.27).—In internally-heated galvanising baths (*cf.* B.P. 220,398; B., 1924, 836) the rate of transfer of heat from the layer of lead to that of zinc is increased by means of metallic conducting bars establishing thermal connexion between the layers and passing through the interposed layer of dross.

J. S. G. THOMAS.

Treatment of pyritic [tin] ores, residues, etc. F. L. WILDER, E. MORRIS, E. SCHIFF, and E. S. KING (B.P. 291,919, 6.4.27).—The roasted material is ground to a fine sand which is leached under pressure in an

autoclave successively with an ammoniacal solution of ammonium carbonate, an alkaline brine, and an acid brine. The first treatment removes copper and tungsten, the second stannic acid, and the third lead, bismuth, and silver, whilst the residue contains cassiterite and the precious metals.

A. R. POWELL.

Dearsenification of ores and metallurgical products. NORDDEUTS. AFFINERIE (B.P. 286,285, 17.10.27. Ger., 3.3.27).—Arsenic is removed from arsenide ores or speiss by heating a mixture of the finely-ground arsenical material with 25% of pyrites and 20% of fine bituminous coal in a multiple-hearth furnace to which a restricted supply of air is admitted. The charge is self-combustible, and if mechanically rabbled does not cake.

A. R. POWELL.

Treatment of oxidised arsenical lead ores. DR. A. NATHANSOHN, METALL- u. FABRIKWERKE A.-G., OTAVI MINEN- u. EISENBAHN-GES., A. HIRSCH & SOHN, ZINK-HÜTTE HAMBURG, and COMP. MÉT. FRANCO-BELGE DE MONTAGNE (SOC. ANON.) (G.P. 445,854, 1.11.24).—Arsenical lead ores are leached with a concentrated solution of a chloride containing free hydrochloric acid, and the arsenic dissolved is precipitated by the addition of lime followed by oxidising substances with or without the addition of a ferric salt.

A. R. POWELL.

Extraction of metals from metallic sulphides. **Extraction of metallic nickel.** A. B. BAGHDASARIAN [A. B. BAGSAR] (U.S.P. 1,671,003–4, 22.5.28. Appl., [A] 17.8.25, [B] 19.7.26).—(A) The sulphides are chloridised at a suitable temperature, and the chlorides so produced are heated and reduced with hydrogen to give the metals. (B) A solution containing nickel is first treated to remove objectionable impurities, and then most of the nickel is precipitated by addition of metallic zinc. This is collected, the clear solution is boiled, and to it is added more zinc to complete the precipitation of the nickel.

F. G. CROSSE.

Cyaniding apparatus for treating gold ores. R. KANDA (U.S.P. 1,673,982, 19.6.28. Appl., 13.9.26. Japan, 24.9.25).—The cyaniding tank has a conical bottom to which is connected an annular pipe through which inlet nozzles project at a tangent into the tank. These nozzles communicate with the annular pipe from which they convey liquid to the tank.

Treatment of ores. W. E. STOKES, Assr. to U.S. PROCESSES, INC. (U.S.P. 1,674,806, 26.6.28. Appl., 18.11.22).—Ores are roasted, at a temperature below their fusion point, with an alkali bicarbonate.

F. G. CROSSE.

Workable copper-silicon alloys resistant to corrosion. F. DOEBLIN (G.P. 447,247, 11.7.24).—Copper alloys resistant to corrosion by alkalis and acids contain 5–10% Si, 1.5–12% Ni, CO, and/or Fe, and up to 5% Cr, W, Mo, Ti, or V.

A. R. POWELL.

Manufacture of [nickel] alloy. R. FRANKS and B. E. FIELD, Assrs. to HAYNES STELLITE CO. (U.S.P. 1,675,798, 3.7.28. Appl., 8.1.25).—The alloy contains 3–5% Al, 3.5–6% Si, 5–12% W, 6–12% Zr, up to 1% B, and the rest chiefly nickel.

F. G. CROSSE.

Treatment of nickel-copper-aluminium alloys. T. S. FULLER, Assr. to GEN. ELECTRIC CO. (U.S.P.

1,675,264, 26.6.28. Appl., 17.3.24).—The physical properties of such alloys containing only a small proportion of aluminium are improved by deforming the alloys at about 1000° and completing the treatment at 500—1000°.

F. G. CROSSE.

Bimetallic element. H. SCOTT, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,671,490—1, 29.5.28. Appl., 27.12.26).—A thermostatic element comprises strips of two elements having different coefficients of expansion, the element having the lower coefficient being (A) a ferrous alloy containing 38—45% Ni or (B) an alloy of 45% Ni and 55% Fe, and the element having the higher coefficient being (A) iron with 10% Mn and 10% Ni or (B) a steel alloy containing 0.1% C and 15—23% Mn. The elements are very susceptible to temperature changes between 150° and 400°.

A. R. POWELL.

Soldering lead-bearing metal castings. METALLBANK U. METALLURGISCHE GES. A.-G. (G.P. 447,133, 23.1.25).—The molten bearing metal is treated with a small quantity of magnesium and poured into its bed which has previously been coated with a suitable solder.

A. R. POWELL.

[Aluminium] alloy. Heat-resisting alloy and structure. N. V. HYBINETTE (U.S.P. 1,675,708—9, 3.7.28. Appl., [A] 23.9.22. Renewed 17.11.27. [B] 2.2.24).—(A) An alloy contains at least 90% Al, substantial amounts of nickel and iron, considerably less than 1% of chromium, tungsten, or molybdenum, and traces only of copper. (B) The alloy contains 5—10% Ni, 15—25% Cr, 1—2% C, 0.5—2% Al, and remainder iron.

F. G. CROSSE.

Welding medium for aluminium. W. REUSS (U.S.P. 1,675,664, 3.7.28. Appl., 3.12.27. Ger., 24.7.25).—A mixture of equal parts of borax, boric acid, and caustic potash is claimed.

F. G. CROSSE.

Treatment of metals. [Coating wires with aluminium.] G. H. HOWE, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,673,624, 12.6.28. Appl., 2.1.25).—Metal wire is passed downwards into a bath of molten aluminium, then upwards through the bath in a direction parallel to its downward path. At the lowest point of the bath the wire operates a baffle, which sets up a circulation in the bath, and the metal is heated by means of a gas flame at the point at which the wire leaves it.

A. R. POWELL.

Treatment of aluminium alloys. H. C. HALL and T. F. BRADBURY (B.P. 292,407, 5.11.27. Addn. to B.P. 281,912; B., 1928, 128).—The casting previously described is brought to the required degree of hardness and tensile strength by being heated at 160—190° for at least 20 hrs., and then quenched.

H. ROYAL-DAWSON.

Removal of oxide films from metal surfaces. H. WAGNER (G.P. 446,652, 3.5.25. Addn. to G.P. 368,537).—The surface is covered with a mixture of alkali, lime, zinc dust, and a polyhydric phenol, xanthic acid, or thiocarbamide, and the metal heated until deoxidation is effected.

A. R. POWELL.

Drying and removing gases from metal powders. E. C. R. MARKS. FROM RINGSDORFF-WERKE A.-G. (B.P. 291,949, 28.5.27).—Moist metal powders are

slightly compressed and alcohol is forced, sucked, or centrifuged through the mass to displace water, whereupon the mass is allowed to dry in the air either with or without being compressed into any desired shape.

A. R. POWELL.

Working-up of mixed shavings of white metal and red metal. A. SCHWARZ, Assce. of INTERNAT. METALL A.-G. (B.P. 270,740, 5.5.27. Ger., 5.5.26).—To separate the constituents of mixed shavings obtained in the turning of bearings, the mixture is heated to the m.p. of the eutectic of the white metal in a rotating drum provided with baffles, whereby the white metal is broken up into small particles by the abrasive action of the red metal (bronze) turnings. Subsequent sieving of the mixture effects almost complete separation of the powdered white metal from the unchanged red metal.

A. R. POWELL.

Apparatus for the electrolytic production of metallic sheets and similar articles. J. C. F. A. SCHÜTTE (B.P. 292,015, 14.11.27).—An apparatus for the recovery of sheet metal by electrolysis direct from ore or scrap metal comprises a wood or stoneware vat, the walls of which are lined with lead which serves as the cathode. The anode consists of lump ore or scrap metal packed in the annular space between two perforated cylinders each of which can be rotated independently or together in the same or in opposite directions. A rotating paddle device within the inner cylinder further assists in the free circulation of the electrolyte throughout the vat.

A. R. POWELL.

Cupola. A. H. COPLAN, Assr. to ECONOMY METAL PRODUCTS CORP. (U.S.P. 1,675,255, 26.6.28. Appl., 10.6.27).—See B.P. 288,072; B., 1928, 412.

Manufacture of ferrobore and boron-steel. T. MIYAGUCHI (U.S.P. 1,674,119, 19.6.28. Appl., 19.10.21).—See B.P. 195,678; B., 1923, 661 A.

Manufacture of copper alloys. O. VON ROSTHORN (U.S.P. 1,675,008, 26.6.28. Appl., 11.5.21. Ger., 4.2.20). See B.P. 158,882; B., 1922, 596 A.

Production of technical pure beryllium respectively for freeing metallic beryllium from impurities. H. FISCHER, Assr. to SIEMENS & HALSKE A.-G. (U.S.P. 1,673,043, 12.6.28. Appl., 7.12.25. Ger., 10.9.25).—See G.P. 443,944; B., 1928, 58.

Recovery, separation, or extraction of gold, platinum, and other metals, applicable also for separation of precious stones from earth or material containing them. G. C. E. F. HANCIAU (U.S.P. 1,673,675, 12.6.28. Appl., 25.9.23. U.K., 9.10.22).—See B.P. 213,626; B., 1924, 522.

Treatment of ores containing refractory oxide. T. R. HAGLUND, Assr. to INTERNAT. PATENT CORP. (Re-issue 17,001, 19.6.28, of U.S.P. 1,569,483, 12.1.26).—See B., 1926, 164.

Drying and extracting gases from metallic powders. E. SEYFFERTH, Assr. to FULMIT GES.M.B.H. (U.S.P. 1,674,230, 19.6.28. Appl., 16.6.27. Ger., 12.3.25).—See B.P. 291,949; preceding.

Treatment of ores (B.P. 279,447).—See II. Electron-emission material (U.S.P. 1,670,463).—See XI.

XI.—ELECTROTECHNICS.

Addition agents in electrodeposition. V. Application of the complex cation theory of crystalloidal addition agents to base metals. I. G. FUSEYA and K. MURATA. II. G. FUSEYA and R. YUMOTO (J. Soc. Chem. Ind. Japan, 1928, 31, 323—331, 331—342; cf. B., 1926, 950).—I. In an attempt to show the formation of complex cations between glycine present as an addition agent and lead or zinc ions, by spectroscopic and electrometric methods, it was found by the former method that a mixed solution of lead fluosilicate or perchlorate and glycine extended its absorption bands toward longer wave-lengths in the ultra-violet zone than did its components, thus indicating the formation of complex compounds in both solutions. With a mixture of zinc sulphate and glycine no such increased absorption occurred. Similarly, ferric and chromic sulphates formed complex compounds with glycine, but none was detected spectroscopically in the case of nickel and cobalt sulphates. From measurements of the *E.M.F.* of cells of the type: $Pb|0.1N-Pb(NO_3)_2|0.1N-Pb(NO_3)_2+M\text{-glycine}|Pb$, and the degree of ionisation of salt solutions calculated from conductivity ratios, the degree of complex formation was computed. It was found that about 94% of lead and about 75% of zinc were in complex forms in their 0.1*N*-solutions when 1 mol. of glycine was present, and these compounds by migration experiments were found to be cations.

II. No increase in weight of the deposits nor decrease in size of the crystals was found in the electrodeposition of lead and zinc from lead nitrate, lead fluosilicate, and zinc sulphate solutions containing glycine. It is concluded that in such electrolytes the glycine does not behave as an addition agent but forms complex cations with these metallic ions.

S. OKA.

Anodic behaviour of copper-antimony alloys. H. NEUMARK (Metall u. Erz, 1927, 24, 305—311; Chem. Zentr., 1927, II, 1394—1395).—Electrolysis of alloys of antimony and copper in sulphate electrolytes with or without the addition of tartaric acid soon ceases owing to the formation of a non-conducting film of basic salts of copper and antimony on the anode. Copper may, however, be recovered from antimonial alloys by electrolysis in a solution of copper nitrate. No antimony enters the electrolyte and the current efficiency is high. The anode slime consisting chiefly of antimonic acid does not adhere to the anode, so that the electrolysis can be carried to completion. If the alloy contains lead the best results are obtained with a slightly acid bath which is 2*N* with respect both to copper sulphate and copper nitrate.

A. R. POWELL.

Duriron as an insoluble anode for gold cyanide solutions. M. DE K. THOMPSON [with COSGROVE, SARROS, HAUEISEN, and DINAN] (Amer. Electrochem. Soc., Sept., 1928, 54. Advance copy. 5 pp.).—Corrosion experiments show that duriron anodes are much more resistant than peroxidised lead anodes in cyanide solutions of the concentration used in the electrolytic extraction of gold. Duriron anodes cannot be used, however, for the oxidation of chlorate to perchlorate, of chromic salts to chromate, or of manganate to permanganate.

