# BRITISH CHEMICAL ABSTRACTS

# B.—APPLIED CHEMISTRY

OCTOBER 1, 1926.

# I.—GENERAL; PLANT; MACHINERY.

Measurement of currents of air and gases, with especial reference to dynamic principles. O. MATTNER (Chem.-Ztg., 1926, 50, 533-534).—The method of measuring a flow of gas by determining the pressure drop at a constriction in the pipe is quite general, and its application depends only on a suitable choice of the type of orifice and method of pressure measurement. The static gas pressure can be determined by Ser's or Nipher's apparatus (perforated plates parallel to the gas stream). The total pressure is indicated by a Pitot tube; and the dynamic pressure, the difference between the two, can be ascertained by connecting the two devices to a differential manometer. This is the principle of the "pneumometers" of Krell, Prandtl, and Brabbée. The average velocity of a current of gas is given by the formula  $v = \sqrt{2g Pd/\gamma k}$ , where Pd is the pressure difference on the two sides of a perforated plate or similar device,  $\gamma$  is the specific gravity of the gas, and k a constant depending on the apparatus. Types of "pneumometers" are described. C. IRWIN.

Studies in adhesion. I. W. HARDY and M. NOTTAGE (Proc. Roy. Soc., 1926, A, 112, 62-75).-A cylinder standing in a pool of lubricant on a plate can be lifted by any force great enough to overcome the slight resistance offered by the surface tension of the lubricant, provided sufficient time is given. Adhesion is the normal pull required to detach the cylinder instantaneously from the plate. To obtain comparable values, cylinder, plate, and lubricant must be in a mechanically corresponding relation; this occurs when the thickness of the layer of lubricant is such that the Leslie pressure carries the load, and to the force needed to break the cylinder away from this equilibrium position the name A value" is given. For all loads employed, the layer of lubricant corresponding to this position was found to be of sensible thickness. The latent period which elapses before the steady state is reached is given for a number of lubricants, and is assumed to be due either to the orientation of the lubricant molecules in the attraction fields of the solids, or to the cylinder rising or falling in the pool of lubricant. The A values for 24 lubricants using different solids and different loads are tabulated, and the effects of pressure, of the nature of the solid, of the molecular weight of the lubricant, and of temperature, are discussed. The coefficient  $\alpha$  (= A/load) decreases as the load increases, up to the highest load tested (259.6 g.), and the A value is a linear function of the molecular weight of the lubricant, for any one chemical series, and of the temperature. The effect of a change in the nature of the solid is to shift the

curve for A and molecular weight parallel to itself, and the curve for two different solids is half-way between the curves for each solid by itself. It is suggested that the influence of the nature of the solid wall should be taken into account in certain of the standard methods of viscosity measurement.

L. L. BIRCUMSHAW.

Validity of flicker photometer measurements in heterochromatic photometry. A. H. Taylor (J. Opt. Soc. Amer., 1926, 13, 193—204).

See also A., Sept., 898, Adsorptive powers of charcoal (Ogawa); Adsorption by ash-free adsorbent charcoals. Purification of adsorbent charcoals (MILLER). 899, Adsorption of dissolved substances (Charriou).

Percolator. RATTRAY.—See XX.

## PATENTS.

Method of grinding or crushing. E. Barthelmess (E.P. 251,665, 3.5.26. Conv., 2.5.25).—The material is subjected to centrifugal impact by means of a fluid entering the grinding chamber tangentially at a high speed. From the grinding chamber the mixture is led to a separator and only the fluid returns to the pump or fan, where it receives the energy to effect the grinding and then re-enters the grinding chamber.

B. M. VENABLES.

Tube mills for grinding or crushing. Wick-ing'sche Portland-Cement & Wasserkalkwerke, and A. Andreas (E.P. 254,909, 14.9.25. Addn. to 241,174).— Modifications are made in the screening device of a compound ball mill such as is described in the original patent (cf. B., 1926, 344) to improve the transfer of the sifted material to the secondary grinding compartment. To this end the screens may be inclined to the axis of the mill, and may be tangential rather than radial. Curved guides may also be used. Means are also provided for easily changing the screens.

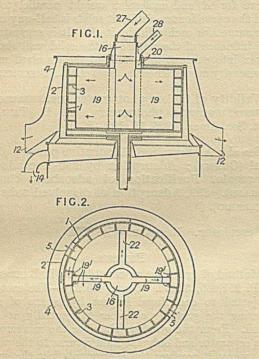
B. M. VENABLES.

Gyratory cone crusher. E. B. Symons, Assr. to Symons Bros. Co. (U.S.P. 1,592,313, 13.7.26. Appl., 4.6.25).—In a crusher in which a gyrating cone is surrounded by an inverted bowl or the stationary part of the crusher, the latter is mounted resiliently in the frame in such a way that it is always pressed downwards towards the gyrating cone.

B. M. Venables.

Centrifugal machines. V. Redlich (E.P. 252,339, 23.4.26. Conv., 19.5.25).\*—A centrifugal machine with

continuous discharge of both solid and liquid separated products comprises an outer basket, 1, 2, and inner feed devices, 19, 22, rotating at the same high speed, also an inner cellular basket, 3, rotating very slightly faster than the above-mentioned rotating parts. The outer basket comprises an inner perforated wall, 1, which forms the filter surface, and an outer non-perforated wall, 2, which serves to collect the clear liquor.



At intervals slots, 5, are formed through both walls, through which the dried solid matter is discharged, being collected by the fixed outer wall, 4, of the apparatus. The feed of the original material is through passages, 19, to cells that have just left the discharge ports, 5. Passages, 22, serve for the admission of wash liquor at a later stage of the draining. The annular space of the outer basket is divided into segments by partitions, so that the original filtrate and washings may be collected separately. When it is desired to collect the solid matter in pieces of a definite size and shape, e.g., sugar cubes, the cells are made to suit the desired form, and the collecting wall, 4, is made up of a series of guides designed to collect the solid and reduce its speed without sho k.

B. M. Venables.

Concentrating solutions, even those liable to incrust to a great extent and corrosive. P. Bringhenti (E.P. 254,944, 7.11.25).—The apparatus comprises a preheating vessel where an inert liquid, e.g., melted paraffin, is heated by any suitable external source, a heating vessel in which the solution to be evaporated is heated by contact with sprays or streams of the inert liquid, the latter afterwards rising to the top, and a separator in which the inert liquid is freed from any entrained solution, the latter returning to the heating tank and the former to the preheater. The heated

solution falls or is pumped to a second vessel, where it is permitted to evaporate, the residual solution being returned to the heating vessel and the vapour being condensed preferably in the preheater of another similar effect operating at a lower pressure. B. M. Venables.

Filter-medium support. S. E. WOODWORTH, Assr. to Hamilton, Beauchamp, & Woodworth (U.S.P. 1,593,051, 20.7.26. Appl., 30.1.24).—Spaced strips, set at an angle to the longitudinal axis of the filter drum, are placed between the drum and the filter medium surrounding it. H. Holmes.

Removal of vapours or gases from moist gaseous mixtures. Badische Anilin- & Soda-Fabr. (F.P. 604,207, 3.7.25. Conv., 5.9.24).—An absorbent, such as silica gel and, if necessary, activated charcoal, is used in several vessels, through which the gas passes in succession.

W. G. Carey.

Process of drying materials. Fuller Fuel Co., Assees. of M. D. Jones (E.P. 231,151, 3.3.25. Conv., 18.3.24).—See U.S.P. 1,568,738; B., 1926, 349.

Refrigerating systems. Stitt Refrigeration Co., Assees. of R. R. Stitt (E.P. 246,814, 6.1.26. Conv., 2.2.25).—See U.S.P. 1,570,080; B., 1926, 224.

Purifying, clarifying, decolorising and deodorising liquids or gases. H. W. A. Branco (E.P. 256,316, 4.5.25).—See F.P. 598,826; B., 1926, 304.

Refrigerating machine. Brit. Thomson-Houston Co., Ltd., Assees. of C. Orr (E.P. 244,768, 12.12.25. Conv., 16.12.24).

Self-acting apparatus for mechanical separation of liquid mixtures. H. HITCHON (E.P. 256,012, 19.6.25).

Protection of metallic surfaces against incrustation. A. L. Mond. From W. Thalhofer (E.P. 256,481, 27.1.26).

# II.—FUEL; GAS; DESTRUCTIVE DISTILLATION; MINERAL OILS.

Reduction of carbon monoxide. H. Tropsch, A. Schellenberg, and A. von Philippovich (Abhandl. Kennt. Kohle, 1925, 7, 63-67; Chem. Zentr., 1926, II, 847-848).—The formation of methane from a mixture of carbon monoxide (1 vol.) and hydrogen (3 vols.) in contact with nickel, which normally occurs quantitatively at 240-250°, is greatly diminished by adding ammonia to the gas mixture. Neither formaldehyde nor hexamethylenetetramine was formed, and the final mixture contained 13% of methane, 15% of carbon monoxide, 64% of hydrogen, and 8% of nitrogen. With a nickel catalyst which had been treated with alkali, the same gas mixture yielded, at 400° and atmospheric pressure, 53% of methane and 1.1% of unsaturated hydrocarbons, the only liquid reaction product being water. The same reaction occurred with a nickel

catalyst which had not been treated with alkali, except that no unsaturated hydrocarbons were formed. A mixture of equal volumes of carbon monoxide and hydrogen, in contact with an alkaline nickel catalyst at 400°. deposited much carbon. With a feebly alkaline catalyst the final gas contained 27% of carbon dioxide, 2% of carbon monoxide, 26% of hydrogen, and 45% of methane, whilst with a more strongly alkaline catalyst it contained 11% of carbon dioxide, 29% of carbon monoxide, 50% of hydrogen, and 9% of methane. With both alkaline and neutral catalysts the methane contained 1.5% of higher homologues, reckoned as ethane. A mixture of equal volumes of carbon dioxide and hydrogen, in contact with neutral nickel at 400°, gave a final gas containing 10% of carbon dioxide, 42% of hydrogen, and 48% of methane, whilst with alkaline nickel it gave 7% of carbon dioxide, 70% of hydrogen, and 23% of methane. In no case could methyl alcohol be detected.

W. T. K. BRAUNHOLTZ.

Reduction of carbon monoxide by hydrogen in contact with a heated iron spiral in a hot-cold tube. F. FISCHER and A. JAEGER (Abhandl. Kennt. Kohle, 1925, 7, 68—74; Chem. Zentr., 1926, II, 847).— When an electrically-heated iron wire spiral was brought to red heat in a mixture of 15% of carbon dioxide, 23% of carbon monoxide, and 56% of hydrogen, contained at 120—150 atm. in an iron autoclave surrounded by cold water, the pressure fell to 70-80 atm. and a gas mixture containing about 9% of carbon dioxide, 40% of carbon monoxide, 22-29% of hydrogen, and 15-19% of methane hydrocarbons was formed. Water containing a considerable amount of formaldehyde was also produced, and solid carbon was deposited on the spiral. By heating the spiral more carefully (to about 620°) the hydrogen disappeared almost completely and a larger amount of saturated hydrocarbons was produced, together with much free carbon and an aqueous liquid of acid reaction containing formaldehyde. The results were practically identical if the iron spiral was previously treated with alkali, except that the yield of formaldehyde appeared to be less.

W. T. K. BRAUNHOLTZ.

Determination of traces of water in mineral oils. W. Boller (Chem.-Ztg., 1926, 50, 537—538).—The water vapour evolved on heating the oil is passed through a calcium carbide tube and the acetylene liberated determined as copper acetylide. The carbide tube, 50 cm. long, is arranged within an iron tube and mounted in an air oven. After a preliminary drying of the carbide until free from acetylene, pure and dry hydrogen is passed through the apparatus and the oil sample heated to 140° in an oil bath. After 1 hr. the carbide tube is heated to 180—200° and maintained at this temperature for 1 hr. The absorbent consists of 10 c.c. of 1:10 copper sulphate solution to which 4 c.c. of concentrated ammonia and then 3 g. of hydroxylamine hydrochloride have been added, and the whole diluted with water to 30 c.c. The precipitated copper acetylide, after washing with 2% ammonia solution, is converted into cupric oxide or determined volumetrically by its reaction with ferric

sulphate. Results as found in the case of an oil containing 0.01% of water are good, though tending to be slightly low.

C. IRWIN.

Improvement of the lubricating efficiency of oils by graphite, and its study by the aid of measurements of the heat of wetting. W. Bachmann and C. Brieger (Kolloid-Z., 1926, 39, 334—346).—The parellelism between lubricating efficiency and heat of wetting previously demonstrated (B., 1925, 435) is applied to explain the improved efficiency of oils to which graphite, especially colloidal graphite, has been added. Measurements show that the wetting heat oil/graphite is with a good oil 7—10 times greater than that of oil/ metal. The graphite forms a layer on the surface of the metal so that the efficiency of the lubricant is determined by the wetting heat oil/graphite. Almost all the oils investigated (good and bad) show the same wetting heat against graphite so that they are all raised to the same efficiency by its addition. The improvement brought about by the addition of a small percentage of an unsaturated fatty acid is also explained by the elevation of the heat of wetting which it causes.

N. H. HARTSHORNE.

See also A., Sept., 893, Determination of heats of combustion of volatile substances in the calorimetric bomb (Verkade and Coops). 909, Isothermal calorimetry (von Wartenberg and Lerner-Steinberg).

Ethylene obtained by cracking of petroleum for production of alcohol. Gerr and Popov.—See XX.

## PATENTS.

Transformation of lignites, coals, and other carbonaceous matter into fuels of high calorific power, including smokeless fuels. H. Debauche (E.P. 254,964, 28.11.25).—The carbonaceous matter is first subjected to low-temperature distillation, and the hot semi-coke is screened, graded, mixed, and agglomerated with a suitable binder (e.g., pitch), all the operations being carried on continuously. W. T. K. Braunholtz.

Treatment of peat. E. H. WINTER (E.P. 254,994, 16.2.26).—A hard and dense fuel is obtained from peat, without the necessity of expensive drying operations, by mixing the peat with a caustic substance (e.g., lime), an amylose carbohydrate (e.g., starch), an alum, a sugar (e.g., glucose, molasses), and an oxidiser or nitrate, and allowing the mixture to dry in the air. The materials are added, in aqueous solution or emulsion, while the peat is being macerated or disintegrated, a suitable mixture being 8000 pts. of wet peat, 40 pts. of lime, 10 pts. of starch, 1 pt. of alum, 2 pts. of saltpetre, and 12 pts. of maize-sugar or confectioner's glucose. A heat-insulating or packing material can be made in like manner, the nitrate being omitted and the quantity of the other added materials being reduced.

W. T. K. BRAUNHOLTZ.

Device for measuring flue-gas losses. T. Stein, Assr. to General Electric Co. (U.S.P. 1,591,444,

6.7.26. Appl., 10.2.25. Conv., 28.2.24).—The apparatus is designed to operate in accordance with the following modification of the Siegert formula:  $V = (W + c_1/k_n)$  ( $\Lambda t + c_2 k_2$ ), where V = total flue gas loss, W = content of unburned material in flue gas,  $k_2 =$  water content of flue gas,  $k_n = \mathrm{CO}_2$  content of flue gas after re-burning,  $\Lambda t =$  temperature difference between flue gas and entering air;  $c_1$  and  $c_2$  are constants. Three devices are provided for measuring, respectively, W,  $k_n$ , and  $\Lambda t$ , and these are connected to a measuring device which indicates the flue gas loss in any suitable units.

R. B. CLARKE.

Gas generating plants. A. Breisig (E.P. 235,891, 17.6.25. Conv., 18.6.24).—To obviate the use of a large relief holder for water-gas plants a heavy gas holder, with a capacity of only  $\frac{1}{20}$  to  $\frac{1}{100}$  of the hourly output and of sufficient weight to drive the gas through the purifying apparatus is installed between the generator and condensers. A throttle valve is fitted between the gas holder and the condensers, and is so adjusted that rather more than the average hourly output will pass through the valve and the holder will come right down at the end of each blowing period.

A. C. Monkhouse.

Manufacture of a mixture of water-gas and coalgas. A. Breisig (E.P. 241,902, 20.10.25. Conv., 21.10.24. Addn. to 207,651).—The method of carbonising fuels by internal heating and the use of heat accumulators as described in the chief patent (B., 1924, 122), is modified so that in the preliminary carbonisation of the coal, coke in excess of that required for the producer is made, thus producing a richer gas; the extra heat for carbonisation is provided by increasing the blow period.

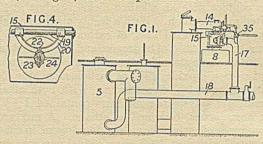
A. C. Monkhouse.

Air-blast device for semi-water-gas producers. Soc. Anon. D'Exploit. DES Brevets "Cousin" DITE "LE CHAUFFAGE IND." (E.P. 242,597, 14.8.25. Conv., 6.11.24).—A water tank is arranged centrally at the base of the producer, and an air blast is delivered to the centre of the latter under a bell, the lower edge of which extends below the water level in the tank, so that the air blast carries forward a fine spray of water which will instantly be vaporised. A cover is supported above the water tank so as to distribute the moist air blast below the zone of clinker formation, or it is arranged in a stepped disposition to afford the blast exits at various heights. level of the water in the tank can be adjusted or can be maintained constant by means of an overflow. The airblast device may be used in a producer with water-sealed ash-pit, the water tank being constituted by the ash-pit itself or by a tank immersed in it.

W. T. K. BRAUNHOLTZ.

Water-gas manufacture. G. R. Steere and J. B. Eberlein (E.P. 254,064, 19.5.25).\*—A "back-run" water-gas plant is provided with a swinging valve which allows the wash-box to be put into communication with either the superheater or the back-run pipe. The valve is operated by the ram of a hydraulic cylinder, which also actuates a two-way steam cock in such a way that steam enters the generator either via the "back-run" pipe or

via the superheater and the carburettor, according to the position of the valve. During the "blow" the swinging valve (see Fig. 4) is in the position indicated in Fig. 1,



and the air blast enters the generator via 18. During an "up-run" steam enters the bottom of the generator via 17 and 18. When using the "back-run" the swinging valve is reversed, so as to cut off 14 from the wash-box 8, and steam is admitted through 14 to the superheater, whence it passes through the carburettor to the top of the generator. The gas made during the "back-run" leaves the generator through 18 and enters the wash-box via 17. Thus 18 serves as blast main, a "back-run" pipe, and a steam main to the generator. S. Pexton.

Pressure-gas producers. K. Koller (E.P. 254,392, 2.4.25).—To enable the steam-air mixture to be correctly distributed at the base of a grateless gas producer without obstructing the passage of ash and clinker, a distributor in the form of a short cylindrical pipe or dome in which are openings is used. The outlet openings are regulated by slides in the blast nozzle, thus altering the velocity of the emerging mixture; the steam and air are supplied through channels in the distributor in order to keep it cool. Another form of distributor consists of rings separated by the nozzle openings with a common space in the cap or dome; the path of the steam-air mixture in this case is reversed before emerging at the nozzles and adjustment is made by a rotary slide.

A. C. Monkhouse.

Construction of regenerative coke-oven flues. R. F. F. Fabry (E.P. 254,652, 24.3.26).—The principles of E.P. 105,047 (B., 1917, 497) are applied to regenerative ovens with vertical flues. Each vertical flue has a nozzle at its lower end which serves alternately as air inlet and as outlet for the products of combustion; a single gas inlet common to both vertical flues is arranged under the partition which divides them in order to direct the supply of combustible gas always into that vertical flue which for the time receives an ascending supply of hot air from the corresponding regenerator. The gases are diluted by the products of combustion descending the other vertical flue.

A. C. Monkhouse.

Coke ovens. H. Schröder (E.P. 254,960, 24.11.25).

—In horizontal coke ovens, having regenerators beneath and parallel to the oven chambers, the substructure supporting the chambers and heating walls is strengthened by the provision of arches extending over the width of a number of ovens and each containing a regenerator separated from the adjacent ones by supporting walls.

Each regenerator is covered by two arches, one for support and the other permitting expansion of the brickwork.

W. T. K. BRAUNHOLTZ.

Manufacture of coke. H. J. Toogood (E.P. 254,811, 14.4.25).—Metallic retorts are divided by longitudinal and transverse partitions into narrow compartments, open at the top and bottom, and a machine travels along the gasworks retort house and inserts and removes these retorts bodily into and from other retorts, such as the usual gasworks retorts. The stoking machines are provided with means for quenching the discharged coke and utilising its heat for drying and preheating coal for future carbonisation, and also with means for grading the fuel and compacting it as it is fed into the metallic retorts.

W. T. K. Braunholtz.

Coking coal. M. W. Ditto and W. M. Duncan (U.S.P. 1,591,023, 6.7.26. Appl., 29.3.23).—A retort, divided by a vertical wall into two chambers, is fed by a conveyor below the retort. The two chambers are sealed by a rolling contact between the division wall and the coal in the conveyor. Fuel is burnt with air in the one chamber and the products of combustion carbonise the fuel in the other chamber.

A. C. Monkhouse.

Distilling bituminous coal with recovery of gaseous distillate. R. M. Folsom and C. A. Raymond, Assrs. to New England Fuel & Transportation Co. (U.S.P. 1,591,672, 6.7.26. Appl., 19.8.25).—Coal is carbonised under suction in a battery of coke ovens with overhead charging. To prevent loss of gaseous products the suction is increased during charging and the products are withdrawn at the opposite sides of the charging openings.

A. C. Monkhouse.

Making activated carbon. H. Rodman, Assr. to Rodman Chemical Co. (U.S.P. 1,591,235, 6.7.26. Appl., 21.8.18).—Carbon which is kept in motion is heated in a furnace to the required temperature by direct contact with hot gases.

A. C. Monkhouse.

Absorbent charcoal. E. Urbain (F.P. 604,181, 31.12.24).—Activated charcoal is made under such conditions that products of different density are obtained which exhibit different activity towards the same gases and vapours. W. G. Carey.

Activated charcoal. J. Magtegaal (F.P. 604,417, 10.10.25. Conv., 15.10.24).—The gases from the dry distillation of carbonaceous material, cooled and purified if necessary, are led from the retorts into a furnace, where they are burned and pass over incandescent carbon.

W. G. Carey.

Preparation of a highly active charcoal. I. Deiglmayr Chem. Fabr. A.-G., Assess. of H. Schwarf (G.P. 430,031, 24.5.23).—A mixture of beet molasses and brewers' grains is carbonised in presence of potassium carbonate, the product is washed with dilute acid, and ignited again in the absence of air. W. G. Carey.

Filling mass for containers for explosive gases. C. Ness, Assr. to Prest-O-Lite Co. (U.S.P. 1,591,397, 6.7.26. Appl., 24.8.25).—A mixture of plaster of Paris, water, and acetone is used as a filling mass for receptacles for storing explosive gases.

A. C. Monkhouse.

Treating motor fuels. T. MIDGLEY, JUN., ASST. to GEN. MOTORS CORP. (U.S.P. 1,592,953, 20.7.26. Appl., 4.10.22).—A solid pellet for adding to motor fuels contains lead tetraethyl and p-toluidine. H. MOORE.

[Motor] fuel. T. Midgley, jun., Assr. to Gen. Motors Corp. (U.S.P. 1,592,954, 20.7.26. Appl., 19.5.23).

—A motor fuel is mixed to a homogeneous liquid with an anti-knock substance containing lead and a substance which combines with the lead during combustion to form a compound having a lower fluxing action than lead oxide.

H. Moore.

Motor fuel. T. Midgley, Jun. (U.S.P. 1,592,955, 20.7.26. Appl., 22.1.26).—An internal-combustion engine fuel contains a volatile phenyl compound of a metal. H. Moore.

Improving motor fuel. D. R. Stevens, S. P. Marley, and W. A. Gruse, Assrs. to Gulf Refining Co. (U.S.P. 1,593,040, 20.7.26. Appl., 28.12.25).—A material containing an aluminium halide is treated with a combustible organic liquid, which is then added to motor fuel to improve its knocking qualities. H. Moore.