H. J. T. ELLINGHAM.

Speed controller. VEIHMAYER and GIVAN.—See XVI. **Purification of water.** ATEN.—See XXIII.

PATENTS.

Electric annealing furnace. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of T. STASSINET (B.P. 262,800, 9.12.26. Ger., 12.12.25).—In an electric annealing furnace for intermittent working comprising a heating chamber formed by fireclay walls carrying resistances and surrounded by insulating walls, the fireclay wall is of minimum thickness consistent with strength and resistance, whilst the thickness of the insulating walls is such that the sum of the accumulator heat loss and that due to radiation and conduction is a minimum.

J. S. G. THOMAS.

Optical pyrometer. D. C. GALL (B.P. 291,971, 29.6.27).—In an optical pyrometer of the disappearing-filament type, in which the voltage drop in the filament is taken as the indicator of temperature, potential leads are connected to parts of the filament which are not appreciably affected by changes in the temperature of the surroundings. When the resistance of the filament inserted in a form of Wheatstone bridge is employed as temperature indicator, a compensating filament resistance inserted in one arm of the bridge eliminates errors due to the temperature of parts of the principal filament, *e.g.*, the ends, being affected by changes in the temperature of its surroundings or by conduction etc.

J. S. G. THOMAS.

Magnetic core material. STANDARD TELEPHONE & CABLES, LTD. FROM WESTERN ELECTRIC CO., INC. (B.P. 290,736, 19.2.27).—An iron-nickel alloy containing more than 25% Ni is treated with less than 5% of antimony, and the molten mixture is thoroughly oxidised by means of air, oxygen, or a metal oxide. The product is broken up, the fragments are coated with an insulating material, and the product is made into loading coils. (Cf. U.S.P. 1,669,649; B., 1928, 490.)

A. R. POWELL.

Apparatus and method for gaseous reactions. J. M. WEED, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,673,654, 12.6.28. Appl., 23.4.24).—Gases are supplied under pressure to the region outside the porous, refractory wall of a chamber in which an arc is operated, and means are provided for carrying away gases which have passed into the arc through pores in the chamber wall.

J. S. G. THOMAS.

Incandescence electric lamp. R. A. PRICE, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,673,267, 12.6.28. Appl., 15.5.24).—The lamp filament, supported by insulating material, is positioned by an alloy containing more than 50% Au and more than 10% Ag.

J. S. G. THOMAS.

Filaments for electric incandescence lamps. SOC. FRANÇ. DES LAMPES À INCANDESCENCE "LUXOR," Assees. of J. A. M. HAWADIER (B.P. 281,685, 1.12.27. Fr., 1.12.26).—A metallic core of relatively high m.p., *e.g.*, of tungsten, is covered with a metal of lower m.p., *e.g.*, molybdenum, and this is coated with a material, *e.g.*, chalk, magnesium carbonate, which has great illuminating power at a temperature below the m.p. of the metallic coating.

J. S. G. THOMAS.

[Electron-emission] composition. H. L. BERGER (U.S.P. 1,671,007, 22.5.28, Appl., 19.1.27).—A composite material of high emissive power is produced by boiling silver in an alkaline-earth hydrogen sulphide solution. A. R. POWELL.

Preparation of electron-emission material [thorium-molybdenum alloy]. J. W. MARDEN, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,670,463, 22.5.28, Appl., 11.4.23).—A mixture of molybdenum powder and the compound Th_3Al_4 is formed into bars, which are sintered in a high-frequency induction furnace *in vacuo* until the aluminium is volatilised and the thorium is thoroughly incorporated with the molybdenum. The alloy is then swaged, forged, and drawn or rolled as required. Alternatively, a molybdenum plate or anode is coated with Th_3Al_4 and heated in an evacuated valve whereby the thorium becomes incorporated with the molybdenum and the aluminium acts as a getter in the valve. A. R. POWELL.

Activation of electron-emission material. T. P. THOMAS, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,670,487, 22.5.28, Appl., 14.4.23).—The material is coated with thorium oxalate, introduced into an evacuated vessel, and heated to decompose the oxalate and liberate thorium. F. G. CROSSE.

Arc lamp electrodes. H. BECK (G.P. 446,881, 17.5.24).—The electrodes are provided with an outer layer of chromium which either volatilises completely or forms a porous slag. A. R. POWELL.

Primary cell. M. L. MARTUS and E. H. BECKER (U.S.P. 1,673,198, 12.6.28, Appl., 5.10.26).—A self-depolarising cell has an electrolyte of caustic alkali, and a negative element of activated carbon extending above the normal level of the electrolyte.

H. ROYAL-DAWSON.

[Diatomaceous absorbing medium for] battery. E. J. PETRIE and G. R. HANNAN (U.S.P. 1,673,421, 12.6.28, Appl., 10.3.26).—Electrolyte is absorbed in a medium consisting of diatomaceous shells substantially freed from foreign matter by heating to a temperature above the drying temperature and sufficiently high to drive off volatile constituents and destroy organic matter, but which is below the temperature at which fusion of the diatoms occurs. J. S. G. THOMAS.

Utilisation of the contents of used dry batteries. F. TROSTLER, and CHEM. FABR. JOHANNISTHAL G.M.B.H. (G.P. 447,740, 23.7.26).—The material is dried and intimately mixed with sulphur. The mixture is burnt in a multiple-hearth furnace with indirect firing, and the product is treated in an oil flotation machine. The concentrates are extracted with acid to remove manganese compounds and obtain a carbon of higher electrical conductivity than the original graphite used in making the batteries. A. R. POWELL.

Lead plates for accumulators. E. F. DRAISMA (B.P. 291,833, 22.2.27).—A plate of soft lead is enclosed in a frame or support of a lead-antimony alloy.

J. S. G. THOMAS.

Regeneration of the metal content of electrolytic [chromium] baths. ELEKRO-CHROM-GES. M.B.H. (G.P. 446,249, 28.12.23).—An auxiliary chromium cathode is

short-circuited with the anode of the plating bath, and the current strength of the auxiliary circuit is regulated by an electromagnet operated by the current in the main circuit, preferably through a relay. A. R. POWELL.

Electrolytic condenser and electrolyte therefor. H. N. MILLER, Assr. to FANSTEEL PRODUCTS Co., INC. (U.S.P. 1,672,899, 12.6.28, Appl., 5.3.27).—Filmed aluminium and conducting plates are retained in spaced relation by a dry gelatin-glycerin composition containing sodium bicarbonate. J. S. G. THOMAS.

Electrolytic condenser. E. F. ANDREWS, Assr. to ANDREWS-HAMMOND CORP. (U.S.P. 1,673,434, 12.6.28, Appl., 7.11.27).—A positive electrode composed of a nickel-iron alloy is used. J. S. G. THOMAS.

[Dry] battery cell. H. CSANYI, Assr. to L. KLOPMAN and W. W. HUSELTON (U.S.P. 1,675,973, 3.7.28, Appl., 17.9.24).—See B.P. 240,140; B., 1925, 997.

[Electrical] protection of metallic parts against corrosion. F. VON WURSTEMBERGER (U.S.P. 1,671,173, 29.5.28, Appl., 30.4.24, Switz., 14.5.23).

Bitumen for insulation (B.P. 291,858).—See II. **Metallurgical furnace** (U.S.P. 1,674,947). **Metallic sheets** (B.P. 292,015).—See X. **Articles from synthetic resins** (B.P. 291,477).—See XIII. **Electrodeposition of rubber** (B.P. 291,471).—See XIV.

XII.—FATS; OILS; WAXES.

Composition of the fatty acids present as glycerides in Elasmobranch oils. T. P. HILDITCH and A. HOULBROOKE (Analyst, 1928, 53, 246—257).—The oils examined consisted of (a) the mixed residual oils (after removal of squalene by vacuum distillation) from the livers of various species, including *Scymnorhinus lichia*, *Centrophorus granulosus*, *Lepidorhinus squamosus*, and *Etmopterus spinax*, and (b) the residual liver oil from *Scymnorhinus lichia*. Methods used were broadly those described previously (B., 1928, 306), and in the case of the first-named residue a check of the main results was obtained by analysing the fatty acids after complete hydrogenation. The total combined fatty acids from (a) contained solid acids 61.2%, liquid acids 38.8%. The solid and liquid acids, respectively, consisted of myristic 0.8, 0.2; palmitic 11.8, 1.4; stearic 1.2, 0.1; arachidic 1.2, —; myristoleic —, 0.2; palmitoleic 1.1, 2.4; oleic 18.2, 17.2; gadoleic 9.0, 7.4; cetoleic 12.3, 3.5; selacholeic 5.6, 6.4%. The composition of (b) agreed fairly closely with the above up to the C_{18} acids, but there was a difference of 6% less of oleic acid, and more cetoleic acid was present. Compared with normal marine animal oils, the degree of unsaturation of the fatty acids is relatively low, and they contain but little less saturated than monoethylenic derivatives, but the non-fatty matter is exceedingly highly unsaturated. Although C_{20} and C_{22} acids are present in about the same proportion as in other marine animal oils, they are mainly monoethylenic; there is also about 10—15% of a monoethylenic C_{24} acid present. D. G. HEWER.

Tung oil. D. HOLDE, W. BLEYBERG, and M. A. AZIZ (Farben-Ztg., 1928, 33, 2480—2484).—The iodine value (Hanus) of tung oil has been found to rise markedly

with increasing time of reaction from $\frac{1}{4}$ hr. to 2 hrs., and afterwards to remain steady at 230–240, corresponding to saturation of the three double linkings of the elæostearic acid present. A 0.2*N*-solution of iodine bromide in carbon tetrachloride gave a similar result in 3 hrs. As with other vegetable oils a constant iodine value is obtained after $\frac{1}{4}$ hr., it is suggested that the presence of tung oil in linseed oil might be recognised quantitatively by observation of an iodine value increasing with the time of reaction, and *vice versa*. In the case of β -elæostearic acid, the iodine value rose from 176 to 279 during $\frac{1}{2}$ –2 hrs. treatment (45% excess of iodine bromide); 60% excess of the reagent gave iodine values up to 300, and, in a few cases, values up to 350 (equivalent to four double linkings) were attained, resulting possibly from the occurrence of substitution or of traces of water in the glacial acetic acid etc. Solutions of iodine or bromine alone were less reactive than was iodine bromide. E. LEWKOWITSCH.

Organic peroxides as bleaching agents: application to oils and fats. F. V. HOORT (Oil & Fat Ind., 1928, 5, 180–182).—The use of benzoyl peroxide as an oxidising bleaching agent for oils and fats is suggested. The reagent (about 0.5–0.2%) is mixed to a paste with some of the oil, and the mixture added to the bulk, which is heated to 80–90°. The whole is stirred and left for a sufficient time (20 min. or longer). The most suitable proportion of reagent, temperature and time of reaction should be determined by preliminary laboratory tests for each oil to be treated. No filtering is necessary, and steaming removes residual benzoic acid. E. LEWKOWITSCH.

Report of Du Boscq Colorimeter Committee. D. WESSON (Oil & Fat Ind., 1928, 5, 173–175).—To test the suggestion that by the use of the Du Boscq colorimeter it should be possible to read the colour of cottonseed oils in terms of "prime summer yellow," duplicate sets of samples were tested by four different laboratories. Considerable differences in the readings were shown (greater than with the Lovibond tintometer). Matching was complicated by differences in hue due to the presence of several different colouring matters, and to the effect of bleaching etc. of the oils, and different results were obtained with different depths of the standard oil. It is concluded that the Du Boscq instrument, although suitable for measuring relative amounts of the same colouring matter in optically similar liquids, needs modification for successful application to oil colorimetry. E. LEWKOWITSCH.

Paraffin waxes. MEYERS and STEGEMAN.—See II.

PATENTS.

Manufacture of acid- and lime-resisting derivatives of unsaturated fatty acids. I. G. FARBENIND A.-G. (B.P. 288,127, 23.12.27. Ger., 1.4.27).—A lower fatty acid or its anhydride is treated with fuming sulphuric acid to give the corresponding acyl derivative, which is then coupled with an unsaturated hydroxy-fatty acid, anhydride, or ester. The process has the advantage that it may be carried out at moderate temperatures and gives light-coloured products of good stability. E. HOLMES.

Production of sulphonation products from fats, oils, or their acids. H. T. BÖHME A.-G., and H. BERTSCH (B.P. 274,104, 8.7.27. Ger., 8.7.26).—Fatty materials which may be sulphonated by means of sulphuric acid in the presence of anhydrous strong organic acids, or their anhydrides or chlorides, give products of enhanced utility if they are first polymerised. Thus "Floridin," which results from the dry heating of castor oil, may be used (cf. B.P. 261,385; B., 1928, 418). E. HOLMES.

Apparatus for sulphonating oils. A. HORWITZ (U.S.P. 1,671,586, 29.5.28. Appl., 22.6.21).—Uniform sulphonation of large batches of oil is achieved by forcing the sulphonating acid under air pressure through numerous nozzles placed above the surface of the oil, which is stirred in an open tank fitted with cooling coil and run-off valve. C. HOLLINS.

Purification or separation of mixtures of fatty or mineral oils. J. Y. JOHNSON. From I. G. FARBENIND A.-G. (B.P. 291,817, 7.1.27).—The application of solvents consisting of the formate of a lower fatty alcohol or of mixtures of such formates is claimed. Thus methyl formate readily removes free acids from sesamum oil, sulphur compounds from Mexican crude oil, and a golden-yellow resin from the higher oily residue from the catalytic hydrogenation of a crude rock oil. E. HOLMES.