Conversion of crude mineral or shale oils or tar oils into light oil or spirit and preparation of light oil or spirit from coal, lignite, or other carbonaceous material. E. Schultz (E.P. 254,011, 25.3.25).—Carbonaceous material is heated in a still to 400°, or to a temperature sufficient to release all volatile matter, and the vapours are led to a converter containing chemicals such as common salt, zinc chloride or oxide, lime, zinc shavings, bauxite, aluminium chloride, or magnesia, and maintained at a temperature up to 350°, but always lower than that of the retort. A retort such as is described in E.P. 252,422 (B., 1926, 654) may be used. Alternatively, crude oil may be heated in a still and the vapours passed to such a converter. The matter in the converter may be supported on a rotating screw. The vapours from the converter are passed to a condenser. The condensate is washed with acid, neutralised, and fractionally distilled.

H. MOORE.

Cracking and hydrogenation of hydrocarbon oils. G. W. Wallace (E.P. 255,159, 17.4.25).—The process is carried out in a horizontal or inclined chamber through which pieces of material, such as carbon, carbon briquettes, firebrick, alumina, etc., are continuously passed, and are agitated by rotary crushers to expose fresh surfaces. The material may be coated with a catalyst such as nickel. The oil is introduced as spray or vapour, and passes in counter-current to the catalytic material, which is circulated by a plunger and drops into a vertical extension of the chamber, from which it may

be withdrawn, and is then returned to the horizontal chamber either immediately or after regenerative treatment in the hot state with gas, air, or steam. Air may be introduced with a portion of the oil to form a combustion zone either within or without the chamber. The oil before entering the chamber may be preheated by the combustion of the gases produced by cracking. Hydrogen or steam, or producer gas generated by the action of air and steam on the hot carbon deposited on the material, may be introduced to assist hydrogenation. The cracked vapours are withdrawn by suction near the end where the contact material is introduced.

H. MOORE.

Refining oil. B. V. Stoll (U.S.P. 1,593,275, 20.7.26. Appl., 26.10.22).—Oil is cracked by heating in a coil to about 370—400° under a pressure of about 150 lb. per sq. in. and the product expanded in a cylinder still at about 200—260° under a pressure of about 100 lb. per sq. in.

H. Moore.

Treating hydrocarbon gases. J. E. Kobernik, Assr. to Newton Process Manuf. Co. (U.S.P. 1,594,014, 27.7.26. Appl., 27.10.25).—A hot absorbing medium containing hydrocarbons is brought into intimate contact with a hot lean hydrocarbon gas, and the gas and the medium are cooled and subsequently again brought into intimate contact, these operations being repeated as often as is necessary. The medium is afterwards distilled to recover a commercial stabilised gasoline.

H. Moore.

Apparatus for distilling oils. L. E. Lee (U.S.P. 1,594,296, 27.7.26. Appl., 9.11.21).—The still has a vertical inner shell, against the upper end of which a nozzle projects a jet of oil, and from which the oil is projected on to a dome above the upper end of the shell. The oil is atomised by its impact against the dome, and thence directed to the outer surface of the inner shell, which is heated to vaporise the oil passing over it. An outer shell surrounding the inner shell forms a space to receive the vapours.

H. Moore.

Refining mineral lubricating oils. J. W. Weir (U.S.P. 1,592,058, 13.7.26. Appl., 14.5.25).—Lubricating oil stock of relatively low viscosity is treated with sulphuric acid until the action is complete, then, without removing the sludge, is mixed with lubricating oil stock of relatively high viscosity, which is acted upon by the acid sludge.

D. Woodroffe.

Production of fuel mixtures. T. H. BUTLER, H. W. ROBINSON, and D. W. PARKES (E.P. 256,107, 8.12.25).—See U.S.P. 1,583,573; B., 1926, 572.

Treatment of certain fuels [lignites] to improve their calorific value. W. A. Bone (U.S.P. 1,594,994, 3.8.26. Appl., 5.7.19. Conv., 30.7.18).—See E.P. 130,445; B., 1919, 709 A.

Separation of dry materials [coal]. K. C. Appleyard, P. W. Bewick, J. F. Laycock, M. R. Portal, and W. E. Manners (E.P. 255,924, 24.4.25). Coal pulverising machines. A. Herbert and R. Jackson (E.P. 255,957, 2.5.25, and 255,966, 5.5.25).

Coke cooling plant. C. Schwartz (E.P. 252,118, 22.12.25. Conv., 12.5.25).

[Quenching] coke when discharged from retorts and the like. Drakes Ltd., and J. W. Drake (E.P. 256,142, 29.5.25).

## III.—TAR AND TAR PRODUCTS.

Application of the Bergius process to coal tar. L. Rheinfelder (Mitt. Schles. Kohlenforschungsinst. Kaiser-Wilhelm-Ges., 1925, 2, 34-67; Chem. Zentr., 1926, II., 519-520).- "Berginised" tar yielded light oils which, after removal of acidic and basic constituents, were divisible into three fractions boiling between 70° and 170°, in which benzene, toluene, and xylene respectively could be identified. The neutral oils boiling below 70° appeared to be a mixture of aliphatic or cyclic saturated hydrocarbons with small quantities of unsaturated and aromatic hydrocarbons. Low-boiling aliphatic or cyclic saturated hydrocarbons (perhaps also cyclohexane) were isolated in very small quantities. "Berginisation" of tar has the effect of increasing the content of low-boiling phenols and bases at the expense of the higher homologues. Aniline was isolated in one experiment. De-alkylation appears to take place, and a large quantity of methane hydrocarbons is found in the gases finally leaving the autoclave. The nature of the final products, i.e., the extent of de-alkylation and decomposition, will depend on the temperatures and pres-W. T. K. BRAUNHOLTZ. sures employed.

## PATENTS.

Conversion of tar oils into light oils (E.P. 254,011).
—See II.

Extraction process (G.P. 430,087).—See XX.

## IV.— DYESTUFFS AND INTERMEDIATES.

Viscosity and hydration of dye solutions. LIE-PATOV.—See A., Sept., 903.

Organic dyes in vulcanised rubber. Drakeley.—See XIV.

#### PATENTS.

Manufacture of condensation products and dyestuffs of the benzanthrone series [isodibenzanthrones]. J. Y. Johnson. From Badische Anilin- & Soda-Fabrik (E.P. 255,277, 7.10.25).—isoDibenzanthrones are obtained by the action of alkaline condensing agents on a mixture of a benzanthrone, not substituted in the 3-and 4-positions, with a Bz-halogenbenzanthrone having a free 4-position. The condensing agents used may be mixtures of caustic alkalis with alkali alkoxides in the presence of an inert diluent, or metal arylides, such as sodium anilide, and oxygen should be excluded during the reaction. By moderating the reaction (e.g., by the

use of a lower temperature), dibenzanthronyls are formed as intermediate products, and these may be further converted into isodibenzanthrones by the action of alkaline or acid condensing agents. For example, a mixture of 20 pts. of benzanthrone and 23 pts. of Bzchlorobenzanthrone is added, in a current of nitrogen, at 5° to sodium anilide (prepared from 6 pts. of sodium and 400 pts. of aniline), and the mixture stirred for several hours, the temperature being allowed to rise to 12°. After removal of aniline and extraction of the residue with acetone, 3:4'-dibenzanthronyl, yellow needles, m.p. 326°, is obtained. To convert this into isodibenzanthrone, 10 pts. of it are added, in a current of nitrogen, to sodium anilide (from 3 pts. of sodium and 200 pts. of aniline) at 125° and the mixture is heated at 140—150°. The following additional new compounds are described: 8-chloro-3: 3'-dibenzanthronyl, brownish-yellow, m.p. above 360°; 9-chloro-3: 4'-dibenzanthronyl, m.p. above 360°; 8-methyl-3: 4'-dibenzanthronyl, yellowish-green, m.p.above 360°. 8-Chloroisodibenzanthrone dyes cotton in much bluer and stronger shades than isodibenzanthrone.

A. DAVIDSON.

Preparation of sulphurised condensation products [dyes]. I. G. Farbenind. A.-G., Assees. of J. Binapfl and L. Strohmenger (G.P. 427,970, 2.11.23).—Mixtures containing azobenzene or its homologues and aromatic amines or phenols, or the condensation products obtained from azo-compounds and aromatic amines or phenols, are heated with sulphur dichloride or sulphur in the presence of a condensing agent, the coloured products being dyes or capable of conversion into dyes by further heating with concentrated or fuming sulphuric acid. For example, a bluish-green vat dye is obtained by heating at 210° a mixture of azobenzene, o-cresol, sulphur, and zinc chloride.

A. J. Hall.

Manufacture of [stable] preparations containing diazotised p-nitroaniline. I. G. Farbenind. A.-G., Assees. of K. Schnitzspahn (G.P. 426,033, 28.12.24).— Solid sodium nitrite is incorporated in a mixture containing 1 mol. of an aromatic nitroamine having no sulpho- or carboxyl-groups, more than 1 mol. of sulphuric acid (the mixture may also consist partly of equivalent proportions of the sulphate of a nitroamine and bisulphates), and a suitable diluent such as partly dehydrated aluminium sulphate or alum, arylsulphonates or mixtures of arylsulphonates and partly dehydrated alum; the products are very stable and liberate the diazo-compound when dissolved in cold water.

A. J. Hall.

Monoazo dyes. W. Duisberg, W. Hentrich, and L. Zeh, Assis. to Grasselli Dyestuff Corp. (U.S.P. 1,595,178, 10.8.26. Appl., 26.11.24. Conv., 5.12.23).—See E.P. 225,862; B., 1925, 583.

Halogenated vat dyestuffs of the anthraquinone series. I. G. Farbenind. A.-G., Assees. of A. Holl (U.S.P. 1,595,549, 10.8.26. Appl., 9.6.25. Conv., 20.6.24).—See E.P. 235,919; B., 1925, 703.

Extraction process (G.P. 430,087).—See XX.

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

Viscose. II. Ripening of viscose. M. Numa (Cellulose Ind., Tokyo, 1926, 2, 136-148).—The effects of temperature, light, duration of ripening, and other influences on the ripening of alkali-cellulose have been determined by studying the colloidal properties of cuprammonium hydroxide solutions of the regenerated cellulose, the colloidal properties of the viscose solutions, and the physical properties of the resulting viscose pellicles. The relationship between intensity of colour as measured by a Duboscq colorimeter and the degree of dispersion, observed by Svedberg in the examination of colloidal solutions of gold, has been found to apply in the case of cuprammonium hydroxide solutions of cellulose. The degree of dispersion of alkali-cellulose reaches a maximum after a relatively short period of ripening, and then slowly decreases; this point of maximum dispersion marks the optimum time of ripening, as is confirmed by an examination of the viscose solution and of the films prepared therefrom. Light and temperatures above 20° have a deleterious effect on alkali-cellulose; there is, however, no necessity to conduct the ripening process in the presence of inert gases provided that free access of air is avoided. The best conditions of ripening are, therefore, 5-20 hrs. at a low temperature in a closed, opaque vessel containing as little air as possible.

D. J. NORMAN.

Esparto pulp by the "Keebra" process. J. D'A. CLARK (Pulp and Paper Mag., 1926, 24, 815).—The "Keebra" process (digestion with pure normal sodium sulphite solution at, for example, 130 lb. per sq. in. for wood and 50 lb. per sq. in. for esparto) offers difficulties in the recovery of the soda: if, however, a modified ("Semi-Keebra") process is adopted, recovery of the soda becomes a practical proposition. This modified process is a soda process in which about 25% of the caustic soda is replaced by its equivalent of normal sodium sulphite; somewhat lower pressures may be used and the yield, though not so high as with the full "Keebra" process, is about 10% higher than that given by the ordinary soda process. "Semi-Keebra" esparto pulp, moreover, gives a stronger sheet of paper with more "Semi-Keebra" wood pulp resembles soda pulp but is slightly harder, stronger, and more easily bleached. D. J. NORMAN.

See also A., Sept., 899, Adsorbent properties of cellulose nitrate (Duclaux). 902, Colloidal characters of cellulose (Herzog); Viscose solutions (Herzog, Gaebel, and Jancke). 903, Diffusion researches on solutions of cellulose in copperammonia solution (Herzog and Krüger). 935, Detection of hydroxyl groups of different types. Application to lignin (Freudenberg and Hess).