Wax-like compounds (B.P. 291,849, 292,056, 292,058, and 292,057).—See III. **Fatty still residues.** (U.S.P. 1,669,490—1).—See XIV.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Loss of material of paints by weathering. H. A. MÖLLER (Farben-Ztg., 1928, 33, 2484–2486).—Quantitative experiments on the weathering of white lead paints spread on sheet iron were made and the "half-life periods" of the coatings calculated for comparison. Greywhite lead paints proved notably more resistant than white, the weathering varying according to the nature of the black pigment incorporated. Sulphate white lead paints preserved the surface gloss longer, collected less dirt, and had a longer life than those containing carbonate white lead. In all cases loss of material was less for those paints having a red lead undercoat. E. LEWKOWITSCH.

Composition of zinc chrome. B. A. ELLIS, J. J. FOX, and J. F. HIRST (J. Oil and Colour Chem. Assoc., 1928, 11, 194–198).—The divergence in composition assigned to zinc chrome in the literature is due to incomplete methods of analysis and to different methods of manufacture. A complete scheme of analysis is detailed and the results of examination of commercial zinc chromes of various types are tabulated. The probable composition $5\text{ZnO} \cdot 4\text{CrO}_3 \cdot \text{K}_2\text{O} \cdot 4\text{H}_2\text{O}$ is indicated for potassium zinc chrome and $2\text{ZnO} \cdot 2\text{CrO}_3 \cdot (\text{NH}_4)_2\text{O} \cdot \text{H}_2\text{O}$ for the dark-orange ammonium zinc chrome, but approximate agreements with stoichiometrical ratios are not considered of much importance for such complicated substances. S. S. WOOLF.

Precipitation of zinc chromate. J. L. BUCHAN and J. J. FOX (J. Oil and Colour Chem. Assoc., 1928, 11, 198–200).—A description of some experimental precipitations of zinc chromes, undertaken in order

to ascertain how the various types of commercial pigments (cf. preceding abstract) may be obtained.

S. S. WOOLF.

Titanium dioxide rubber enamels. R. DITMAR (Chem.-Ztg., 1928, 52, 506—507).—Fine-grained titanium dioxide is very suitable for the production of snow-white and highly luminous, coloured rubber enamels, since it has a better white-colouring power than, for example, zinc oxide, improves the rubber material, and, unlike colouring agents containing zinc, is non-poisonous. Such enamels may be produced from vulcanised rubber since the latter is rendered very plastic and adhesive by the titanium oxide. By the use of sulphur monochloride vapour the enamels may be vulcanised in 6 min. instead of the usual 34—44, giving well-aged and more durable products.

B. P. RIDGE.

Malayan damars. II. T. H. BARRY (J. Oil and Colour Chem. Assoc., 1928, 11, 203—216; cf. B., 1925, 815).—An account of the characteristics, composition, and analytical constants of dead damar, black damar, and damar Minyak.

S. S. WOOLF.

[Liebermann]-Storch-Morawski reaction for rosin. E. ELSNER (Farben-Ztg., 1928, 33, 2547—2548).—The unreliability of the positive Liebermann-Storch-Morawski reaction for rosin is discussed. It is further shown that solutions of rosin and various rosin products in turpentine give a negative reaction up to a concentration of 10%, and a very fleeting positive reaction at somewhat higher concentrations. A negative rosin test obtained on varnishes containing turpentine cannot, therefore, be relied on, contrary to previous views.

S. S. WOOLF.

Resins of lignite. WIGGINTON.—See II.

PATENTS.

Preparation for stripping paint, varnish, and the like. F. C. DEAN and O. P. SWIFT (B.P. 291,812, 8.12.26).—A preparation which remains wet is made from pure benzol, crude benzol (or coal-tar naphtha), methylated spirit, paraffin wax, and ceresin wax.

B. FULLMAN.

Production of printing ink. J. J. JAKOSKY (U.S.P. 1,673,245, 12.6.28. Appl., 1.11.26).—A portion of a quantity of oil suitable for use as a vehicle in printing ink is heated locally to a temperature sufficiently high to decompose it partly into carbon and gaseous products, when it is immediately mixed with the surrounding oil. The process is continued until at least part of the oil contains sufficient carbon for use as printing ink, and this is then separated from the remaining oil.

L. A. COLES.

Manufacture of water-colour printing ink. J. HOEFFLER, Assr. to J. HOEFFLER & Co., Inc. (U.S.P. 1,660,196, 21.2.28. Appl., 12.3.27).—A mixture of gum arabic solution and an excess of glycerin is dehydrated by heat to produce a paste, a pigment is added, and the product ground.

H. ROYAL-DAWSON.

Manufacture of coloured solutions of nitrocellulose. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 291,539, 10.3.27).—Insoluble mineral or organic colouring matter in water or other indifferent liquid is mixed (in a Kollergang or kneading machine etc.) with

nitrocellulose wet with water, in the absence of gelatinising agents, to homogeneity. The product is pressed, freed from water by extraction or heat, and dissolved in the usual varnish solvents.

B. FULLMAN.

Coating materials. [Water japans.] BRIT. THOMSON-HOUSTON Co., LTD., Asses. of W. P. DAVEY (B.P. 275,955, 25.7.27. U.S., 16.8.26).—To the water japan made according to B.P. 212,248 (B., 1925, 557) ammonia is added. The coatings electrically deposited from the japan are thickened and improved.

B. FULLMAN.

Coating by depositing emulsified japan ingredients. BRIT. THOMSON-HOUSTON Co., LTD., Asses. of H. CHISLET (B.P. 275,960, 26.7.27. U.S., 16.8.26).—Surfaces are coated with a solution of an electrolyte (preferably an alkali), dried, and dipped into an aqueous japan emulsion. The disperse phase in a layer of the emulsion is coagulated by the electrolyte, forming an adherent layer on the surface.

B. FULLMAN.

Production of varnish bases and masses insoluble in water. G. WALTER (B.P. 262,148, 29.11.26. Austria, 28.11.25).—The primary condensation products from formaldehyde and carbamide or thiocarbamide are heated rapidly to above the m.p., with the addition of carbamide, thiocarbamide, or acid amides in general, and/or alcohols (benzyl alcohol), together with acids, such as chloroacetic or acetic acid with or without buffer salts. The additions may be made to the solid or molten condensation product, and, if desired, a non-aqueous solvent (volatile or not) may be employed. Glassy or white masses of varying degrees of hardness are obtained according to the ingredients and conditions. The use of much acetamide and benzyl alcohol leads, after addition of volatile solvent (methyl alcohol), to clear lacquers. The products may be hardened by a further heat treatment; the process and the hardening may be performed under pressure.

C. HOLLINS.

Treatment of rosin. R. C. PALMER, Assr. to NEWPORT Co. (U.S.P. 1,660,762, 28.2.28. Appl., 4.10.24).—A mixture of zinc chloride and rosin is heated at 250—330°, and the product distilled *in vacuo* to produce rosin oils.

H. ROYAL-DAWSON.

Resinous reaction product of urea [carbamide] and formaldehyde. F. LAUTER, Assr. to ROHM & HAAS Co. (U.S.P. 1,671,596, 29.5.28. Appl., 7.5.26).—In order to avoid cloudiness due to methylenecarbamide, hot carbamide is added to a boiling solution of formaldehyde.

C. HOLLINS.

Manufacture of articles from synthetic resins or shellac. DUNLOP RUBBER Co., LTD., and R. C. DAVIES (B.P. 291,477, 3.12.26).—Synthetic resins, *e.g.*, of the phenol-formaldehyde type, or mixtures of these with natural resins, *e.g.*, shellac or rubber, in the form of aqueous dispersions are electrodeposited on an anode or on a porous mould surrounding the anode. Instead of using preformed resins, the dispersion may contain the resins in a partially formed condition or the components for forming the resins. Compounding ingredients also may be present in the dispersion. The deposit, which may be left on the anodic mould or may be stripped therefrom, is then dried and heated to complete the hardening.

D. F. TWISS.

Manufacture of plastic masses from albuminous substances, like casein. R. and M. SCHRODER, E. JAROSLAW, and S. LEVIS [JAROSLAW'S ERSTE GLIMMERWAREN-FABR. IN BERLIN] (B.P. 272,947, 17.6.27. Ger., 17.6.26).—Alkaline solutions of albuminous substances, *e.g.*, casein, blood, intestinal mucus, etc., containing a phenol are treated with an aldehyde in the presence of ammonia. B. FULLMAN.

Hardening of plastic masses from casein and the like. I. G. FARBENIND. A.-G. (B.P. 268,804, 31.3.27. Ger., 31.3.26).—Hardening takes place during hot pressing if the plastic mass contains compounds (*e.g.*, methylolureas and urethanes) which, in the temperature range employed, yield formaldehyde and difficultly volatile substances which improve the properties. B. FULLMAN.

Hardening resinous exudations. E. A. DANIELS and H. S. SNELL, ASSTS. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,673,803—8, 19.6.28. Appl., 21.8.24).—Resinous exudations are condensed with (A) a guanidine, (B) aldehyde-ammonia, (C) an aminonaphthalene, (D) a secondary aromatic amine, (E) a piperidine-carbon disulphide additive product, (F) sodium β -naphthoxide. L. A. COLES.

Condensation of urea and formaldehyde under pressure. A. GAMS and G. WIDMER, ASSTS. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,674,199, 19.6.28. Appl., 4.1.26. Switz., 17.1.25).—See B.P. 246,127; B., 1927, 684.

Mills for reducing paints, colours, and pigments. J. R. TORRANCE, and TORRANCE & SONS, LTD. (B.P. 292,431, 3.1.28. Addn. to B.P. 209,829; (B., 1924, 238).

Blanc fixe (G.P. 445,668). Lakes from phosphotungstomolybdenum compounds (B.P. 292,253).—See VII. Acid-proof structures (B.P. 292,334).—See IX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Ageing of vulcanised rubber. IV. Relation between wave-length of light and deterioration of vulcanised rubber. T. YAMAZAKI (J. Soc. Chem. Ind. Japan, 1927, 30, 804—813).—Vulcanised rubber became darker when merely heated in the dark at 70°, but when exposed to sunlight at 60—70° the change in colour was far more marked. The higher the degree of vulcanisation, the greater was the colour change. Moreover the rubber was hardened by the action of light, such action being quicker when the wave-length of the light was shorter and the temperature higher. The action of light of 4300Å. was feeble, except for over-vulcanised rubber, which was noticeably hardened even by light of longer wave-length. Acetone extract increased with time of exposure, the rate of increase being less when light of longer wave-length was used. In some cases the acetone extract reached a maximum and then decreased. Remarkable decrease in free sulphur by the action of light and heat was also noticed. Y. NAGAI.

Ageing of vulcanised rubber. V. Effect of sunlight and heat on the mechanical properties of vulcanised rubber. VI. Effect of sunlight filtered through coloured glass on mechanical properties of vulcanised rubber. T. YAMAZAKI (J. Soc. Chem.

Ind. Japan, 1928, 31, 233—242, 243—257).—The time of vulcanisation of rubber-sulphur mixes was found to have a marked effect on the ageing properties. The deterioration due to sunlight was great even in winter, and still more pronounced in spring at 60°. The content of acetone-soluble matter increased during ageing and the content of free sulphur decreased. The outer layers of test pieces exposed to sunlight were quite brittle and non-elastic, but the inner layers were fairly good. Test pieces subjected to Geer tests showed little difference between outer and inner layers. Test pieces exposed to the light through a coloured glass filter transparent down to 3300Å. showed serious deterioration even at 40°, but those exposed to a filter transparent to 4360Å. showed only slow deterioration at 40° but a somewhat marked one at 65°. S. OKA.

Homogeneous rubber and the conception of an elastic molecule. H. FEUCHTER (Kautschuk, 1928, 4, 103—106, 118—123).—Features in common between the characteristics of liquid crystals and those of the elastic-plastic state of rubber are indicated, together with the possibility of applying knowledge of the former to elucidation of the constitution of rubber. The conversion of anisotropic racked rubber into the isotropic material probably represents, in principle, the same thermal function as the melting of liquid crystals. The rubber fibres are regarded as bundles or parallel groupings, possibly spiral in configuration, of units with a linear molecular character, which presumably represent the chains of isoprene or dimethyloctadiene nuclei suggested earlier, *e.g.*, by Pickles, Harries, or Staudinger. From the possibility of removing the racking effect, it is necessary to postulate the existence of periodic weaker linkings between some of the isoprene links in a chain; the linking arrangement in rubber, therefore, resembles in some respects an elastic lattice or cross-woven fabric. Rubber in its various elastic forms can be regarded as a dynamic, completely reversible mixture of an ideal elastic, or "iso-elastic," rubber ("ideale Kautschuk"), to which "diffusion rubber" is an approximation, with another ideal hard, vitreous form ("glasartige Kautschuk"), which is designated "para-elastic."

D. F. TWISS.

Rubber enamels. DITMAR.—See XIII.

PATENTS.

Production of rubber [by electrodeposition]. S. O. COWPER-COLES (B.P. 291,471, 20.11.26, 14, 15, 16, and 22.12.26, 8.1, 3.2, 11.2, 14.6, and 2.8.27).—In the production of rubber sheets, tubes, etc. from latex, containing compounding ingredients if desired, by electrodeposition on an anode mould of cadmium or zinc, the escape of anodic bubbles is facilitated and homogeneity of the deposit is ensured by effecting deposition intermittently, *e.g.*, by an intermittent or alternating current, by periodic withdrawal of the anode from the latex, or by rotating the anode on a horizontal axis giving only partial immersion of the mould. A soft, electrically-conductive deposit is ensured by neutralisation of the ammonia in preserved latex with acetic acid. Metal powder may be incorporated in the deposit by dusting or by electrodeposition subsequent to the formation of the rubber deposit. Electrodeposition of rubber is also

effected by spraying latex from a negatively-charged nozzle on to an anodic mould. D. F. TWISS.

Treatment of latex and products obtained therefrom. W. A. GIBBONS and J. MCGAVACK, Assrs. to U.S. RUBBER PLANTATIONS INC. (U.S.P. 1,673,672, 12.6.28. Appl., 11.5.27).—Latex containing an anti-coagulant which permits bacterial action is kept until the protein constituents are substantially decomposed. On evaporation a transparent film is obtained with a pitted surface. D. F. TWISS.

Manufacture of rubber articles. M. C. TEAGUE, Assr. to AMER. RUBBER CO. (U.S.P. 1,673,649, 12.6.28. Appl., 25.7.25).—A relatively thick layer of an aqueous dispersion of rubber, in the presence of a hydrophilic agent, is exposed to drying conditions, whereby moisture is withdrawn substantially uniformly from all parts without the formation of a dried surface film. D. F. TWISS.

Manufacture of hollow rubber articles. DUNLOP RUBBER CO., LTD., D. F. TWISS, and E. A. MURPHY (B.P. 291,479, 4.12.26).—Hollow articles of rubber or similar material are formed from a concentrated and/or compounded dispersion on a temporary form or mandrel, e.g., of fusible metal, wax, or clay, which may subsequently be removed as a liquid or as a suspension in a fluid. Vulcanisation can be effected before or after removal of the form. D. F. TWISS.

Production of rubber-like bodies. W. B. PRATT, Assr. to DISPERSIONS PROCESS, INC. (U.S.P. 1,671,314, 29.5.28. Appl., 17.2.26).—Natural rubber is imitated by dispersing a terpene (isoprene, dipentene, or polymerides) in water in presence of a hydrophile protective colloid of protein character, the terpene particles being about the size of the particles in rubber latex. The coating of protective colloid is then tanned by the addition of cresol, creosole, or other tanning agent, so as to prevent rupture of the globules during milling. When coagulated the product forms a rubber-like mass capable of vulcanisation, swelling in organic solvents without rupture of the coatings. Natural or synthetic resin may be added to give an acetone-soluble ingredient. C. HOLLINS.

Reclaiming and dispersing vulcanised rubber. T. G. RICHARDS and G. P. F. SMITH, Assrs. to DISPERSIONS PROCESS, INC. (U.S.P. 1,671,316, 29.5.28. Appl., 17.5.27).—In the alkali process for reclaiming vulcanised scrap rubber, a saponifiable substance (resin oil, acid resins, pine tar, higher fatty acids) is added so that a soap is formed *in situ*, which enables the reclaim to be dispersed readily in water. C. HOLLINS.

Vulcanisation accelerator. H. O. CHUTE (U.S.P. 1,673,801, 19.6.28. Appl., 21.12.22).—An accelerator is prepared by adding carbon disulphide to a mixture of magnesia with an aromatic amine in the cold, and subsequently heating below 140° until evolution of hydrogen sulphide is practically complete. L. A. COLES.

Vulcanised pitches. Treatment of fatty still residues. W. SAVAGE, Assr. to SAVAGE-RUBBER CORP. (U.S.P. 1,669,490—1, 15.5.28. Appl., [A] 17.1.22, and [B] 12.6.23).—The black tarry residue obtained as a by-product in the refining of fatty acids by distillation is

vulcanised (A) with 7% of sulphur at 150—205° in the presence of zinc oxide and thiocarbanilide, or (B) with more than 5% of sulphur at 135—205° in the presence of an organic accelerator base and an active alkaline compound, e.g., magnesia, magnesium carbonate, or lead oxide. The products are soft, rubbery substances, which may be hardened by the introduction of any of the usual rubber fillers. A. R. POWELL.

Covering of wire, thread-like and filamentary material, etc. with india-rubber, gutta-percha, balata, etc. DUNLOP RUBBER CO., LTD., D. F. TWISS, and E. A. MURPHY (B.P. 291,485, 28.1.27).—Wire and thread-like material is passed through a bath of concentrated and/or compounded aqueous dispersion of rubber, gutta-percha, or balata, so that it issues therefrom with a uniform coating of the dispersion. In leaving the bath the filament conveniently passes through a short constricted tube one end of which is immersed in the bath. The coating is dried without contact with any foreign material. The filament may consist of colloidal material capable of being absorbed into the coating during subsequent drying or vulcanisation. D. F. TWISS.

Antimony pentasulphide (U.S.P. 1,671,203).—See VII.

XV.—LEATHER; GLUE.

Depilation [of skins] by mould enzymes. G. ABT (J. Soc. Leather Trades' Chem., 1927, 11, 520—530).—*Aspergillus oryza*, *A. effusus*, *A. parasiticus*, *A. ochraceus*, and *A. flavus* contain enzymes capable of loosening the hair on skins. The most active is *A. oryza*, which produces an amyolytic enzyme, a lipase, and a protease. It is the protease which causes depilation, but the lipase probably assists. It is most active at a much lower temperature than pancreatin. It will loosen the hair at 20°, but 25—28° is the optimum temperature and 35—38° is best for goatskins. 0.4% boric acid solution or 0.4—0.8% sodium bicarbonate solution should be added to the unhairing liquor as an antiseptic. For depilation the process occupies about 24 hrs. Dried skins should be soaked back before treatment, and goatskins should be treated in water at 28°. The action of the mould enzymes is stimulated by pretreating the skins with a 0.2—0.4% solution of sodium hydroxide for 24 hrs. The optimum effect of the mould protease on casein solution is at p_H 6.5—7.0, but the optimum unhairing effect appears to be at p_H 8.0. The two effects are not the same. Sections of skins unhaird by this mould show the entire removal of the epidermis, the hair bulbs, fat, and sweat glands, and good separation of the fibrils. The appearance resembles that obtained after the best bating materials. Pancreatin appears to attack the elastic and collagen fibres, whilst the protease in *A. oryza* has a selective action on the epidermal tissues, the epidermal mucous layer, and the hair structures. It also exerts much less hydrolytic action on collagen than does pancreatin, but it hydrolyses limed collagen fairly readily. D. WOODROFFE.

Chamoising of skins. P. CHAMBARD and L. MICHALLET (J. Soc. Leather Trades' Chem., 1927, 11, 559—576).—Chamoising consists of two different processes; the tannage proper, in which the collagen is converted

into a new non-hydrolysable substance with changed properties, and a filling or adsorption process, in which a certain amount of saturated glycerides lubricate the leather. Various theories as to the reactions taking place during chamoising have been critically examined, and experimental work has failed to confirm either the peroxide or the aldehyde theory, to the exclusion of the other. It is concluded that the intermediate oil-oxidation products are chiefly responsible for the tannage, and that the secondary adsorption process is essential to the properties of chamois leather. The products of complete oxidation and of polymerisation of the neutral glycerides and fatty acids are absorbed by the skin. The oxidation and polymerisation products of the oil are soft, clear, and amorphous, whilst the polymerised fatty acids are brownish-black, hard, and brittle. Hence the difference between skins tanned with fish oils and fatty acids respectively. Neutral cod oil is the best chamoising agent and produces a leather of good quality, but the tannage is slow. Free fatty acids furnish a rapid method of tannage, but produce a harsh leather. The most practicable chamoising oils contain some free acids. Resinates of cobalt, iron, lead, and manganese catalyse the chamoising in order of decreasing effect. 0.1% can be used to increase the amount of oil combined with the pelt after 24 hrs.

D. WOODROFFE.

Tanning barks in Madagascar. F. H. DE BALSAC and A. DEFORGE (J. Soc. Leather Trades' Chem., 1927, 11, 551—558).—Analyses of five samples of mimosa bark, *Acacia decurrens*, showed water 9.39—12.65%, tans 24.3—27.84%, soluble non-tans 5.36—10.24%, insolubles 51.86—57.2%, and ratio of tan/non-tan 2.7—5.3. Their tannin content was lower than that of Natal mimosa bark, but the ratio of tan/non-tan was equal to or better than that of the Natal bark. The Madagascar barks are not suitable for export, but only for local use or the manufacture of dry extract. Other barks examined are: badamier (*Terminalia Catappa*), lalona (*Weinmannia Bojeriana*, Tul), filao (*Casuarina equisetifolia*, Forst), rotra (*Eugenia sp.*), fany (*Peptadenia chrysostachys*, Benth), and sakoa (*Soleroarya caffra*, Sonder), the percentage tans and non-tans in which are: 11.8, 4.4; 13.7, 2.7; 15.3, 3.7; 12.6, 12.2; 3.1, 17.0; and 3.5, 4.2, respectively. Fany, sakoa, and rotra contain too much non-tans to be useful in tanning. Lalona gives too dark a tannage for European tanners, and if used for extract manufacture it would require decolorising. Filao is the most promising material for cultivation purposes. D. WOODROFFE.

Chromium collagenates. C. SCHIAPARELLI and G. BUSSINO (J. Soc. Leather Trades' Chem., 1927, 11, 531—550).—Samples of delimed bated pelt were shaken with 3% and 5% solutions of chrome alum respectively. The increase in weight and the temperature of gelatinisation (T_G) were noted at regular intervals. T_G increased as the weight increased. The rate of tannage and rise in T_G decreased at a certain stage and then increased again. This was probably due to the conversion of chromium collagenate into dichromium collagenate. The tanned pelt contained 3.91% Cr_2O_3 at this stage, compared with 3.38% for monochromium collagenate, and had T_G 92—94°. The concentration

of the chrome alum solution did not affect the nature of the tannage, but increased its speed. Tannage with a 5% solution of basic chrome alum (basicity 16.6%) showed a break at T_G 108—110° (Cr_2O_3 8.41%) which is attributed to the conversion of dichromium collagenate (Cr_2O_3 6.76%) into the trichromium compound. Tannage with a liquor of basicity 33.3% showed a break at T_G 120° (in glycerol) and the pelt contained on the average 9.37% Cr_2O_3 . With a liquor of 50% basicity, the T_G increased to 130°, tetrachromium and a little pentachromium collagenate being formed. T_G was constant at 134—138° with a liquor of 66.6% basicity, and the increase in weight was nearly double that obtained with the liquor of 50% basicity. A heptachromium collagenate has been obtained, but not the octachromium compound (cf. Thomas and Kelly, B., 1922, 640 A). The results confirm Wilson's theory on chromium collagenates and the preponderating chemical nature of chrome tannage with chrome alum. Ageing is the conversion of simple chromium collagenates into higher compounds, with the elimination of acid. A 16.6% basic chrome alum solution suffices for a light chrome tannage, and a 33.3% solution is most suitable for "box" or moderate tannages. Experiments show that the boiling test does not indicate complete tannage. The determination of T_G would afford a better control.

D. WOODROFFE.

Synthetic tannins. L. MEUNIER and C. CASTELLU (J. Soc. Leather Trades' Chem., 1927, 11, 495—507).—See B., 1928, 133.

Determination of insoluble [matter] in tanning extracts. A. TURNBULL (J. Soc. Leather Trades' Chem., 1928, 12, 202—3).—See B., 1928, 534.

PATENTS.

Manufacture of tanning agents. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 291,245, 18.6.27).—The water-soluble products obtained from phenols (phenol, *m*-cresol, *p*-chlorophenol) by condensation with formaldehyde and a sulphite are heated with sulphur and water in presence of alkali, particularly the alkali liberated when a neutral sulphite is used with the formaldehyde. C. HOLLINS.

Production of [metal]-decorated articles from casein. A. I. G. WARREN, and PRECIOUS METAL INDUSTRIES, LTD. (B.P. 291,877, 10.3.27).

XVI.—AGRICULTURE.

Soils of Prince Edward Island. F. T. SHUTT (Can. Dept. Agric., 1928, Bull. No. 100, 20 pp.).—Analyses of a large number of cultivated soils are recorded. For the maintenance of fertility, the adoption of green manuring supplemented by the judicious use of fertilisers and lime is recommended.

A. G. POLLARD.

Exchange acidity in primary soils. R. PÜRCKHAUER (Z. Pflanz. Düng., 1928, 11A, 359—386).—The weathering of primitive rocks tends to produce an acid soil which becomes more neutral during cultivation. Examination of numerous soil profiles indicates that the reaction of the primary soil lies within a fairly narrow range of p_H values and is not appreciably influenced by the depth of the weathered stratum or the nature of the

original rock. Forest growths tend to intensify the acidity of these soils. Differences in the reaction of meadow and arable land are small and traceable to differences in manuring. Pasturage maintains a favourable reaction, and use for garden cultivation tends to produce an alkaline reaction. Acid fertilisers do not markedly influence the p_H value of this type of soil, but lime causes profound changes. Natural manures are the most suitable as the soils usually have low buffer capacities. Good growth of clover is possible with soils of p_H value less than 4.5. In acid soils white charlock (*Raphanus raphanistrum*) occurs in its deep yellow modification, and a relationship is suggested between this phenomenon and the p_H value of the nutrient medium. A. G. POLLARD.