Flax wax. Honneyman.—See XII.

Fluorescence of sulphite-cellulose extract. MEUNIER and JAMET.—See XV.

Nitrous esters of cellulose. Blechta.—See XXII.

#### PATENTS.

Treating fibres. OBERRHEINISCHE HANDELSGES. M.B.H., and L. UBBELOHDE (E.P. 254,357, 2.3.25).—The process previously described (cf. E.P. 239,605; B., 1925, 877) is modified in that a proportion of the roughening agents is left on the fibre, retention being assisted where necessary by the use of adhesive agents such as grease: alternatively, the fibres may be mixed with an aqueous suspension of the roughening material, centrifuged, dried under pressure, and finally freed from dust. Suitable roughening agents are carborundum, keiselguhr, lime, and the like, but, for long fibres such as artificial silk, materials with softer edges, for example, silica gel, may be satisfactorily used. The retention of roughening agents by the fibre tends to make the yarn more voluminous and more resistant to tearing.

D. J. NORMAN.

Felting process for woollen and like textile fabrics. E. RICALENS (E.P. 254,609, 19.1.26).—The fabric, after a slight fulling operation, is circulated in a bath containing soap and wool dust until the required quantity of wool dust has been taken up. The treated fabric is then pressed, and washed to remove the impregnating liquor. The soap solution is preferably prepared by saponifying olein with sodium carbonate.

D. J. NORMAN.

Oxidation (chlorination) of textile materials made from animal fibres, e.g., wool and silk. W. H. Schweitzer (G.P. 430,109, 21.5.24).—Animal fibres, with or without previous treatment with an oxidising agent such as sodium hypochlorite, are treated at high or low temperatures with a solution containing 1% (on the weight of fibre) of sodium p-toluenesulphochloramide.

A. J. Hall.

Treatment of artificial [viscose] silk and th like. Viskose A.-G., Assees. of C. Becker and A. Bernstein (E.P. 242,993, 11.11.25. Conv., 14.11.24).— Viscose threads or filaments, coagulated and wound in the usual way, are unwound, while still wet in the acid condition or after washing, and passed successively through an alkaline desulphurising bath and a weak acid bath. The treated threads are finally re-wound in a washing bath.

D. J. Norman.

Improving artificial fibrous materials. L. LILIEN-FELD (E.P. 253,853, 16.7.25. Conv., 17.6.25).—Cellulose silk yarns in skeins or fabric are treated with caustic soda of concentration not exceeding 5% and then dried before or after removal of the alkali by washing, or souring and washing with water or solutions of inorganic salts (e.g., sodium chloride and ammonium sulphate), the yarns being stretched during at least a part of the treatment; the resulting yarns have an increased strength of 30—100%. For example, viscose, cuprammonium, or Chardonnet silk is simultaneously impregnated with a 0·2—0·3% solution of caustic soda and stretched, then dried, soured with 10% sulphuric acid, washed with water, and dried. Cellulose silk thus treated may be subsequently steamed or heated, and especially good results are obtained if the treatment with alkalis is effected with solutions of less than 1% concentration.

A. J. Hall.

Improving artificial fibrous materials. L. LILIEN-FELD (E.P. 253,854, 17.7.25. Conv., 17.6.25. Addn. to 231,806; B., 1925, 985).—Artificial silk (viscose, cuprammonium, Chardonnet, and cellulose acetate) yarn in skeins or fabric is impregnated with a solution of a cellulose thiourethane in which at least one hydrogen atom of the amino-group is replaced by an alkyl radical, the solvent being subsequently removed; the resulting silk has 40-100% increased tensile strength. Suitable solvents include aqueous solutions of alkalis such as caustic soda or ammonia, and volatile solvents such as pyridine. The silk yarn is preferably stretched during the treatment (cf. E.P. 253,853; preceding), and the impregnated silk may be rendered highly flexible by exposure to the vapours of a suitable organic solvent, e.g., pyridine, as described in the chief patent and also in E.P. 248,994 (B., 1926, 532). For example, viscose yarn is drawn in a stretched condition through a solution containing 100 pts. of a cellulose-N-phenylthiourethane prepared as described in the chief patent or E.P. 248,994 or 248,246 (B., 1926, 400), 900 pts. of 2% caustic soda, and 2000-6000 pts. of water, then passed through a precipitating bath containing 10% of sulphuric acid, washed, and dried; after-steaming is optional.

A. J. HALL.

Manufacture of artificial silk. Courtaulds, Ltd., and C. M. Whittaker (E.P. 254,531, 27.7.25).—The difficulty of ensuring that the whole of the silk in any batch of viscose silk has a uniform affinity for dyes may be overcome by treating a number of skeins in the same dye-bath for the same time (using a dye that tends to produce uneven shades and is at the same time easily removable), sorting those skeins which show substantially the same shade into separate batches and finally removing the dye. The preliminary dyeing should be carried out at a comparatively low temperature to enhance any unevenness of shade. (Cf. Wilson and Imison, J.S.C.I., 1920, 323 t.)

Artificial [silk] fibre and process of manufacture. R. ALLWATER and A. HEINEMANN (E.P. 255,623, 4.7.25). —Fibres are manufactured by the usual spinning process from a cellulose solution prepared by dissolving a protein such as keratin, fibrin, spongin, or konchoelin in a solution of a caustic alkali and adding successively cellulose and carbon disulphide and a further quantity of alkali if necessary to maintain the alkali in excess. The most suitable protein is keratin obtained from feathers, hair, horn, or hoof. A satisfactory spinning solution is prepared by dissolving 100 pts. of keratin in a 10% solution of caustic soda, adding 100 pts. of cellulose and 10-30 pts. of carbon disulphide, sufficient alkali being then added to maintain the product alkaline. The resulting fibres have greater resistance to water and absorb dyes more readily than viscose silk and to some extent have the properties of natural silk. A. J. HALL.

Manufacture of artificial [coating, sizing, etc.] materials. L. LILIENFELD (E.P. 247,223, 8.2.26. Conv., 6.2.25. Addn. to 231,805; B., 1925, 985).— Derivatives of aniline are used instead of the bases proposed in the chief patent as solvents for cellulosexantho-fatty acids in the presence of water since, with the exception of pyridine, the previously proposed bases yield solutions which become gelatinous on keeping. Suitable derivatives of aniline include the monoalkylderivatives or their homologues, and usually not more than 1 mol. of the base is sufficient to dissolve 1 mol. of cellulose-xantho-fatty acid, the resulting solutions being stable at room temperature for months. Although turbid solutions are obtained when an excess of a base is used, the stability of such solutions is good when derivatives of aniline are used as bases. The clear solutions dry to products which are insoluble in water and the resistance of which to water may be increased by heating or steaming. A solution suitable for conversion into artificial materials or for use as a coating material is obtained by kneading 100 pts. of air-dried cellulosexanthoacetic acid (prepared as described in the chief patent) with 1,900 pts. of water containing 23 pts. of monomethylaniline. A. J. HALL.

Process of making cellulose acetate. W. R. Webb and C. J. Malm, Assrs. to Eastman Kodak Co. (U.S.P. 1,591,590, 6.7.26. Appl., 7.10.25).—Cellulosic material is treated in the presence of chlorine with an acetylating agent comprising a homogeneous mixture of acetic acid, acetic anhydride, and red phosphorus.

D. J. NORMAN.

Cellulose acetate composition. B. K. Brown and C. Bogin, Assrs. to Commercial Solvents Corp. (U.S.P. 1,591,652, 6.7.26. Appl., 14.10.25).—The composition contains cellulose acetate and a multivalent metal salt of a monoalkyl ester of phthalic acid. D. J. Norman.

Chemical pulp-cooking process. G. Sivola (U.S.P. 1,590,987, 29.6.26. Appl., 4.12.25).—The progress of the digestion of sulphite-pulp is ascertained in the following manner:—A sample of the liquor is removed and its content of total sulphur dioxide determined. Simultaneously, a sample of pulp from the digester is washed, freed from water, and 2.5 g. (calculated on the dry product) are shaken with 200-300 c.c. of 0.01Npotassium permanganate at 25°, and the time taken for the solution to change from violet to yellow is noted. The speed of this reaction is a measure of the delignification of the pulp. These tests are repeated at halfhour intervals and the temperature is also taken. The results when tabulated enable the operator to decide on a temperature rise best suited to the economical production of a desired quality of pulp. R. B. CLARKE.

Retting flax and other vegetable fibres. H. Thellier, Assr. to Soc. Pour L'Appl. Ind. des Brevets Peufaillit (U.S.P. 1,594,389, 3.8.26. Appl., 26.12.24. Conv., 18.1.24).—See E.P. 227,836; B., 1925, 396.

Coating paper pulp vessels, plates, or the like for the protection thereof against the action of moisture, fat, soap, and the like. Baumgärtner, Katz & Co. G.M.B.H. (E.P. 241,876, 23.9.25. Conv., 27.10.24).

Electrodeposition of rubber on fabrics (U.S.P. 1,589,325).—See XI.

Electrodeposition of cellulose compounds (U.S.P. 1,589,326—8).—See XI.

Electrodeposition of mixtures of rubber and cellulose compounds (U.S.P. 1,589,320 and 1,589,322).
—See XI.

Converting sulphite-cellulose waste liquors into tanning extracts (U.S.P. 1,592,062—3).—See XV.

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

New dyeing processes using Indigosol O. G. Friedländer (Textilber., 1925, 6, 916-917; cf. B., 1926, 705).—A full indigo shade four times faster to soap and also faster to rubbing than vat-dyed indigo is obtained directly by slop-padding fabric with Indigosol O and suitable assistants (cf. Indigosol DH, Vaucher and Bader, B., 1924, 864), particularly a large proportion of ammonium vanadate, and drying in a hot flue or even on drying cylinders. New effects are obtained by dyeing cotton with a mixture of Indigosol O and a suitable direct dye, e.g., Diamine Pure Blue, and afterwards treating with iron or copper salts whereby the fastness to light of the resulting shade is improved; reserve effects are obtained with sodium thiosulphate, and both dyes may be discharged to white by means of a hyposulphite. A combination of Aniline Black and Indigosol O is being used in Japan. Brown grounds are produced with combinations of Indigosol O and Para Red or m-Nitraniline Orange. Fabric impregnated with a mixture containing 60 g. of Indigosol O, 5 g. of sodium thiosulphate, 223 g. of a solution of ammonium chlorate of 15° B. (d 1.115), and 712 g. of water, and dried at a low temperature, is sensitive to light and may be used for photographic reproduction. Indigosol O is more easily discharged than vat-dyed indigo by the usual chlorate discharge process. Fabrics having red and blue, or orange and blue, effects are produced by slop-padding the fabric with an Indigosol O preparation, drying, and then printing with a nitrosoamine red (p-nitroaniline) or orange (m-nitroaniline) reserve. Fabric printed with Indigosol O and aluminium, chromium, or iron mordants and afterwards steamed may then be dyed with alizarin or nitrosonaphthol. A. J. HALL.

[Prevention of] bronzy shades in dyeing with sulphur dyes. W. Kosche (Textilber., 1925, 6, 665).— The production of undesirable bronzy shades characteristic of the dyeing of cotton with black and blue sulphur dyes, particularly when a standing bath is used,

may be prevented by treating the dyed fabric with a 0·1% solution of sodium sulphide at 30°, or preferably with a solution containing 3% of acetic acid and 2% of sodium dichromate at 60°. Fabrics dyed with blue sulphur dyes and subjected to oxidation with a 1% solution of sodium perborate at 60° to develop the full shade are treated with sodium sulphide before the oxidation. Bronzy fabrics which have been dried are directly treated with a 0·2% solution of sodium sulphide, previous wetting-out being unnecessary.