Nature of base exchange and soil acidity. H. W. KERR (J. Amer. Soc. Agron., 1928, 20, 309—335).—The exchange reactions of soils are ascribed to the open structure or colloidal nature of the complex, and a mass-action equation for the heterogeneous equilibrium is formulated. Zeolitic materials appear to be present in the soil. Experiments with colloidal aluminosilicic acid show that acids need not be in true solution in order to give acid p_H values by an electrometric method. The difference in the chemical behaviour of colloidal and dissolved materials is of degree rather than of kind.

CHEMICAL ABSTRACTS.

Replaceable bases and dispersion of soil in mechanical analysis. M. D. THOMAS (Soil Sci., 1928, 25, 419—427).—The maximum amount of dispersion is to be obtained in the majority of cases by producing sodium saturation in the soil mass. In view of the experimental difficulty attending the preparation of sodium-saturated soil it is likely that a treatment with dilute acid followed by the addition of sodium carbonate is the best routine method of dispersing non-calcareous soil. H. J. G. HINES.

Aqueous vapour pressure of soils. IV. Influence of replaceable bases. M. D. THOMAS (Soil Sci., 1928, 25, 484—493; cf. B., 1928, 538).—The vapour pressure-moisture curves of soils treated to exchange their bases for a single base are greatly influenced by the nature of the replaceable base. In dry soils the potassium-treated material has the least and the calcium-treated material the greatest water-absorbing power, but at high moisture contents the sodium-saturated material has the greatest absorbing power. In the study of a colloidal mineral it was shown that various hydrates of its sodium complex exist. Soils exhibit similar properties to this mineral, and it is suggested that the soil colloidal material resembles it in composition. H. J. G. HINES.

Studies with sulphur for improving alkali soil. J. D. HAYNES (Soil Sci., 1928, 25, 443—446).—The permeability of the soil was improved (a) by alternate wetting and drying, (b) by treatment with sulphur. Lime and manure maintained this improvement. Of the substances examined, sulphur was the most effective in decreasing the p_H of the soil. H. J. G. HINES.

Behaviour of the nitrogen of spent wash in the soil. E. PARISI (Annali Chim. Appl., 1928, 18, 198—204).—The absorptive power of soil for the amino-

acids of spent wash from molasses distilleries is either feeble or non-existent. The ammonification of the amino-acids and betaine occurs regularly in the soil, but is very slow, so that there is danger of the washing-out of these substances. Losses due to the development of gaseous nitrogen in soils fertilised by spent wash and containing a proper proportion of moisture are practically negligible, and even when large amounts of the slop are used, the nitrogen is transformed almost quantitatively into nitrates. T. H. POPE.

Calcium carbonate-soil equilibrium and the lime requirement. S. MATTON (Soil Sci., 1928, 25, 429—431).—When calcium carbonate is added to the soil, part of the calcium is adsorbed and part dissolved as bicarbonate by the carbon dioxide so liberated. By placing soil colloidal material in a parchment bag which was in contact with a paste of calcium carbonate, it was shown that the soil-calcium carbonate equilibrium was reached in seven days. By replacing the adsorbed calcium with ammonium chloride, the degree of unsaturation, and hence the lime requirement, can be found. H. J. G. HINES.

Rate of availability of various forms of sulphur fertilisers. J. D. HAYNES (Soil Sci., 1928, 25, 447—453).—A sulphur preparation inoculated with *Thiobacillus oxidans*, a colloidal sulphur preparation, and other commercial by-products were mixed with soil, and the rate of oxidation to sulphate was measured. Of the by-products, black gas-sulphur (the colloidal preparation) was oxidised as rapidly as the inoculated sulphur. On alkali soils sulphur decreased the p_H value, increased the microflora of the soil, and produced a decided deflocculating effect. H. J. G. HINES.

Do soils become more acid on drying? W. BRENNER (Z. Pflanz. Düng., 1928, 11A, 141—144).—The reaction of normal soils is but slightly altered on drying. H. J. G. HINES.

Influence of pulverisation and drying of soil on its productivity. A. ACHROMEIKO (Z. Pflanz. Düng., 1928, 11A, 65—89).—The drying of soil in the sun results in increased solubility in water of phosphates and organic matter. Repeated drying multiplies this effect, which is even more pronounced on oven-drying or steam-sterilisation. On re-wetting the soil reversion occurs. Similar effects on the reaction were also noticed and ascribed to microbiological action. The alterations in structure on drying and re-wetting are due mainly to the changes in the organic matter. H. J. G. HINES.

Phosphate applications and their influence on chernosem [soils]. IV. Reversion of phosphates in soils. M. A. EGOROV (Soil Sci., 1928, 25, 463—472; cf. Egorov and Mackov; B., 1926, 763).—The addition of sodium phosphate to soil causes a large part of the organic matter to become water-soluble. Sodium hydrogen phosphate gives a less coloured extract. It is proposed to use this action on the soil organic matter to follow the changes in composition of various phosphates when added to the soil. H. J. G. HINES.

Chemical determination of soil fertility. K. BAMBERG (Z. Pflanz. Düng., 1928, 11A, 115—141).—The

phosphoric acid in soils (particularly humus soils) is rendered more soluble in citric acid by drying; the solubility in nitric acid, however, is but little changed. A comparison of the Mitscherlich and Neubauer methods with a number of chemical methods was carried out on a large number of soils. The chemical methods included extraction of phosphates and potash with citric acid, ammonium citrate, nitric acid, and mixtures of ammonium citrate and nitric acid. It is concluded that the value of chemical methods cannot be disputed, and that they do give true indications as to the response of soils to phosphatic manuring.

H. J. G. HINES.

Theoretical considerations on the determination of soil nutrients. S. GERICKE (*Z. Pflanz. Düng.*, 1928, 11A, 144—150).—The citric acid extraction method is criticised on the ground that the final reaction of the extracted soil varies according to the initial characteristics of the soil. In the Neubauer method the influence of the added sand modifies the texture of heavy soils considerably, and hence no true notion of the actual behaviour of the soil is obtained. The seedling method also takes no account of the influence of one nutrient on the amount of another nutrient taken up by the plant.

H. J. G. HINES.

Use of the Dumas-Dennstedt method in the determination of total nitrogen in soils and agrarian products. E. PARISI (*Annali Chim. Appl.*, 1928, 18, 196—197).—For determining nitrogen in soil, flour, and other agricultural products, Dennstedt's method is recommended in place of that of Dumas. The hard glass combustion tube may be replaced by one of opaque quartz and the manganese carbonate or sodium hydrogen carbonate used as a source of carbon dioxide by a Kipp's apparatus. If 8—10 g. of soil are taken, duplicate tests agree within about 2—3%.

T. H. POPE.

Determination of organic matter and especially humified substances in soil. U. SPRINGER (*Z. Pflanz. Düng.*, 1928, 11A, 313—359).—Comparative examination of existing methods is recorded. Among processes for determining the total organic matter in soils a modified chromic acid method is recommended. Methods for the selective analysis of humic material by extractive processes are unsound, in that portions only of the humic matter are removed (except in the case of sodium hydroxide extracts) and also some undecomposed plant residues are frequently affected. A modification of Grandeau's method (extraction with 5% caustic soda solution and oxidation with permanganate) yielded results generally satisfactory for comparative work, but lower than those obtained by the acetyl bromide method. During the colorimetric examination of alkaline extracts of soil some decomposition and loss of colour intensity may occur. Of the direct methods that of Lapicque and Barbé, whilst of little value for quantitative measurements, yields valuable information as to the state of decomposition of humus and is simpler than those of König and of Robinson and Jones. The acetyl bromide method is the most accurate of those examined.

A. G. POLLARD.

Use of highly viscous fluids in the determination of volume weight of soils. S. H. BECKETT (*Soil Sci.*,

1928, 25, 481—483).—A rapid and accurate determination can be made by removing the soil with an auger, and collecting and drying the sample; the cavity thus formed is filled with heavy oil or cane syrup, the container and oil being weighed before and after the operation.

H. J. G. HINES.

Simple speed controller, especially adapted to the moisture-equivalent centrifuge [for soils]. F. J. VEIHMAYER and C. V. GIVAN (*Soil Sci.*, 1928, 25, 455—460).—Control is established in the electrically-driven machine by a vibrating reed in a shunt circuit.

H. J. G. HINES.

Determination of the toxic substance in insecticides. IV. Volumetric determination of mercury in seed preservatives. J. BODNÁR, L. E. RÓTH, and I. TERGINA (*Z. anal. Chem.*, 1928, 74, 81—105. Cf. B., 1926, 997; 1927, 30, 856).—The destruction of the organic matter may be carried out without loss of mercury by heating the substance with sulphuric acid and nitric acid or, preferably, hydrogen peroxide in a conical flask fitted with a reflux air condenser 40 cm. long. The operation takes 30 min. with the peroxide and 1—2 hrs. with nitric acid. In either case the solution, if free from chlorides, is diluted and titrated directly with thiocyanate; if chlorides are present it is made just alkaline with sodium hydroxide and the mercury precipitated by addition of formaldehyde. After 30 min. the precipitate is collected on a close-textured paper, washed, and dissolved in nitric acid for titration; an addition of 0.6 mg. of mercury is made to allow for slight loss during destruction of the organic matter in the presence of chlorides.

A. R. POWELL.

Boll-weevil poisons. WALKER.—See VII. **Toxicities of arsenicals and fluorine compounds.** MARCOVITCH.—See XXIII.

PATENTS.

Drying or curing hay or like crops. H. WADE. From A. J. MASON (B.P. 292,211, 9.3.27).—The freshly-cut crop is formed into a matted layer of uniform thickness and density before being submitted to the drying process. Suitable apparatus is described.

A. G. POLLARD.

Fungicide. I. G. FARBENIND. A.-G. (B.P. 271,480, 18.5.27. Ger., 18.5.26).—The product contains a complex organic mercury compound soluble in alkalis, e.g., sodium mercuritolyxide, and a copper compound soluble in alkalis, e.g., a mixture of copper sulphate and sodium potassium tartrate, with or without the addition of other fungicides, wetting agents, or inert material.

L. A. COLES.

Germicides (B.P. 292,222).—See XX.

XVII.—SUGARS; STARCHES; GUMS.

Wood-sugar problem. I. Saccharification of the cellulose. P. LEONE and A. NOERA (*Annali Chim. Appl.*, 1928, 18, 205—239).—A critical summary is given of previous work on this subject, both in the laboratory and on an industrial scale. The experimental results given show that, when hydrochloric

acid of 36—37% concentration acts on cellulose, the greatest concentration of the resulting dextrose solution is 13.66%, the amount of acid employed being about seven times that of the sugar obtained. Addition of zinc chloride to the acid raises the attainable concentration of the sugar and lowers the consumption of acid. Thus with a solution containing 37% of hydrogen chloride and 20% of zinc chloride, a 34% sugar solution is obtainable with the value 3 for the ratio acid : sugar. These results are of interest since, with 40% hydrochloric acid alone, the sugar concentration never exceeds 30%, and if sawdust is treated with such acid counter-currentwise, the mean final concentration of the sugar is 28%.
T. H. POPE.

Boiling of white sugars. F. NOSEK (Z. Zuckerind. Czechoslov., 1928, 52, 441—447).—Insoluble particles present in the liquor from which white sugars are boiled (fibres, char dust, mineral matter, etc.) if sufficiently fine may serve as grain in the subsequent crystallisation, causing the sugar to be dull. Their presence is therefore to be avoided, though the greyish tinge of granulated sugar may also be due to occluded air bubbles. In forming grain by "seeding," the sugar dust used should be boiled from the best liquors, and care must be taken thoroughly to disperse it throughout the contents of the pan, otherwise conglomerates are formed. Dust obtained by grinding is less suitable than specially boiled very fine grain. Fine "pilé" should not be used. In boiling white sugars the formation of the grain and its growth can be followed to advantage under the microscope, and useful information is thus obtained as to the conditions requisite for the production of uniform crystals.
J. P. OGILVIE.

Use of permutite for the purification of [beet] sugar juices. I. MUTTI and C. REGINELLI (Zymologica, 1928, 3, 91—95).—The sodium and potassium present in a beet juice prepared in the laboratory may be almost completely removed by treatment with calcium permutite, which acts also as a decolorising, filtering material and retains considerable proportions of mucilaginous substances removable only with difficulty during the regenerative washing of the permutite; this difficulty may possibly be overcome by ignition of the permutite.
T. H. POPE.

Methods of decolorising sugar solutions by means of active carbons. V. SÁZAVSKÝ (Z. Zuckerind. Czechoslov., 1928, 52, 413—420).—In the suspension method a certain amount of the carbon is mixed with the juice during 15 min. at about 80° and pumped through filters of suitable type, whilst in layer filtration the carbon is previously deposited in a layer about 25 mm. thick and the liquor under decoloration pumped through this. In the latter method a better utilisation of the carbon is realised by working in two stages so that the liquors are passed first through almost exhausted carbon and subsequently through fresh material. Some factories use a combination of the suspension and layer methods. Which method is the most suitable depends mainly on the nature of the carbon and on the liquors under treatment. In general, the layer method in two stages is the most favoured for high-power carbons.
J. P. OGILVIE.