A. J. Hall.

[Prevention of] bronzy shades in dyeing cotton and artificial silks with sulphur, basic, and direct dyes. C. Koox (Textilber., 1926, 7, 543—544; cf. Kosche, preceding abstract).—For removing the bronzy appearance of cotton and artificial silk fabrics dyed with sulphur dyes, the dyed fabric is treated with the usual hot "brightening" bath (containing a Monopole soap or oil and lactic, formic, or acetic acid) to which 1-2 kg. of gelatinised potato starch is added per 60-100 kg. of dyed fabric. The resulting non-bronzy fabric is somewhat harsh in handle, but this defect is removed by treating the material afterwards in a solution at 35-40° containing lactic, formic, or acetic acid and diastase (100 g. per 10 kg. of dyed material), and then washing it free from the starch hydrolysed by the diastase. Bronzy shades on artificial silk dyed with basic dyes are avoided by the addition of potato starch (50-100 g. per 10 kg. of dyed material) to the dye-bath and afterwards correcting the resulting harshness and loss of brilliancy of shade by treatment with a solution of diastase; the resulting shade has increased fastness to rubbing.

A. J. HALL.

Influence of after-treatments on the fastness to light of dyeings obtained by means of Naphthol AS compounds. E. KAYSER (Textilber., 1926, 7, 437-440).-Cotton dyed with Naphthol AS-SW and Fast Red KB base contains the resulting yellowish-red pigment uniformly distributed throughout the cell wall, the cuticle and lumen being free from pigment, but during subsequent soaping for ½ hr. at boiling point the pigment becomes crystalline and bluish-red, and the greater part migrates to within the lumen. This behaviour, which confirms the observations of Haller and Ruperti (B., 1926, 316), is more readily seen in fibres swollen by means of caustic alkalis or cuprammonium solution, so that the cell wall (cellulose) occupies about two-thirds and the lumen one-third of the fibre. Migration of pigment does not occur in boiling water or boiling (ca. 109°) solutions of sodium chloride, but does occur in boiling water containing an alkali and also to a limited extent in solutions of soap containing sodium chloride. Migration is dependent on swelling of the cotton cellulose and is accompanied by an increase of fastness of shade to light. For example, shades produced on cotton by means of Naphthol AS and Fast Red GL base, and Naphthol AS-SW and Fast Red KB base, afterwards washed in cold water and soaped at 50°, 75°, and boiling point showed increasing fastness to light. Photomicrographs of sections of dyed fibres before and after soaping are given. Changes of shade produced by after-treatment are due to optical effects and not to change of constitution of the dye.

A. J. Hall.

Leather dyeing. III. H. SALT (J. Soc. Leather Trades Chem., 1926, 10, 168—171; cf. B., 1926, 153).— Basic dyes give full shades on chamois leather, but are not readily absorbed and retained if the leather is re-tanned with chromium salts. Unchromed chamois leather will retain 1% of basic dye in the surface layer. The basic dye diffuses evenly through chromed chamois. Direct dyes alone are of little use in dyeing chamois leather. Acid dyestuffs will dye chamois leather whether re-tanned with chromium salts or not. It is suggested that both the basic dyes and the chromium compounds combine with the acid groups in the hide substance molecule. Hence the chamoising must affect these groups very little, if at all. Chroming of chamois leather does not affect the dyeing with acid dyes, hence the chromium salts cannot combine with the free basic D. WOODROFFE. groups in chamois leather.

Leather dyeing .. IV. H. SALT and A. ASTROM (J. Soc. Leather Trades Chem., 1926, 10, 197-199; cf. preceding abstract).—Chrome leather was dyed with 2% of Primuline extra, Chlorazol Brown G.M., or Chlorazol Brown M., without the application of tannin, then washed and drummed in 500% of water (on weight of stock) containing 4% of hydrochloric acid and 2% of sodium nitrite for 20 min. at the lowest possible temperature. After rinsing, the dyed leather was transferred to a solution of 0.3% of phenol, resorcinol,  $\alpha$ - or  $\beta$ naphthol, and 0.1% of caustic soda or a solution of 0.3% of m-phenylenediamine and 0.14% of soda ash. This coupling process changed the colour, but it was fast to water, hot soap solution, sun, air, and 1% sodium carbonate solution. The dyed leather should be treated with 0.5% borax solution to neutralise any acid left D. WOODROFFE. from the diazotising process.

Reserves obtained by means of algin. Kunig (Textilber., 1926, 7, 538-539).-" Algin," or "norgin," a product (containing alginic acid) obtained from seaweed, forms soluble sodium and magnesium salts, but with salts of alkaline-earth and heavy metals yields insoluble horn-like products which resist penetration by dye liquors, and may therefore be used in reserve pastes for printing and batik dyeing. Veined batik effects obtained by means of fabric prepared by impregnation with an 8-10% solution of algin, drying, coagulating with a suitable metal salt, washing, and drying, are exceptionally clearly delineated; fabric thus prepared is superior to that prepared in the usual manner with wax, since it may be calendered, heated, or steamed without reducing its resist properties. A particularly suitable method for preparing fabric with algin consists of impregnating it with an ammoniacal solution of copper sulphate containing algin and subsequently fixing the copper alginate in an insoluble form by steaming. Algin resists on fabrics are removed by successive treatment with an acid and a weak alkali, e.g., sodium carbonate, so that only dyes fast to these treatments may be used.

A. J. Hall.

### PATENTS.

Dyeing cellulosic fibres. Durand & Huguenin, S.A. (E.P. 231,189, 20.3.25. Conv., 20.3.24).—More intense dyeings may be obtained on cellulosic fibres, particularly cotton, mercerised cotton, viscose, and the like, when using the ester salts of vat dyestuffs described in E.P. 186,057 (cf. U.S.P. 1,448,251; B., 1923, 398 A) if there is added to the dye-bath a salt, such as common salt or Glauber's salt, which lowers the solubility of the ester salt and thereby increases the proportion taken up by the fibre. Example: Viscose is immersed for 15 min. at the ordinary temperature in a bath containing per litre 6.6 g. of the sodium salt of the leuco-indigo sulphuric acid ester previously described (loc. cit.), 6.6 g. of sodium nitrite, and 33 g. of anhydrous sodium sulplate, the total weight of the solution being about ten times that of the fibre under treatment. The fibre is then wrung out and developed in twenty times its weight of a solution containing 2% of sulphuric acid (96%) and preferably the same proportion of sodium sulphate as was present in the dye-bath. The viscose is finally washed and dried.

D. J. NORMAN.

Dyeing fast shades on wool. Badische Anilin- & Soda-Fabr. (F.P. 601,430, 31.7.25).—Dyes not sufficiently soluble in water to allow of their direct application to wool from aqueous solutions are formed within the fibre by impregnating wool or half-wool materials with monohydroxy-compounds, except α- or β-naphthol, polyhydroxy-, amino-, or aminohydroxy-compounds and developing subsequently with suitable diazo-compounds. For example, brown (fast to milling), red (fast to milling and potting), and bluish-red shades are obtained by means of the sodium salt of 1:5-dihydroxynaphthalene and diazotised m-xylidine (in feebly alkaline solution), 2: 3-hydroxynaphthoyl-7-hydroxy-2-naphthylamide and diazotised p-nitroaniline, and di-(2:3-hydroxynaphthoyl)-1:5-naphthylenediaminedisulphonic acid and diazotised m-xylidine respectively. 2:3:6-Benzamidonaphtholsulphonic acid and diazotised m-xylidine, di-(acetoacetyl)-o-tolidide and diazotised aminoazobenzene or o-chloroaniline yield red, orange, and yellow shades respectively. Black and reddish-brown shades are obtained by dyeing wool with the monoazo-dyes prepared from α-naphthylamine and 1:8:5-aminonaphtholsulphonic acid, and 5-nitro-2-amino-p-xylene and 2:8:6aminonaphtholsulphonic acid, and then developing with diazotised acetyl-p-phenylenediamine or diazotised mxylidine respectively. Deep black shades are obtained by dyeing wool with the monoazo-dye obtained from o-aminophenol-p-sulphonic acid and 1:5-dihydroxynaphthalene, then chroming and developing with diazotised m-xylidine. A. J. HALL.

Treatment of animal fibres with acid, alkaline, oxidising, or reducing liquors. M. Bergmann, E. Immendörfer, and H. Löwe (G.P. 426,624, 1.3.23).—Not less than 0·1% of saponin or a product containing saponin is added (as a protective colloid) to liquids cap-

able of deleteriously affecting animal fibres. This use of saponin is applicable in the dyeing of wool with vat dyes from an alkaline hyposulphite vat, and in the carbonisation and chroming of wool.

A. J. Hall.

Production of colour-tone and coloured effects on vegetable fibres. I. G. Farbenind. A.-G., Assees. of G. Ruddiff (G.P. 428,039, 23.9.23; Addn. to 348,530; cf. E.P. 173,313, B., 1922, 139 A).—Fabric woven from untreated yarn and yarn which has been mordanted with sulphurised phenols or their homologues and substitution derivatives, or condensation products of such phenols and aldehydes, or products obtained by treating sulphurised phenols with sulphites, is piece dyed. Alternatively, fabric woven from untreated and mordanted (with sulphurised phenols) cotton yarns is again mordanted in the piece with sulphurised phenols and then dyed with basic dyes, the twice-mordanted yarn then appearing darker in shade.

A. J. Hall.

Dyeing cellulose esters, particularly cellulose acetate silk. I. G. Farbenind. A.-G., Assees. of K. H. Meyer and H. Hopff (G.P. 428,176, 1.3.24).—Cellulose acetate silk is dyed in shades fast to washing by means of slightly water-soluble, feebly basic nitroarylamines or their derivatives. Suitable orange-yellow, greenish-yellow, reddish-yellow, and yellow dyes are 8-nitro-2-naphthylamine, 3-nitro-4-aminobenzophenone, 4-nitro-2-aminodiphenylamine, and the condensation product of 3-chloro-6-nitroaniline and formaldehyde. A. J. Hall.

Dyeing cellulose silks, e.g., viscose silks. E. O. Sanner (G.P. 428,263, 27.4.23).—Fabric consisting wholly or party of viscose silk is boiled in a solution containing 6% of Marseilles soap and 5% of an alcoholic soap solution (prepared by saponification of a vegetable fat in alcohol containing sodium carbonate), then bleached, hydro-extracted, and dyed in a boiling dyeliquor containing 6% of the alcoholic soap solution described above, and 2—3% of 85% formic acid added to the cold dye bath. The dyed material is withdrawn, hydro-extracted, and dried. The resulting fabric has a handle and lustre similar to those of natural silk. A. J. Hall.

Dyeing fast shades on animal fibres. I. G. FARBENIND. A.-G., Assees. of H. KRZIKALLA (G.P. 428,238, 24.6.23).—Shades fast to light and washing are obtained by dyeing wool in the usual manner for acid dyes with dyes produced by coupling arylides of 2:3-hydroxynaphthoic acid with diazotised aromatic compounds containing a sulphonic acid group. For example, a fast red dye is obtained from diazotised 2-chloro-5-toluidine-4-sulphonic acid and 2:3-hydroxynaphthoic acid. The dyeings are after-chromed when the dyes used contain suitable groups, e.g. the dye obtained from 6-nitro-2-aminophenol-4-sulphonic acid and 2:3-hydroxynaphthoic acid β-naphthalide.

A. J. HALL.

Manufacture of fabrics [with pattern effects]. C. Dreyfus (E.P. 254,354, 6.2.25).—The process described

in E.P. 249,946 (B., 1926, 532) is applied in the production of pattern effects on fabrics composed wholly or partly of ethers or organic acid esters of cellulose.

D. J. NORMAN.

Production of discharges by means of hyposulphites. O. Kunze (G.P. 426,024, 21.7.23).—Leather, skins, horn, celluloid, paper, and other materials susceptible to the action of heat and hyposulphites are treated with a discharge paste containing a hyposulphite, to which a substance of good heat conductivity may be added, and subjected to the action of acids in the presence of steam or gases at a suitable temperature; the discharge effect is thus completed in a short time at a temperature below 100°, and the material suffers no damage.

A. J. Hall.

Process of decorating material. A. Y. Pearl (U.S.P. 1,590,850, 29.6.26. Appl., 1.8.22).—A mother-of-pearl finish is produced on paper, celluloid, etc. by treating the material with ammonium alum solution, and then, while still moist, with ammonia fumes. The aluminium hydroxide film thus formed when dried is transparent, rendering the material iridescent. A dye may be dissolved in the alum solution. R. B. CLARKE.

Weighting silks. A. Pepper (E.P. 256,479, 25.1.26. Conv., 14.8.25).—See U.S.P. 1,565,390; B., 1926, 123.

Producing multicolour effects on vegetable fibres. I. G. Farbenind. A-G., Asses. of J. Rath and W. Christ (U.S.P. 1,594,853, 3.8.26. Appl., 15.1.25. Conv., 7.2.24).—See E.P. 228,878; B., 1925, 845.

Dyeing apparatus [for hat bodies]. В. Вöнм (Е.Р. 248,359, 16.2.26. Conv., 25.2.25).

Devices for dyeing or similarly treating materials with liquids. C. H. Hartig (E.P. 254,254, 29.12.25. Conv., 27.6.25).

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

Technology of the manufacture of water-glass [sodium silicate]. R. Deckert (Chem.-Ztg., 1926, 50, 535-536).—Sodium silicate is now prepared exclusively in regenerative gas-fired kilns similar to those used in glass manufacture, the working temperature being 1300-1400°. The wear on the furnaces is so heavy that the number in reserve must at least equal that in work. The sand, sodium sulphate, and sodium carbonate used must be perfectly dry, finely divided, and well mixed. The use of a greater proportion of sulphate than of carbonate, while giving a less alkaline silicate, renders the subsequent filtration easier. Examples of proportions recommended are given. The solid silicate, which must be kept free from organic impurities, is dissolved without crushing in rotating drums while steam is injected at 5—6 atm. When the solution reaches  $d \cdot 1.19$  (cold) it is filter-pressed, the clear solution being usually somewhat coloured. Coloration can be minimised by washing the solid silicate with cold water in the drum before dissolving, or removed by filtration through blood charcoal, of which 500 g. are sufficient for the treatment of 10 tons of solid silicate. Sodium silicate solution of  $d \cdot 1 \cdot 7$  is prepared by adding caustic soda to the more dilute solution and concentrating in vacuo. As the concentrated solution cannot be filtered, purity of the initial solutions is essential. The final density reached varies with the proportion of Na<sub>2</sub>O to SiO<sub>2</sub> in the solution. No use has so far been discovered for the filter-press residues from this process. C. Irwin.

Electrolytic preparation of perborate. D. V. STEPANOV (Trans. Karpov Inst. Chem. [Russia], 1925, [4], 98—106; Chem. Abstr., 1926, 20, 2288).—The electrolyte contained 30 g. of borax, 120 g. of anhydrous sodium carbonate, and 1 drop of alizarin and potassium dichromate per litre. For operation in presence of fluoride, which increases the current efficiency at the beginning of electrolysis, but later attacks the platinum anode with consequent catalysis of the decomposition of perborates, alizarin was replaced by naphthenic acids and sulphoacids. The addition of sodium silicate checks the spontaneous decomposition of the perborate. High current density favours the formation of ozone, which does not contribute to the formation of perborate, but accelerates its decomposition, as also does rise of temperature. A current efficiency of 70-80% was attained. Since perborate is removed by crystallisation during the electrolysis, the addition, from time to time, of 0.382 g. of borax, 0.111 g. of sodium carbonate, and 0.167 g. of sodium hydrogen carbonate per litre is necessary.

A. A. ELDRIDGE.

Stability of bleaching powder. D. V. Stepanov (Trans. Karpov Inst. Chem. [Russia], 1925, [4], 107—116; Chem. Abstr., 1926, 20, 2393).—Bleaching powder prepared from lime calcined at 600° is unstable on account of the presence of carbonate, and the product is insufficiently chlorinated; the carbonates decompose at 930°. Calcination above 950° leads to an incompletely chlorinated product on account of the combination with silica. The best results on chlorination are obtained with slaked lime containing 4% of uncombined water. Traces of iron, manganese, nickel, or cobalt catalyse the decomposition of the bleaching powder, but the effect of iron is minimised by calcination of the lime at 950°. The presence of much air in the chlorination chamber is undesirable; the activity and stability of the product are inexplicably increased if the chlorine is first heated in a porcelain tube and then cooled.

A. A. ELDRIDGE.

Testing for the presence of barium chloride in calcium chloride. F. RICHARD (J. Pharm. Chim., 1926, [viii], 4, 49—53).—Calcium chloride solution, slightly acidulated with hydrochloric acid, is treated with a saturated solution of calcium sulphate and heated. On keeping, any barium or strontium present appears as a turbidity or precipitate. They may be distinguished

by treating the calcium chloride solution with a saturated solution of strontium chromate, when barium is precipitated as chromate, and may be weighed. Calcium chloride may be purified from barium by treatment with sulphuric acid, but will then contain traces of sulphate.

B. FULLMAN.

Manufacture of manganese sulphate, sodium nitrite, lead dichromate, and antimony salts. J. MILBAUER [with L. PICK, O. VOGEL, A. MEISL, J. CHLOUPEK, B. SLEMR, and V. JUDENIO (Chem. Listy, 1926, 20, 389-396).-Manganese sulphate may be prepared from pyrolusite and ferrous sulphate by heating a mixture of equal parts of the two substances in a crucible above a layer of anhydrous ferrous sulphate at 700°. If the operation is carried out in an open furnace, serious losses of sulphur trioxide occur. The reduction of sodium nitrate to nitrite is best effected by heating the salt with an excess of 15% of granulated lead. The reaction commences at the m.p. of the nitrate, and, after 2 hrs. at 420°, complete reduction is obtained, but less lead than the theoretical amount is oxidised owing to a certain proportion of the salt being decomposed thermally with the evolution of oxygen. Lead dichromate, prepared by boiling the normal salt with concentrated chromic acid (Preis and Layman, Ber., 1880, 13, 343), may be obtained in a pure dry form by washing the crystals with a mixture of 90% of acetone and 10% of glacial acetic acid. Very finely ground antimony dissolves fairly rapidly in solutions of tartaric acid, alkali tartrates, citric acid, and alkali citrates if a rapid current of air is passed through the solution, and much more slowly in lactic, hydrofluosilicic, and oxalic acids under the same conditions. An analytical method for the determination of nitrites, carbonates, formates, and oxalates in the presence of nitrates is suggested based on the reduction of aqueous solutions of nitrates by water-gas. A. R. POWELL.

Rapid method for the analysis of sulphur chloride. E. Benesch (Chem.-Ztg., 1926, 50, 565).—50 c.c. of distilled water and 10 c.c. of 10% sodium hydroxide are placed in a 500 c.c. conical flask and about 0.5 g. of sulphur chloride, accurately weighed, is added, and a few drops of methyl-orange. The flask is warmed gently for 2—3 min. until all the sulphur chloride is decomposed, i.e., until no oil remains on the bottom of the flask. Slight excess of 10% sulphuric acid is added, the mixture is heated to boiling for 10 min. to agglomerate the precipitated sulphur, a few more drops of methylorange are added, and after cooling, the liquid is diluted to 200 c.c. and 100 c.c. are neutralised with 0.2N-sodium hydroxide, and the chlorine is titrated with 0.1N-silver nitrate solution, using potassium chromate as indicator.

W. G. Carey.

See also A., Sept., 907, Hydrolysis of aqueous solutions of sodium silicates (Harman). 909, Reduction of chromium sesquioxide and uranium dioxide with carbon, and action of nitrogen on uranium carbide (Heusler). 915, Action of nitric acid on metals in presence of catalysts (Palit and

DHAR). 916, Formation of nitric oxide at high temperatures (Briner, Boner, and Rothen); Catalytic decomposition of sodium hypochlorite solutions by finely-divided metal oxides (Chirnoaga); Decomposition of potassium chlorate (Burrows and Brown; Roginski and Schulz; Belenki). 917, Catalytic oxidation of carbon monoxide (Bray and Doss). 918, Catalytic preparation of sulphuric acid (Poliakov). 922, Basic copper sulphates (Fowles). 923, Decomposition of alkaline-earth sulphates (Zawadzki and others). 924, Hydrolysis of sodium silicate (Hägg). 927, Determination of chlorine in perchlorates (Dobroserdov and Erdmann). 928, Determination of perchlorates (Dobroserdov); Determination of sulphurous acid and sulphites (Alsterberg); Reactions for carbonates, hydrogen carbonates, sulphites, and hydrogen sulphites (GASPAR Y ARNAL).

Determination of arsenic. Evers.—See XX.

## PATENTS.

Manufacture of concentrated and fuming sulphuric acid. Kudoh (F.P. 604,636, 29.8.25. Conv., 13.1.25).—A mixture of sulphur dioxide and oxygen is catalysed, the sulphur trioxide is absorbed with sulphuric acid, and the remaining gas is mixed with fresh sulphur dioxide and oxygen and again passed through the contact chamber.

W. G. Carey.

Production of highly concentrated commercial sulphuric acid. Metallbank U. Metallurgische Ges. A.-G. (G.P. 429,835, 1.3.24).—Gases containing sulphur dioxide are brought, in the presence of the necessary moisture and oxygen, into contact with highly concentrated nitrous vitriol, whereby a solution of nitrosylsulphuric acid is formed; from this an amount is abstracted corresponding to the daily output of sulphuric acid and is denitrated by means of gases poor in oxygen and containing more or less sulphur dioxide. The denitration may be assisted by heat, or may be completed by means of a reducing agent, such as hydrogen sulphide.

W. G. Carey.

Manufacture of hydrogen bromide. J. D. RIEDEL A.-G., Assees. of R. HUETER (G.P. 428,225, 11.1.23).—Pressure is maintained above atmospheric in the reaction chamber in the production of hydrogen bromide from its elements by ignition. W. G. CAREY.

Transforming barium and strontium sulphates into other barium and strontium compounds. F. Rothe and H. Brenek (E.P. 242,996, 11.11.25. Conv., 12.11.24).—Barium or strontium sulphate is mixed with silicic acid and heated to about 1200° in a rotary furnace in the presence of steam, in an oxidising atmosphere. The barium or strontium silicate thus obtained may be decomposed by acids to produce barium or strontium salts, and silicic acid which is used again in the process. It is advantageous to use the silicic acid in such proportions that the silicate produced

is of a type between {M<sub>2</sub>SiO<sub>4</sub> and M<sub>3</sub>SiO<sub>5</sub>. Such silicates when decomposed with water yield the alkalineearth hydroxide, and the residue after treatment with water consists chiefly of the alkaline-earth metasilicate which is used again in the process in place of silicic acid.

H. ROYAL-DAWSON.

Manufacture of barium sulphide [and lithopone]. G. Richardson (E.P. 255,167, 20.4.25).—Barium sulphate ground to 100-mesh or finer, is mixed with powdered carbon free from nitrogen, and formed into cylindrical briquettes with hollow cores, which are heated to redness in closed retorts. The barium sulphide produced is dissolved in water, and the solution may be treated with a solution of zinc sulphate to precipitate a mixture of barium sulphate and zinc sulphide (lithopone).

H. ROYAL-DAWSON.

Purification of barium silicates. C. Degude (G.P. 429,169, 9.4.25).—Barium silicate, with excess of silica, or alumina, is comminuted under water and treated with carbon dioxide, whereby the silica or alumina combined with the barium is set free in a gelatinous form, and is then removed from the barium carbonate or carbonate-silicate mixture by known methods, e.g., in the form of soluble akali silicate and aluminate.

A. COUSEN.

Separation of barium chloride from solutions. Rhenania Verein Chem. Fabr. A.-G., Assees. of F. Rüsberg (G.P. 429,716, 6.10.23).—Sodium chloride is added with stirring and heating, and in such amount that a nearly saturated solution of sodium chloride remains.

W. G. Carey.

Catalytic processes and apparatus for reactions between gases [e.g., synthesis of ammonia]. Syn-THETIC AMMONIA & NITRATES, LTD., and F. H. BRAM-Well (E.P. 255,232, 17.7.25).—The gases, e.g., for synthesis of ammonia, are caused to flow first downwards through a mass of the catalyst and then upwards through one or more narrow tubes embedded in the mass. Removal and replacement of the catalyst are facilitated by supporting it on a perforated grid spaced from the closed lower end of the container. The open lower ends of the tubes extend through the grid and their upper ends form part of or are connected with the inner tubes of a heat-exchanger. The gases are supplied to the upper end of the container through the outer tubes of the heat-exchanger. H. HOLMES.