Determination of chlorine in sugar factory products. C. E. BUDLOVSKÝ (Z. Zuckerind. Czechoslov., 1928, 52, 421—430).—In the case of products soluble in water or dilute nitric acid, the acidified solution is treated with a slight excess of silver nitrate, clarified with ferric phosphate, and the precipitate centrifuged and dried without washing. It is then distilled with concentrated sulphuric acid in the presence of copper in a special apparatus, the hydrochloric acid condensed in water being titrated by Votoček's method. Insoluble products are distilled with concentrated sulphuric acid in the same apparatus, following the lines of the Vaubel-Scheuer method (B., 1906, 286), and the hydrochloric acid is determined as before. Results obtained by these two methods were consistently higher than those found by determining the chlorine in the ash of the product examined.
J. P. OGILVIE.

Molybdomanganometric determination of lactose. G. FONTÈS and L. THIVOLLE (Lait, 1927, 7, 547—555; Chem. Zentr., 1927, II, 1314).—The cuprous oxide precipitated by the action of lactose on Fehling's solution is dissolved in a solution of molybdic acid in phosphoric acid, and the resulting blue reduced solution is titrated with permanganate.
A. R. POWELL.

Lactose from whey. BELL and others.—See XIX.

XVIII.—FERMENTATION INDUSTRIES.

Hydrogen ions in brewing processes. IV. G. HAGUES (J. Inst. Brew., 1928, 34, 362—376; cf. B., 1927, 589).—There is no relationship between the duration of fermentation and the p_H value of a fermenting wort, and the hydrogen-ion concentration is the combined result of the total acidity and the buffer substances remaining in solution. In the first stages of fermentation, when yeast reproduction is active, the total acidity remains constant or falls slightly, and the simultaneous decrease of p_H value is due to a removal of buffer substances. In the latter stages acid substances are formed, and the decrease of the p_H value slows down with the concurrent increase in the buffer content of the wort. The manner in which the buffering of a wort alters during fermentation is not influenced by the initial p_H value of the wort between the values 4 to 6. The presence of gypsum in the mashing water has very little effect on the buffering of the wort. Increasing amounts of gypsum to a maximum content of 20 grains per gallon decrease the p_H value and increase the total acidity of the wort. With a further increase of gypsum the p_H value increases. The rate of yeast multiplication is optimum at p_H values 4 and 8—8.5, whilst a minimum occurs at p_H 6.2.
C. RANKEN.

Sulphites [in beer]. H. HERON (J. Inst. Brew., 1928, 34, 377—385).—The determination of sulphur dioxide in beer by the Monier-Williams method, where the sulphur dioxide distilled from the acidified beer is introduced into hydrogen peroxide, gives slightly lower results than those obtained with the method recommended by the Institute of Brewing, in which bromine water replaces the hydrogen peroxide. To a wort of gravity 1045° the malt contributes 3—8 pts. of sulphur dioxide per million, whilst hops, malt extract, and various

priming syrups also yield small amounts. Although a proportion of the sulphur dioxide is oxidised in the mash or lost during boiling, an appreciable amount becomes stabilised in the wort and appears in the beer at tracking owing to its combination with certain of the wort constituents to form hydroxysulphonic acids, which are non-volatile and resistant to oxidation. The loss during storage of sulphur dioxide, which is added to the finished beer in the form of sulphites, or as a constituent of the finings, seldom amounts to more than 25% of the quantity added, which loss occurs within the first two or three weeks. C. RANKEN.

Comparison of six different strains of the organism commonly concerned in large-scale production of butyl alcohol and acetone by the biological process. E. R. WEYER and L. F. RETTGER (*J. Bact.*, 1927, 14, 399—424).—The organism is diastatic, saccharolytic, and proteolytic; all the common, and most of the rare, sugars are sources of available carbon. Nitrogen in the form of proteins or peptones is necessary; ammonium salts and simple amino-acids are inadequate. The organism is not susceptible to the antiseptic action of butylresorcinol.

CHEMICAL ABSTRACTS.

Depilation of skins. ABT.—See XV. **Yeast fermentation.** JAMES and HUBER.—See XIX.

PATENTS.

Manufacture of yeast. J. Y. JOHNSON. From H. BÜCHER (B.P. 292,202, 19.2.27).—Yeast is seeded into a slightly acid, aerated wort containing adequate amounts of commercial molasses and a combination of yeast nutrients from which no acid is produced, or from which the acid produced is sufficient for the maintenance of the optimum acidity for yeast growth. The nutrients, which are soluble in water and do not precipitate insoluble compounds among the yeast, include carbamide, ammonium nitrate, and triammonium phosphate as the substances containing nitrogen, and, in addition, various combinations of magnesium sulphate, potassium sulphate, and tricalcium phosphate may be present. By adding fresh nutrients to the wort after complete or partial removal of the yeast and using the wort over again, as many as 12 successive crops of yeast may be grown before the repeated use of the wort inhibits reproduction. C. RANKEN.

Stabilisation of substances used as ferments or antiseptics. I. G. FARBENIND. A.-G., Asses. of R. BIELING (G.P. 446,130, 23.3.24).—The stabiliser used is dihydroxyacetone or esters or ethers derived from it.

A. R. POWELL.

Manufacture of yeast. R. L. CORBY and W. H. F. BÜHRIG, Asses. to FLEISCHMANN Co. (U.S.P. 1,673,735, 12.6.28. Appl., 16.8.24).—See B.P. 238,554; B., 1925, 897.

XIX.—FOODS.

Factors influencing the absorption in experimental baking [of dough]. J. MICKA and E. CHILD (*Cereal Chem.*, 1928, 5, 208—214).—The quantity of flour used in a dough has little effect on the absorption. There is a difference of about 3% between the absorption of a dough consisting of flour and water alone, and

that of a dough containing salt, sugar, lard, and yeast. In the former case the end-point is more difficult to determine. The influence of the ingredients was studied separately and collectively. Salt causes the dough to be slacker at first, becoming stiffer on being kept. In the case of sugar, the initial slackening effect continues. Lard decreases the absorption slightly; yeast increases it. High absorption is more detrimental to the quality of the bread than low, the best results being obtained with 57% absorption. In reporting percentage absorption values, retention power during fermentation and other factors which affect absorption should be considered. They should not be computed to a 13.5% or 15% moisture basis. W. J. BOYD.

Yeast fermentation in flour-water suspensions. T. R. JAMES and L. X. HUBER (*Cereal Chem.*, 1928, 5, 181—191).—A device is described by which the rate of fermentation of flour-water suspensions can be automatically recorded as indicated by the quantity of carbon dioxide evolved per min. Using this method the rate of fermentation was found to be greater for clear flours than for patent flours. The addition of salt increases fermentation even at a concentration of 20 g. of salt in 500 c.c. of water and 200 g. of flour. Oxidising agents have no beneficial effect. Ammonium chloride tends to inhibit fermentation at first, but towards the end of the fermentation period the growth of the yeast is greatly stimulated. Alkalinity decreases fermentation and acidity increases it, but the effect of acidity varies greatly with the type of flour.

W. J. BOYD.

Lipid phosphorus of wheat and its distribution. B. SULLIVAN and C. NEAR (*Cereal Chem.*, 1928, 5, 163—168).—The phosphorus content of the lipoids (*i.e.*, the alcohol-ether extract) decreases with decreasing refinement of the flours, whilst the percentage of lipoids and the total phosphorus in the flours increase. The highest grade of flour has the lowest total nitrogen content, but contains more nitrogen in its lipoid than do the lower grades. The lipoid material in the middlings, bran, and germ contain in proportion more neutral fats and other ether-soluble substances than the flours, and the nitrogen and phosphorus contents of the lipoids are correspondingly low. Extraction with ether alone is inadequate for the determination of phosphatides in wheat. W. J. BOYD.

Action of phosphatides in bread dough. E. B. WORKING (*Cereal Chem.*, 1928, 5, 223—234).—When dough is fermented the acid produced swells the gluten strands by increasing their water absorption and so reduces their tensile strength. At the same time the dough is rendered more ductile through lubrication of the strands by substances such as the phosphatides. These effects are necessary to produce the required loaf-volume and texture. The fermentation period can be omitted if acid and either egg-yolk or an oxidising agent be added. The oxidising agent liberates the phosphatide present in the flour. The addition of alcohol, glycerol, or saponin has a similar effect to that of egg-yolk. A baking formula is given which provides for the addition of lactic acid and sodium chlorate; this has been successfully used in a commercial bakery.

W. J. BOYD.

Effect on their bread-making properties of extracting flours with ether. A. H. JOHNSON (*Cereal Chem.*, 1928, 5, 169—180).—Bread produced from ether-extracted flour was found to be better in colour, texture, and volume than that produced from natural flour. The improvement in loaf-volume was more marked with low-grade than with patent flours. The extensibility of ether-extracted flours was slightly lower than that of the corresponding natural flours, probably due to slowness of hydration of the particles of the former. The quality and quantity of the gluten, the absorption, and the viscosity of the water-extracted acidulated suspension were unaltered. Diastatic activity and reducing sugar content were higher in the ether-extracted flours. The differences in behaviour of the extracted and unextracted flours as regards colour and texture of the bread were not removed by adding lard to both. Treatment of the natural flour with 70% alcohol by doughing, air-drying, and grinding injured its baking properties more than treatment with 96% alcohol, and treatment with water was least injurious. The last-named only reduced the extensibility and viscosity without affecting the bread-making properties.

W. J. BOYD.

“Rope” in bread. E. A. FISHER and P. HALTON (*Cereal Chem.*, 1928, 5, 192—208).—With decreasing p_H value of the bread, the development of rope is delayed. This value rises as the disease progresses owing to the production of ammonia. The buffering effect of the bread decreases till the disease is fully developed owing to the utilisation of the soluble phosphates, and thereafter it increases owing to the liberation of buffering material from proteins and other complex substances. Slight stickiness and faint odour are indicative of the acute stage of the disease. Of various substances tested, including acetic, lactic, and tartaric acids, and acid potassium and calcium phosphates, the most effective in preventing “rope” was the last-named, addition of 2 lb. of which per sack of flour was a complete preventative. Low temperature, ventilation, and cleanliness, however, are the best preventatives.

W. J. BOYD.

Obtaining crude lactose and other solids from sweet whey. R. W. BELL, P. N. PETER, and W. T. JOHNSON, JUN. (*J. Dairy Sci.*, 1928, 11, 163—174).—Sweet whey (<0.2% acidity) is neutralised to p_H 7.3 with 5% sodium hydroxide solution, heated at 60°, evaporated to 62% of solids at 50°, cooled to 25°, seeded, and kept for 42 hrs. at 4° or 18 hrs. at 0°, the lactose being recovered with a centrifuge. The liquor yields on evaporation a soluble powder containing casein and lactalbumin (1:2).

CHEMICAL ABSTRACTS.

Nature of the microscopic grains in cheese. O. LAXA (*Lait*, 1927, 7, 521—525; *Chem. Zentr.*, 1927, II, 1314).—The microscopic grains often found in cheese are shown to be composed of tyrosine or leucine. The presence of lactic acid in the early stages of the ripening of cheese increases considerably the solubility of tyrosine which then remains in solution, whilst leucine is precipitated in the anhydrous form during the final stages of the decomposition of the casein. In highly acid solutions leucine fails to precipitate, and in strongly alkaline media both tyrosine and leucine are soluble.

A. R. POWELL.

Analytical evaluation of technical casein. F. BAUM (*Chem.-Ztg.*, 1928, 52, 517—518).—The proportion of true casein substance obtained after deduction of the fatty acid, neutral fat, ash, and water content of a number of commercial caseins shows variations of 3—4%. The calculated nitrogen content of the true casein substance does not vary more than 0.6%, which is probably due to experimental error rather than to any difference in composition. So far no definite relationship has been revealed between the practical value of a casein and its composition.

F. R. ENNOS. ; }

Analyses of sprayed apples for lead and arsenic. A. HARTZELL and F. WILCOXON (*J. Econ. Entomol.*, 1928, 21, 125—130).—The fruit of apple trees sprayed five times with lead arsenate (5 lb. per 150 gals.) contained (mg. per kg.): lead 0.55—1.80, average 0.912; arsenic as trioxide 0.042—0.193, average 0.099. The results are compared with those of the previous year.

CHEMICAL ABSTRACTS.

Rapid determination of vaseline oil in coffee. (Miss) E. A. GAUTHIER (*Annali Chim. Appl.*, 1928, 18, 194—195).—The following modification of Marotta and Alessandrini's method (*B.*, 1928, 314) is recommended. 25 g. of the coffee beans are extracted by prolonged agitation with about 150 c.c. of ether in two or three portions, the liquid being filtered and the ether distilled off on a water-bath. The brown residue, containing vaseline oil, fats, colouring matters, and resins, is saponified by heating for 15—30 min. in a reflux apparatus with 20 c.c. of 5% alcoholic potassium hydroxide. The alcohol is removed by evaporation and the dry soap dissolved in methyl alcohol, with gentle heating on a water-bath if necessary. The turbid methyl alcohol solution is transferred to a separating funnel, the flask being rinsed first with methyl alcohol (about 50 c.c.) and then with light petroleum, which is used to wash down the funnel and glass rod used. The separator is then vigorously shaken and, when the liquids have separated completely, the methyl alcohol solution is allowed to flow into the original flask, while the petroleum is washed with several small quantities of water and afterwards filtered into a tared dish or, if the solvent is to be recovered, a tared wide-necked flask. After distillation of the petroleum, the extraction of the methyl alcohol is repeated to remove any residual traces of vaseline. The total residue is dried for 30 min. at 100° and weighed. With coffee containing 0.5% of vaseline oil, the errors found ranged from 0.9 to 3.64%.