Catalytic synthesis of ammonia. S. G. S. Decker. From H. Harter (E.P. 255,278, 7.10.25. Addn. to 241,771; B., 1926, 12).—The method of combining high- and low-pressure synthesis with transfer of heat from the high-pressure chamber to the other has proved difficult to operate owing to the action of hydrogen on the chamber walls. The method is therefore modified by insulating the catalyst chamber walls, the temperature of which does not exceed 300—400°. Heat transference is then performed in a separate exterior heat-exchanger

or by radiation from the moderately heated highpressure catalyst chamber wall, or by both methods. C. Irwin.

Basic chromic salts and process of making them. H. RÖSSNER and E. RUNNE, ASSTS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,592,961, 20.7.26. Appl., 16.12.25. Conv., 8.11.24).—Solutions of chromic salts, containing per 152 parts by weight of  $\operatorname{Cr}_2\operatorname{O}_3$  not more acid than is equivalent to 140 parts of  $\operatorname{SO}_3$ , are treated in a finely divided condition with a stream of hot gas to obtain readily soluble basic chromic salts. H. MOORE.

Recovering potassium chloride from brine. J. L. Silsbee (U.S.P. 1,593,038, 20.7.26. Appl., 21.1.21).

—A solution nearly saturated with potassium chloride is mixed with sufficient magnesium chloride, in a concentrated form, to cause separation of practically the whole of the potassium chloride.

H. Royal-Dawson.

Solid calcium nitrate. Badische Anilin- & Soda-Fabr. (F.P. 604,116, 3.10.25. Conv., 4.12.24).—A concentrated calcium nitrate solution is applied to a drum heated to 200°.

A. Cousen.

Decomposition of gypsum. Rhenania Ver. Chem. Fabr. (F.P. 604,699, 30.9.25. Conv., 2.12.24).—Gypsum is mixed with silica or silicious material and heated to 1100° in steam and an inert gas. W. A. Caspari.

Manufacture of potassium hydroxide from crude potassium salts. M. Allinger (F.P. 604,702, 1.10.25). —Kainite, sylvinite, or a mixture of alkali sulphates is treated in concentrated solution with oxalic acid, and the precipitated oxalate treated with calcium hydroxide. The oxalic acid is regenerated from the calcium oxalate by means of sulphuric acid and the calcium sulphate is used, with ammonia and carbon dioxide, to prepare ammonium sulphate. A. Cousen.

Manufacture of alkali sulphates and glaserite. F. STEIN (F.P. 604,891, 20.10.25. Conv., 20.10.24 and 2.2.25).—Sodium sulphate or glaserite, potassium chloride, and water are heated together in such proportion that glaserite is formed and simultaneously the solution becomes saturated with potassium chloride and sulphate, so that these salts are precipitated.

W. G. CAREY.

Manufacture of sodium sulphate. W. Mecklenburg (G.P. 430,092, 15.2.25).—The double salt of sodium and ferrous sulphates, prepared in the usual way, is treated with a solution of sodium chloride of suitable concentration.

W. G. Carey.

Recovery of iodides from iodine adsorbed by charcoal. Bouw-Maatschappij Arina (F.P. 604,989, 23.10.25. Conv., 16.4.25).—The charcoal is treated with a solution of alkali sulphite and washed with water.

W. A. CASPARI.

Direct transformation of kieserite into solid Epsom salts without heating. Kali-Ind. A.-G., and

RATIG (G.P. 429,543, 3.2.23).—Kieserite is stirred with magnesium sulphate solution and during the reaction water is added in such amount that the mixture remains capable of being stirred. W. G. CAREY.

Decomposition of magnesium chloride by steam. J. Kersten (G.P. 429,654, 15.11.24).—Carbon is mixed with the magnesium oxychloride in order that the steam passed over the mixture may be decomposed with production of nascent hydrogen. W. G. Carey.

Production of chromate solutions practically free from iron. H. C. Starck, Komm.-Ges. auf Aktien, F. Klaus, and R. Basler (G.P. 429,655, 12.8.23.—A solution of ferrochrome in sulphuric acid is mixed with potassium dichromate and free sulphuric acid, and boiled under atmospheric or higher pressure; if chrome alum is required, alkali sulphates are then added.

W. G. CAREY.

Preparation of alumina and its salts. I. G. Farb-ENIND. A.-G., Assees. of H. Specketer, G. Münch, E. Marburg, and W. Handrich (G.P. 429,717, 24.6.21).— Acid sulphate solutions resulting from the decomposition of clay are treated for the reduction of the ferric iron, and the alumina is precipitated by means of alkalis, or ammonia, ammonium carbonate, or ammonium sulphate with or without sodium bicarbonate, the total alkali being slightly less than the quantity equivalent to the acid present, so that the precipitate consists of a basic aluminium sulphate free from ammonia, approximately 2Al<sub>2</sub>O<sub>3</sub>,SO<sub>3</sub>, which is converted in the usual way into alumina or its salts. In the case of acid chloride solutions, after reduction of the ferric iron, the basic aluminium sulphate is precipitated by adding sulphates, the precipitation being effected at an elevated temperature and under pressure. W. G. CAREY.

Production of phosphorus in the electric furnace. F. Tharaldsen (Nor.P. 40,306, 16.1.23).—Ferrosilicon or silicon carbide is employed as the reducing agent in the preparation of phosphorus from natural phosphates in the electric furnace.

W. G. Carey.

Sulphur burners. E. C. R. Marks. From Grasselli Chemical Co. (E.P. 256,080, 29.9.25).—See U.S.P. 1,566,538; B., 1926, 155.

Producing hydrogen and phosphoric acid. F. G. Liljenroth, Assr. to Phosphorus-Hydrogen Co. (U.S.P. 1,594,372, 3.8.26. Appl., 2.3.23. Conv., 28.8.22).—See G.P. 406,411; B., 1925, 242.

Continuous separation of sulphur from sulphur solutions. E. LEGELER (U.S.P. 1,594,783, 3.8.26. Appl., 1.12.25. Conv., 7.6.25).—See E.P. 249,044; B., 1926, 406.

Burning of gypsum (G.P. 428,576).—See IX.

Effluents from fuller's earth and similar factories (G.P. 428,486).—See XXIII.

## VIII. -GLASS; CERAMICS.

Temperature-sensitiveness of refractory materials in the glass industry. K. Endell and W. Steger (Glastech. Ber., 1926, 4, 43—57; Chem. Zentr., 1926, II, 480).—The authors define temperature-sensitiveness as equal to the quotient of the coefficient of thermal expansion by the torsional power at 500-600°. The thermal expansions of silica bricks and tank blocks, and the heat conductivity of a highly silicious tank block were determined. The tendency to splitting was determined by heating three bricks in a gas furnace upon one side only to 850°, and then plunging into water, the process being repeated until the brick broke. Quartzose bricks split after 1 to 3 quenchings, but some samples withstood over 15 tests. Bricks were temperaturesensitive only below 600°. A. COUSEN.

Thermal expansion of fused silica. W. Souder and P. Hidnert (Sci. Papers U.S. Bur. Standards, 1926, 21, [524], 1—23).—The expansion of 17 samples was determined, measurements being made over the range - 125° to + 1000°. Pointed cylindrical rods, 300 mm. long, were heated in an air- or gas-tube furnace at the higher, and in a bath furnace at the lower, temperatures. In the former case fine weighted wires hung vertically from the ends of the rod through slits in the chamber and movement of the wires was measured; in the bath chamber the wires extended upwards from finger supports beneath the ends of the specimen. Over the range -125° to +20° a minimum value was obtained at temperatures varying from - 50° to - 97°, coefficients at temperatures below the minima ranging from -0.03 to -0.30 ( $\times 10^{-6}$ ), and, above, from +0.12 to +0.29 $(\times 10^{-6})$ . In the higher ranges the following average values were found:  $20-60^\circ$ ,  $0.40\times10^{-6}$ ;  $20-100^\circ$ ,  $0.45\times10^{-6}$ ;  $20-300^\circ$ ,  $0.53\times10^{-6}$ ;  $20-400^\circ$ ,  $0.55\times10^{-6}$ ;  $20-500^\circ$ ,  $0.52\times10^{-6}$ ;  $20-750^\circ$ ,  $0.50\times10^{-6}$ ;  $20-1000^\circ$ ,  $0.48\times10^{-6}$ . The coefficient of transparent samples was slightly larger than that of opaque ones, and rods annealed at 1000° gave slightly smaller values than unannealed samples. A summary of results by other observers is given. A. Cousen.

Physical properties of glasses. Relationship to chemical composition and mode of preparation. W. E. S. Turner (J.C.S., 1926, 2091—2116).

X-Ray examination of aluminium silicates. Ros-BAUD and MARK.—See A., Sept., 889.

### PATENTS.

Protective lenses. E. Busch A.-G. Optische Ind. (G.P. 424,811, 10.1.25).—As a protection against dangerous visible or ultra-violet rays, lenses are coloured by a suitable glaze, whereby the tint can be regulated as required. If desired, only the upper edge, through which the dangerous rays usually enter the eye, may be so treated.

A. Cousen.

Electrofining glass furnace. W. G. CLARK (U.S.P. 1,594,496, 3.8.26. Appl., 6.4.20).—See E.P. 161,192; B., 1922, 711 A.

Annealing glass. A. E. White. From Mississippi Glass Co. (E.P. 256,040, 17.7.25).

## IX.—BUILDING MATERIALS.

Effect of the process of manufacture on the properties of calcined gypsum. L. E. Smith (Rock Products, 1926, 29, 56—59; Chem. Abstr., 1926, 20, 2401).—A study of the effect of the time and temperature of calcination of gypsum on the tensile strength of admixtures with sand (1:3); maximal values are 195 min. and 197°. The plasticity of the mixtures increases with the fineness of the gypsum and with the hemihydrate content; it is decreased by artificial anhydrite and by silica (5%). The properties of the gypsum are not seriously affected by the presence of calcium carbonate (5%), magnesium carbonate (5%), ferric oxide (2%), magnesium sulphate (3%), sodium chloride (if < 0.125%), or sodium sulphate (if < 0.5%).

A. A. ELDRIDGE.

See also A., Sept., 899, Setting of plaster of Paris (Neville). 923, Microchemical examination of gypsum (Budnikov).

## PATENTS.

Binding material for bricks. A. F. Fabre (F.P. 603,549, 4.8.25).—A bond for all kinds of bricks consists of a mixture of powdered gypsum and natural or artificial puzzuolana, to which sand may also be added, treated with solutions of caustic soda, potash, or baryta. Double sulphates of calcium and sodium, potassium, or barium, together with calcium or barium silicate, are formed, and setting follows with great rapidity.

A. Cousen.

Burning of gypsum. Maschinenbau-Anstalt U. Dampfkesselfabr. A.-G. Darmstadt, vorm. Venuleth & Ellenberger, Göhrig & Leuchs, and A. Steinbrückner (G.P. 428,576, 21.6.24).—Gypsum and similar material is burned in a rotary kiln in which the heating gases, obtained from liquid fuel, are blown with a spiral motion opposed to that of the kiln. For this purpose the kiln is provided with a conical ignition and combustion chamber to the inner wall of which are attached screw-shaped ribs.

A. Cousen.

Refractory heat insulating material. A. J. H. HADDON. From CELITE Co. (E.P. 256,021, 27.6.25).—See U.S.P. 1,544,433; B. 1925, 718.

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

Agglomeration and briquetting of finely divided ferriferous material. M. Ottolenghi (Annali Chim. Appl., 1926, 16, 237—268).—Descriptions of the various methods used in the working up of finely divided iron ores and residues are given including briquetting with and without binders, and sintering. Agglomeration can be performed by fixed or rotating furnaces, with hot or

compressed air, and by roasting. A new process devised by Piccinini is described, which is a modification of the Huntington-Heberlein method, and consists in heating the fine material admixed with coal to incandescence. Hot air is then blown through the mass from below through channels provided for the purpose. A hard solid porous block is obtained which is easily broken up and in very suitable condition for treatment in the blast furnace.