T. H. POPE.

Organic mercury compounds for the control of insects in stored seeds. J. L. HORSEFALL (*J. Econ. Entomol.*, 1928, 21, 147—150).

Determination of nitrogen. PARISI.—See XVI.

PATENTS.

Preservation of meat. H. WATKINS-PITCHFORD (*B.P.* 291,818, 10.1.27).—A method of injecting preservative saline solution into animal carcasses is claimed.

F. R. ENNOS.

Recovery of organic substances from [meat-pickling] liquors. LABORATORIUM “TASCH” A.-G. [“TASCH” LABORATORY, LTD.] (*B.P.* 286,208, 10.11.27. *Ger.*, 28.2.27).—Liquors from the salting of meat are treated with organic sulphonic acids (*e.g.*, guaiacol-

sulphonic acid) or sulphuric acid, which precipitate the albumins etc. as double compounds. B. FULLMAN.

Conditioning and drying grain. T. ROBINSON & SON, LTD., and C. J. ROBINSON (B.P. 291,881, 11.3.27).

[Continuous] method and apparatus for preparing cream fondant and the like. R. HEAD, C. CLAY, BAKER, PERKINS, LTD., and ANC. ETABL. A. SAVY, JEANJEAN & CIE. SOC. ANON. (B.P. 292,237, 15.3.27).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Pharmaceutical preparations of valerian. E. H. MADSEN (Dansk Tidsskr. Farm., 1928, 2, 177—183).—An examination of concentrated valerian preparations intended to give on dilution a product similar to the ordinary infusion of valerian. A suitable preparation can be made by percolation of the shredded root with 10% alcohol, any precipitate forming being collected on a filter after settling. Such an extract contains about 20.6% of the essential oil and is stable on keeping. Other extracts made according to the method of the Dutch Pharmacopœia (extraction of the macerated root once with dilute alcohol and then twice with water, the three extracts being united and evaporated to a syrup) and by a method in which the oil was first removed by distillation in steam and the distillate afterwards mixed with the extract of the residue, both yielded products containing about 50% of the essential oil originally present in the root employed.

H. F. HARWOOD.

Preservative action of "nipagin" [methyl *p*-hydroxybenzoate] and its homologues on pharmaceutical preparations. E. BÖHM and H. JEGLINSKI (Pharm. Ztg., 1928, 73, 480—481).—Materials which are easily susceptible to decomposition by micro-organisms, such as malt extracts, syrups, and some aqueous solutions, were treated with various preservatives, and kept for several months under conditions favourable for the development of bacteria. Nipagin and its methyl and ethyl homologues gave satisfactory results in all cases.

S. I. LEVY.

Simplified method for determination of silver [in medicinals]. G. KOGAN (Pharm. Zentr., 1928, 69, 228—229).—A weighed quantity of the material in a flask is treated on a water-bath with excess of nitric acid (*d* 1.2), nitrous acid is destroyed by means of permanganate, and the solution is diluted and titrated in the usual way with ammonium thiocyanate solution.

S. I. LEVY.

Examination of clove oil. H. W. VAN URK (Pharm. Weekblad, 1928, 65, 345—349).—The best test is the separation of the sodium eugenol compound by shaking with sodium hydroxide solution, but the quantity of the latter specified in the Dutch Pharmacopœia is too great; the best results are obtained with one tenth of the quantity. A modification of the phloroglucinol test is described.

S. I. LEVY.

PATENTS.

Manufacture of substituted glycol monoethers. CHEM. FABR. AUF ACTIEN (FORM. E. SCHERING), Assees. of W. SCHOELLER and H. SCHOTTE (G.P. 446,324, 26.3.24).—Unsaturated alcohols are treated with mercuric salts (acetate) in presence of an alcohol, and the products,

after conversion into halogen compounds, react with amines to give aminoglycol monoalkyl ethers. The process is particularly useful for manufacture of γ -amino- $\alpha\beta$ -glycol monoethers. Thus, allyl alcohol with mercuric acetate and methyl alcohol gives a mercuriacetate, which is converted successively into the mercuribromide, γ -iodo- β -methoxypropyl alcohol, b.p. 65—66°/0.4 mm., and γ -amino- β -methoxypropyl alcohol, b.p. 104—105°/10 mm. (phenylcarbamide described), or its *N*-methyl (b.p. 75°/12 mm.) and *N*-diethyl (b.p. 62—63°/1.2 mm.) derivatives; the piperidino-compound, b.p. 81—82°/0.3 mm., and quaternary trimethyl- γ -hydroxy- β -methoxyethylammonium iodide, m.p. 108—109°, are also prepared. From allyl alcohol, ethyl alcohol, and mercuric acetate the corresponding β -ethoxy-compounds are obtained: γ -iodo- β -ethoxypropyl alcohol, b.p. 78—80°/1.2 mm., gives with diethylamine γ -diethylamino- β -ethoxypropyl alcohol, b.p. 100—101°/12 mm., and with methylamine the γ -methylamino-compound, b.p. 89—91°/13 mm. (picrate, m.p. 92—93°). Cinnamyl alcohol yields a methoxymercuribromide, m.p. 110—111°, and an ethoxymercurichloride, m.p. 86°. From the former are obtained γ -iodo- β -methoxy- γ -phenylpropyl alcohol (not distillable even in high vacuum), which is converted by diethylamine into γ -diethylamino- β -methoxy- γ -phenylpropyl alcohol, which partly distils at 123—125° in high vacuum. *iso*Crotonyl alcohol behaves similarly. The products find therapeutic application. C. HOLLINS.

Manufacture of a derivative of hexylresorcinol. F. HOFFMANN-LA ROCHE & CO. A.-G. (Swiss P. 117,236, 23.7.25).—4-Hexylresorcinol forms with hexamethylenetetramine a compound, m.p. 127—128°, having a pleasanter taste and less irritating effect than hexylresorcinol, and being less sensitive to light and air.

C. HOLLINS.

Manufacture of *p*-carboxybenzenesulphondichloroamide. M. CLAASS (Swiss P. 118,115, 29.8.25).—*p*-Carboxybenzenesulphondichloroamide, m.p. 203°, obtained by treating *p*-carboxybenzenesulphonamide with 0.5*N*-sodium hypochlorite and adding dilute sulphuric acid until acid to Congo paper, is a powerful antiseptic and germicide.

C. HOLLINS.

Preparation of easily soluble derivatives of *m*-aminobenzoic esters. G. B. ELLIS. From CHEM. WORKS FORMERLY SANDOZ (B.P. 291,850, 4.3.27).—Readily soluble alkylsulphonates, which are unchanged by prolonged heating in aqueous solution at 100°, and are non-irritant local anaesthetics, are obtained by interaction of alkyl *m*-aminobenzoates with alkylsulphonic acids in absolute alcohol. Ethyl *m*-aminobenzoate gives a methanesulphonate, m.p. 149—150°, and an ethanesulphonate, m.p. 149—149.5°; *n*-propyl *m*-nitrobenzoate, b.p. 175—176°/17 mm., is reduced to the amino-ester, b.p. 176—177°/16 mm., which yields a methanesulphonate, m.p. 162—163°, and an ethanesulphonate, m.p. 150—151°; isobutyl *m*-nitrobenzoate, b.p. 180—181°/16 mm., is reduced to the amino-ester, b.p. 181—182°/17 mm., which gives a methanesulphonate, b.p. 178—179°, and an ethanesulphonate, m.p. 138—139°.

C. HOLLINS.

Manufacture of camphor solutions. I. G. FARBEININD. A.-G., Assees. of M. BOCKMÜHL and R. SCHWABE (G.P. 446,290, 22.4.25).—Ethers or esters of polyhydric

alcohols are used as solvents for camphor intended for subcutaneous or intramuscular injection. Examples are glycol monoethyl ether, "diethylin," glycerol mono- and di-acetates, these being miscible in all proportions with water or serum. C. HOLLINS.

Manufacture of ω -chloroacetylpyrocatechol [3:4-dihydroxyphenacyl chloride]. E. OTT (G.P. 445,524, 22.4.26).—Pyrocatechol, heated at 100° with chloroacetyl chloride (free from phosphorus compounds) in a solvent (benzene), gives a 40% yield of the *monochloroacetate*, m.p. 81°, which is converted by pure phosphoryl chloride, at 100° or in boiling benzene, into 3:4-dihydroxyphenacyl chloride, an intermediate for the synthesis of adrenaline (cf. Ott, A., 1926, 722). In the first stage of the reaction the *bischloroacetate* is obtained as by-product. C. HOLLINS.

Manufacture of pyridine-2:3-dicarboxylic acid. GES. F. CHEM. IND. IN BASEL (Swiss P. 118,966, 11.8.25).—Quinolines containing in the Bz-nucleus at least one substituent (hydroxyl, amino-, halogen) favouring disruption by oxidation are oxidised with nitric acid or nitric and hydrochloric acids. Examples are: 8-hydroxyquinoline and its 5-chloro-derivative oxidised with 60% nitric acid at 70–90°, or by 60% nitric acid and concentrated hydrochloric acid at 70°, to give pyridine-2:3-dicarboxylic [quinolinic] acid. C. HOLLINS.

Manufacture of derivatives of tertiary aromatic bases containing sulphur and phosphorus. L. CASSELLA & Co. G.M.B.H., Assees. of L. BENDA and W. SCHMIDT (G.P. 445,568, 3.1.25. Cf. B.P. 214,836; B., 1924, 615).—Tertiary aromatic amines are treated with sulphur and phosphorus trichloride, and afterwards with alkali, to give crystalline products useful as antirachitics and stimulants of metabolism. Dimethyl-aniline is heated with sulphur at 100° until the latter is dissolved, cooled to 50°, and warmed with phosphorus trichloride. The thick oily liquid is poured upon aqueous sodium hydroxide and ice, and aniline excess is removed by steam-distillation. Alcohol extracts from the evaporated residue "*p-dimethylaminophenylthiophosphinic acid*" (sodium salt + Aq, m.p. 97–98°). *N*-Ethylcarbazole, heated with sulphur and phosphorus trichloride at 120–130°, gives an analogous product (sodium and calcium salts described). C. HOLLINS.

Manufacture of pure hexamethylenetetramine hydrochloride. PELLURIN-GES.M.B.H. (G.P. 445,646, 7.2.23).—Crude or purified hexamethylenetetramine, e.g., the base obtained by warming its crystallised hydrate at 30°, is dissolved in a non-aqueous solvent (high-strength alcohol, methyl alcohol, acetone, ethyl acetate, or chloroform) and dry hydrogen chloride is led in until no further precipitation occurs. The hydrochloride, $C_6H_{12}N_4 \cdot HCl$, is therapeutically more valuable than the free base. C. HOLLINS.

Manufacture of compounds of 5:5-disubstituted barbituric acids with antipyrin. F. HOFFMANN-LA ROCHE & Co. A.-G. (Swiss P., 120,444–6, 9.5.25. Addns. to Swiss P. 116,465; B., 1927, 829).—The prior process is applied to the preparation of antipyretic and analgesic compounds from equimolecular proportions of antipyrin and (A) 5-phenyl-5-ethyl-, (B) 5:5-

diethyl-, (C) 5:5-diallyl-barbituric acids. The products have m.p. 112°, 118°, and 97°, respectively.

C. HOLLINS.
Manufacture of derivatives of 5:5-disubstituted barbituric acids. J. D. RIEDEL A.-G. (G.P. 445,670, 19.8.25).—Additive compounds, which crystallise readily from water and slowly dissociate at 15°, are obtained by adding a halogenated hydrocarbon to a 5:5-disubstituted barbituric acid, especially one containing an unsaturated substituent (allyl), melted under water. Additive compounds of chloroform with 5- β -bromoallyl- and 5-allyl-5-*sec*-butyl-barbituric acids, and of carbon tetrachloride with 5- β -bromoallyl-5-*sec*-butylbarbituric acid, are described; the last-mentioned has m.p. 125°. *iso*Propyl bromide may also be used. C. HOLLINS.

Preparation of bismuth nucleinate. C. LEVADITI (F.P. 577,407, 19.2.24).—Sodium nucleinate dissolved in a solution of sodium chloride is heated for 6 hrs. at 37° with a solution of sodium potassium bismuthyl tartrate containing 30% Bi. Next day the crude bismuth nucleinate is precipitated by the addition of absolute alcohol. The precipitate is redissolved in sodium chloride solution and the liquid is subjected to dialysis through a collodion membrane suspended in a solution of sodium chloride. When no further bismuth passes through the membrane the colloidal solution is evaporated at a low temperature to a dry residue containing 32% Bi, 5% P, and 10% N. The compound may also be made directly from the solution obtained by extracting minced thyroid glands with sodium chloride solution. A. R. POWELL.

Production of nitropyridinearsinic acids. A. BINZ and C. RÄTH (B.P. 275,590, 18.7.27. Ger., 5.8.26).—Hydroxypyridinearsinic acids, such as the 2:5-compound described in B.P. 250,287 (B., 1926, 512), are nitrated *ortho* to the hydroxyl group by mixed acid at 100°. C. HOLLINS.

Tellurium compounds for germicidal and therapeutic purposes. G. T. MORGAN and H. BURGESS (B.P. 292,222, 14.3.27).—Claim is made for the preparation of the compounds described in A., 1928, 435. All the products find application as germicidal or therapeutic agents. C. HOLLINS.

Preparation of double compounds of alkaloids or amines with antimony or arsenic tri-iodide. E. VIEL (F.P. 577,371, 14.4.23).—Acid solutions of alkaloids or of amines are mixed with acid solutions of arsenic or antimony tri-iodide containing an alkali or alkaline-earth iodide whereby amorphous precipitates are obtained which can be crystallised from hot dilute hydrochloric acid (cf. Caille and Viel, A., 1925, i, 68). A. R. POWELL.

Manufacture of quinine solutions. F. B. DEHN. From CHEM.-PHARM. A.-G. BAD HOMBURG (B.P. 291,565, 4.4.27. Addn. to B.P. 266,209; B., 1927, 317).—The solubilising effect of terpenes on quinine base in olive and similar oils is enhanced by the addition of oil-soluble or fat-soluble alcohols, such as octan- β -ol, *n*-decyl alcohol, benzyl alcohol, β -phenylethyl alcohol, cinnamyl alcohol. Terpenes such as cineole, which have small solubilising effect but are therapeutically valuable, can be used. An example is dry quinine base (6 pts.),

benzyl alcohol (15 pts.), and cineole (15 pts.), made up to 100 pts. with olive oil. C. HOLLINS.

Preparation of benziminazolone-[5]-jarsinic acids. W. KOLLE, K. STREITWOLF, and A. FEHRLE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,674,368, 19.6.28. Appl., 19.7.26. Ger., 31.7.25).—See B.P. 256,243; B., 1927, 670.

Preparation of nitropyridinearsinic acids. A. BINZ and C. RÄTH (U.S.P. 1,675,402, 3.7.28. Appl., 28.7.27. Ger., 5.8.26).—See B.P. 275,590; preceding.

Manufacture of complex antimony compounds. H. HAHL, Assr. to WINTHROP CHEM. CO., INC. (U.S.P. 1,674,362, 19.6.28. Appl., 11.3.26. Ger., 12.5.25).—See B.P. 262,301; B., 1927, 125.

Production of carbohydrate-acridine compounds and solutions thereof. C. SCHNORF and F. HEFTI (U.S.P. 1,674,923, 26.6.28. Appl., 23.10.24. Ger., 12.4.24).—See B.P. 247,250; B., 1926, 341.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photochemistry of silver halides. I. Gravimetric determination of excess silver in photographic layers. H. H. SCHMIDT and F. PRETSCHNER (*Z. wiss. Phot.*, 1928, 25, 293—307).—The gelatin of the thoroughly washed silver halide emulsion is destroyed by boiling with 1 : 30 nitric acid, a concentration which does not decompose the halides, prevents their reduction by gelatin and its decomposition products, and, in the case of iodide, causes no oxidation to iodate. The filtrate containing the excess silver and dissolved silver halides is treated with a known excess of potassium chloride, and the silver obtained by the reduction of the precipitated silver chloride with alkaline hydrazine sulphate is weighed (*a*). By treatment of the solution containing the excess chloride with a known excess of silver nitrate a weight (*b*) of silver is similarly obtained. The difference between the silver equivalent of the potassium chloride and the weight (*b*) gives the excess silver present in the solution, whilst the amount of dissolved silver halides is obtained by subtracting the excess silver from the value (*a*). The halides of the emulsion remaining after the treatment with nitric acid are shown to contain no excess silver or halogen by analysis, according to the above methods, of the liquid obtained from their purification by reprecipitation. Although all the emulsions studied were prepared in presence of excess halide, excess silver was found in each case, the amount being greatest for chloride and least for iodide, but bearing no relation to the method of preparation or the sensitivity of the emulsions. The work of Eggert, Noddack, and Leszynski, on the application of the photochemical equivalence law to silver halide layers, is criticised, since no account was taken of the excess silver present in the emulsions. J. W. GLASSETT.

Photochemistry of silver halides. II. Decomposition of photographic layers with neutral, acid, and alkaline hydrogen peroxide, and with ammoniacal ammonium sulphide. H. H. SCHMIDT and F. PRETSCHNER (*Z. wiss. Phot.*, 1927, 25, 354—362; compare preceding abstract).—Acid hydrogen peroxide

increases the ease of decomposition by decomposing the gelatin, so that the concentration of the nitric acid can be reduced one hundredfold without decreasing the speed of decomposition. The solution can easily be filtered to a clear filtrate to give a residue of silver halide. The halogen ion in the filtrate can only be determined by means of silver nitrate if the hydrogen peroxide be first removed by long boiling of the ammoniacal solution. Neutral hydrogen peroxide completely decomposes the gelatin on boiling, and the silver halide can be collected on a filter. Some metallic silver is formed in the layer by reduction, but the equivalent quantity of halide in the filtrate can be determined as before. When an alkaline peroxide solution is used the gelatin is very rapidly decomposed, but the silver halide is reduced so that the residue contains metallic silver and the filtrate the halogen ion. The reduction is complete in the case of the chloride and bromide, but only partial with the iodide unless the process be repeated several times. Ammoniacal ammonium sulphide easily and completely transforms the silver into sulphide, and by addition of ammonia during washing of the precipitate, peptisation can be prevented and filtration thus facilitated. S. J. GREGG.

PATENTS.

Production of light-sensitive films. U. DIEM-BERNET (B.P. 271,507, 20.5.27. Switz., 21.5.26).—In a method for the preparation of transparent light-sensitive films from viscose, the silver halides are introduced during the manufacture of the film, by impregnating the film mass with soluble halide or silver salt, followed after superficial drying, if desired, by an adequate amount of silver salt or halide such that no opalescence is obtained upon complete drying. Such films are very suitable for reflex copying, and suffer little distortion during development and fixing. J. W. GLASSETT.

Manufacture of polychromatic screens for colour photography. R. C. M. DE BERCEGOL (U.S.P. 1,673,349, 12.6.28. Appl., 2.12.22. Fr., 5.12.21).—The film or plate is coated with a colouring agent, and, after excess has been removed, a layer of waxy insulating material is applied. Parallel lines are then cut through both waxy and colour layers, and the grooves so formed are filled with a second colouring agent. F. G. CLARKE.

Photographic sensitisers. KODAK, LTD. From EASTMAN KODAK Co. (B.P. 292,374, 28.7.27).—Alkylidides or similar quaternary salts of 4-methyl- or 4 : 6-dimethyl-quinoline are condensed with iodoform (in place of formaldehyde) and alkali. In addition to the known sensitising dyes, there are formed under these conditions less soluble by-products which sensitise effectively from 780 to 900 μ , *i.e.*, much further into the infra-red. C. HOLLINS.

Photographic developing processes more especially for use with printing surfaces. Soc. DE PHOTOCHIMIE ELKA (B.P. 265,630, 7.2.27. Fr., 8.2.26).—Gelatin reliefs suitable for all types of printing processes may be obtained from ordinary gelatino-bromide emulsions by partial development of the image in a hardening developer, such as pyrogallol, with a small amount of sulphite, followed by complete development, either in the darkroom or in daylight, with

an active non-hardening agent such as metol. Finally, after normal fixation, the negative is developed in hot water. J. W. GLASSETT.

XXII.—EXPLOSIVES; MATCHES.

Pentaerythritol tetranitrate as a military explosive. A. STETTbacher (Z. angew. Chem., 1928, 41, 716—718).—Pentaerythritol tetranitrate fulfils many of the requirements of a modern explosive, but because of its high m.p. (140—141°) must be used in a compressed state. When compressed it is much less sensitive, and is detonated with difficulty. This disadvantage is overcome by the use of a paste containing 80% of tetranitrate and 20% of nitroglycerol. Pentaerythritol tetranitrate may be prepared by adding 100 g. of the powdered alcohol to 430 c.c. of fuming nitric acid (*d* 1.52) and precipitating the ester by the careful addition of 400 c.c. of concentrated sulphuric acid. The temperature should not exceed 25°. The separated crystals are agitated with 1 litre of ice-water, filtered, and washed. Yield, 225 g. = 97%. The last traces of acid are removed by dissolving 100 g. of the crude ester in 400 c.c. of acetone and boiling under reflux with 3—4 g. of ammonium carbonate. The filtered solution is poured into 800 c.c. of water. The precipitated ester is collected, washed, and dried. Yield about 96% of the crude ester. J. S. CARTER.

PATENTS.

Impregnation of absorptive material for use in explosives. Blasting explosive. B. I. STOOPS, Assr. to HERCULES POWDER CO. (U.S.P. 1,671,792—3, 29.5.28. Appl., [A] 2.4.27 and [B] 4.11.27).—(A) Ammonium nitrate or perchlorate is dissolved in the liquid obtained by heating a hydrated salt, such as magnesium or calcium nitrate or sodium sulphate, until it melts in its water of crystallisation, and a carbonaceous absorptive material, *e.g.*, sawdust, is incorporated in the solution. (B) A blasting explosive comprises a mixture of 5—20% (10%) of nitroglycerin, 20—80% (47%) of ammonium nitrate, 4—30% (15%) of magnesium nitrate hexahydrate, 0—20% (12%) of sodium or potassium nitrate, and 5—30% (16%) of balsa wood sawdust. A. R. POWELL.

Separation of 2:4-dinitrophenol and picric acid (F.P. 575,302).—See III.

XXIII.—SANITATION; WATER PURIFICATION.

Relative toxicities of arsenicals and fluorine compounds to various organisms. S. MARCOVIRCH (J. Econ. Entomol., 1928, 21, 108—114).—Relative and quantitative toxicities of a number of arsenic and fluorine compounds for mosquitos, grasshoppers, worms, etc. are recorded. Toxic doses for man are: sodium fluoride 30 g., sodium fluosilicate 7.2 g., potassium arsenite 0.84 g.

CHEMICAL ABSTRACTS.

Electro-osmotic purification of water. A. H. W. ATEN (Chem. Weekblad, 1928, 25, 211—219; cf. Behrman; B., 1927, 958).—The apparatus manufactured by the Siemens Electro-Osmosis Co. of Berlin for the purification of water is described, and results are given of tests carried out. A current is caused to pass through a series of cells, each of which is divided into three parts

by means of two diaphragms. Electrodes are placed in the outer compartments. Electrolytes are removed from the water in the middle compartments as a result of differences in the resistances of the two diaphragms to the passage of cations and anions respectively. The theory of the process is discussed, and the results arrived at are interpreted to show that it is possible completely to free the water from dissolved electrolytes. Organic matter is also reduced, and the water rendered sterile.

S. I. LEVY.

PATENTS.

Activated sludge process of purifying sewage and other impure liquid. ACTIVATED SLUDGE, LTD., and J. A. COOMBS (B.P. 285,944, 27.10.26).—Sewage is subjected to preliminary agitation and aeration which breaks up grosser suspended solids, stops septic action, facilitates removal of grease, and, generally, renders sewage more amenable to subsequent treatment. After separation of solid matters and removal of grease in specially designed tanks the further treatment of the sewage is divided into two stages: (a) removal or oxidation of carbonaceous matter, (b) oxidation of ammoniacal matter to nitrite and nitrate, with the production and utilisation, in each case, of a special sludge for the work and consequent reduction in the total time involved.

C. JEPSON.

Manufacture of [disinfectants from] new complex bodies containing mercury. W. CARMAEL. From I. G. FARBENIND. A.-G. (B.P. 292,245, 16.3.27).—A soluble mercury salt is incorporated with a soluble arylaminesulphonate in presence of water without heating; mercuration is thus prevented (cf. B.P. 8153 of 1914; B., 1915, 852), but complex salts are formed from which mercury is not removed by sodium hydroxide or sulphide.

C. HOLLINS.

Removal of salts from sea water or salt water. R. J. WOOD (B.P. 285,954, 19.11.26).—Sea water or salt water is rendered safe for use in steam generators by treatment with appropriate reagents and the application of heat in a plant consisting of a reaction vessel, fitted with a steam jacket and agitators, and having valves so connected that they may be set and adjusted simultaneously so as to vary the proportion of salt water to reagent, followed by a decanter vessel in which is a filtering arrangement.

C. JEPSON.

Filtration of alkaline waters. J. GORDON. From R. E. HALL (B.P. 291,970, 28.6.27).—The use of various silicates (*e.g.*, of iron, magnesium, or calcium) in preference to sand for filters dealing with hot boiler waters which are alkaline owing to the pretreatment they have received for softening purposes is recommended because of their lower solubility under such circumstances. For the filter bed a granulated smelter slag obtained from copper smelting may be used. As the silicates suggested have a fairly high sp. gr. the time taken to wash the filter by reversal of flow is not excessive.

C. JEPSON.

Refuse destructors. G. M. CLARK. From STETTNER CHAMOTTE-FABR. A.-G. VORM. DIDIER (B.P. 292,630, 22.1.27).

Base-exchange bodies (B.P. 279,466).—See VII. **Stabilisation of antiseptics** (G.P. 446,130).—See XVIII.