S. B. TALLANTYRE.

Effect of occluded hydrogen on the tensile strength of iron. L. B. Pfeil (Proc. Roy. Soc., 1926, A, 112, 182-195).—Tests were made on iron in the normal finely crystalline condition, on single crystals, and on the boundary between large crystals. Electrolytic pickling was used and the tests were made with the specimen actually immersed in the acid. The variables studied were the strength of the acid electrolyte and the electrolysing current, the time the current passed before beginning the loading, the diameter of the test-pieces, the rate of loading, and the temperature at which the tests were made. The influence of temperature is considerable, the decrease of tensile strength being less marked at temperatures slightly above air temperature. Occluded hydrogen has a considerable weakening effect on the intercrystalline boundaries both of large and small crystals, and decreases the cohesion across the cubic cleavage planes. In a single crystal, the tensile strength is hardly affected, but the appearance of the fracture is entirely altered. Experiments with the finely crystalline aggregate showed that the effect of the hydrogen only persists for a very short time, and unless the pickling is continued up to the actual breaking of the specimens, the effect is scarcely apparent. This indicates how limited in value is the tensile test for the investigation of failures in iron and steel suspected to be due to occluded hydrogen.

L. L. BIRCUMSHAW.

Determination of oxygen in iron. P. OBERHOFFER, J. KEUTMANN, W. HESSENBRUCH, and E. AMMON (Stahl u. Eisen, 1926, 46, 1045—1049; cf. B., 1925, 993).—A.—By the hydrogen process. Improvements in the apparatus (cf. loc. cit.) are described, including the substitution of a Silit for the chrome-nickel furnace. The average of a large number of experiments shows that iron in the form of turnings contains 0.024% more oxygen than when in small pieces. The method in its limited sphere of application (absence of carbon and silica) is considered trustworthy. Contrary to the statement of Chaudron and Blanc (B., 1923, 58 A), higher amounts of manganous oxide than 0.2% can be reduced if sufficient time is taken. B.—By the hot-extraction process. An arrangement has been devised by which the gases can be drawn off in stages and the reduction followed more closely. The manganese-carbon alloy containing 4.2% C and 0.45% Mn is now used exclusively in 20-30 g. amounts for each determination. Good results are always obtained if oxides of iron only are present, but with manganous oxide and silica the reduction is incomplete. This is the case with samples deoxidised with manganese and silicon. The method in its present form gives results for the state of deoxidation which compare well with those obtained by the hydrogen process and are of the order expected when silica is present. The presence of oxygen in a steel is indicated by small areas of hardness, tendency to hard fissures, besides bad rolling and forging properties. C.—By the residue process. Except with synthetic iron-oxygen alloys, the bromine process (cf. loc. cit.) has not fulfilled expectations. If manganese is present the results do not show agreement. Morfitt's process (J. pr. Chem., 1854, 61, 33) gives low results for ferrous iron. The modified iron chloride process of Troilus (Jernk. Ann., 1884, 39, 432) is superior, but does not allow of the determination of manganese. The improved iodine process of Eggertz (Polytechn. J., 1868, 188, 119) is more promising. The results of these processes for the determination of silica show satisfactory agreement with those of the chlorination process. It is impossible to determine iron at the same time as silica by the latter process, the values found decreasing with rising temperature. A series of analyses of poor and good tool steels by the recently described residue process, using bromine, is described. The results show good qualitative agreement. Curves are given showing the effects of lower temperatures and lower bromine concentration on the results. A. COULTHARD.

Nature of the A1 transformation in carbon steels. K. Hond (Anniv. Vol. H. Nagaoka, 1925, 95—106; Chem. Abstr., 1926, 20, 2136).—X-Ray analysis has established the author's theory that the A1 transformation in seel consists of the stepped change: austenite  $\rightleftharpoons$  martensite  $\Rightarrow$  pearlite or, more exactly, austenite  $\rightleftharpoons$  martensite is equivalent to the A3 transformation except for the presence of dissolved carbon. It is considered that in other alloys stepped transformations will take place for all eutectoid changes. The eutectoid ransformation in aluminium bronze at 580° and the ageing of duralumin are specifically discussed from this point of view.

A. A. Eldridge.

Rapid volumetric determination of large quantities of manganese in technical iron alloys. T. HECZKO Z. anal. Chem., 1926, 68, 433—461).—The method deends on the oxidation of manganese to the tervalent fom by heating the phosphoric acid solution of the alloy with permonophosphoric acid (cf. B., 1925, 430). The pproximate manganese content is first determined b a rough test; 0.275 g. of alloy is dissolved in 7 c.c. of 8% phosphoric acid and the hot solution is treated with 2g. of sodium pyrophosphate and 25 c.c. of permonophosporic acid solution and, after boiling for 1—3 min., coold, and titrated till colourless with 0.1Mthiosulphate slution. The true manganese figure is about 103% of the result obtained. For the final test, 0.55 g. is dissoled in 14 c.c. of 83% phosphoric acid; the hot solution, diluted with 25 c.c. of water or, if the alloy contains les than 10% Mn, with 15 c.c. of water and 10 c.c. of 0 M-manganese sulphate solution, and boiling continue with the addition of ammonium persulphate until lecomposition is complete and the

added water expelled. The solution is treated with hydrogen peroxide until it becomes colourless, then with a crystal of ferrous ammonium sulphate to remove excess of the peroxide, 4 g. of sodium pyrophosphate and 6 c.c. more than the theoretical quantity of permonophosphoric acid (calculated from the rough test) are added, and the solution is heated gently over a small flame for 3 min. to decompose excess of the oxidising agent. After cooling and diluting, potassium iodide is added and the liberated iodine titrated with thiosulphate. The permonophosphoric acid is made by mixing 55 g. of phosphorus pentoxide with 12 c.c. of perhydrol in a beaker immersed in ice and diluting to 1 litre; 1 c.c. of the solution oxidises 1% of manganese using 0.55 g. of alloy. Chromium, when less than about 1%, is determined simultaneously with the manganese; larger amounts give erratic results. Much cobalt interferes with the colour of the end-point, and vanadium leads to high results if more than a small proportion is present. For good results for manganese exact adherence to the above directions is essential.

A. R. POWELL.

Palladium alloys in jewellery and their detection. F. Durdik (Chem. Listy, 1926, 20, 406—407).—Palladium is often one of the constituents of the white gold alloys used in jewellery. To detect the palladium in these alloys the metal is rubbed on the usual touchstone and the white streak moistened with a mixture of equal parts of nitric acid ( $d1\cdot 3$ ) and hydrochloric acid ( $d1\cdot 12$ ). The liquid is then absorbed by means of a paper that has been previously soaked in an alcoholic solution of benzoylmethylglyoxime and dried; a light yellow spot on the paper indicates the presence of palladium (cf. Hanus, Jilek, and Lukas, A., 1926, 141). A. R. POWELL.

Constitution and age-hardening of some ternary and quaternary alloys of aluminium containing nickel. (Miss) K. E. BINGHAM (Inst. Metals, Sept., 1926. Advance copy, 17 pp.; cf. B., 1923, 358 A).— The alloys of aluminium with magnesium and silicon possess the property of age-hardening after quenching from a high temperature, as do those of copper with aluminium to a less degree. The ternary alloys of aluminium with copper and nickel do not show this property. This is explained by the constitution of these alloys. Age-hardening is caused by the precipitation of CuAl, the solubility of which, both at high and low temperatures, is increased by the addition of 2% of nickel. Age-hardening occurs in these alloys when 1% of magnesium is added, due to the precipitation of Mg2Si, the silicon being present as an impurity in the C. J. SMITHELLS. aluminium.

Influence of gases on copper at high temperatures. I. A. G. Lobley and D. Jepson (J. Inst. Metals, 1926, 35, 213—219).—See B., 1926, 279.

Hardness of cold-rolled copper. S. L. Hoyt (J. Inst. Metals, 1926, 35, 231—257).—See B., 1926, 325.

Soft soldering of copper. T. B. Crow (J. Inst. Metals, 1926, 35, 55—70).—See B., 1926, 325.

Determination of zinc oxide in brass. B. S. Evans and H. F. Richards (J. Inst. Metals, 1926, 35, 173—180).—See B., 1926, 279.

Brittle ranges of bronze. W. L. Kent (J. Inst. Metals, 1926, 35, 45—53).—See B., 1926, 326.

Mechanical properties at high temperatures of an alloy of nickel and copper. H. J. Tapsell and J. Bradley (J. Inst. Metals, 1926, 35, 75—105).—See B., 1926, 280.

Copper-rich aluminium-copper-tin alloys. D. STOCKDALE (J. Inst. Metals, 1926, 35, 181—212).—See B., 1926, 279.

Die-casting of aluminium alloys. G. MORTIMER (J. Inst. Metals, 1926, 35, 371—405).—See B., 1926, 326.

Corrosion of an ancient tin specimen. C. O. Bannister (J. Inst. Metals, 1926, 35, 71—74).—See B., 1926, 327.

Constitution of the alloys of silver and tin. A. J. Murphy (J. Inst. Metals, 1926, 35, 107—129).—See B., 1926, 278.

Interpretation of macro-structure of cast metals. R. Genders (J. Inst. Metals, 1926, 35, 259—293).—See B., 1926, 327.

Crystal growth in recrystallised cold-worked metals. W. Feitknecht (J. Inst. Metals, 1926, 35, 131—172).—See B., 1926, 366.

Softening of strain-hardened metals and its relation to creep. R. W. Bailey (J. Inst. Metals, 1926, 35, 27—43).—See B., 1926, 366.

Production of single crystals of metals and some of their properties. H. C. H. CARPENTER (J. Inst. Metals, 1926, 35, 409—438).—A lecture delivered on May 19, 1926.

Striation due to working or to corrosion in microscopical metallography. Mode of action of etching reagents. A. M. Portevin (J. Inst. Metals, 1926, 35, 363—370).—See B., 1926, 327.

See also A., Sept., 893, Thermal study of electrolytic lead (Trayers and Houot). 895, Diffusion of solids (Henry). 896, Relation between colour and structure of alloys (Chikashige and others); Separation of supersaturated mixed crystals (Fraenkel, Schaller, and Quincke); Formation of deformation twins in eutectics (Vogel); Influence of small additions of lead to gold (Nowack); Behaviour of platinum metals towards silver and gold during cupellation (Truthe); Limits of resistance of tungsten-molybdenum mixed crystals (Van Liempt). 897, Change of volume of cast iron on solidification and diagram of system iron-carbon (Honda and Endo). 908, Recurrent transition curves

in anisotropic binary systems (Wever, Giani, and Reinecken). 909, System iron-silicon-chromium (Denecke). 926, Action of solutions of salts on ferrous metals (Girard). 931, Device for arcwelding easily oxidisable wires (Coats).

Corrosion of iron water mains. Hickethier.—See XXIII.

### PATENTS.

Process of making alloy steel. A. Kissock (Re-issue 16,396, 27.7.26, of U.S.P. 1,300,279, 15.4.19. Appl., 4.6.26).—See B., 1919, 504 A. The process is applicable to the production of molybdenum steel, the molybdenum being introduced as calcium molybdate.

Heat-treating magnesium alloy. Z. Jeffres and R. S. Archer, Assrs. to Amer. Magnesium Corp. (U.S.P. 1,592,302, 13.7.26. Appl., 18.7.22).—A supersaturated solid solution of magnesium and aluminium is hardened and otherwise improved by being heated at a temperature of 125—300° for a sufficient period of time to cause precipitation of a hard constituent in a finely-divided state.

S. S. Woolf.

Process of recovering nickel. W. S. LIETHARDT, Assr. to Metal & Thermit Corp. (U.S.P. 1,592306—7, 13.7.26. Appl., 16.7.25).—To a solution of nicke containing also sulphuric acid which is to be subjected to cementation an additional reagent is added, viz., (A) a chloride ion, or (B) a sulphate of a metal not precipitated by iron.

B. M. Venables.

Production of pure alkali metals. Soc. DÉLECTRO-CHIMIE, D'ÉLECTRO-METALLURGIE, ET DES ACIÉRIES ÉLECT. D'UGINE (F.P. 603,825, 27.12.24).—H'droxides, fluorides, cyanides, carbonates, nitrates, or suphates of the alkalis are mixed with iron (preferably reduced iron) and heated, in the highest possible vacuum, above the m.p. of the alkali compound, whereupon the pure alkali metal distils in a yield of 70—80%. Chlorides bromides, and iodides, because of the volatility of the reulting iron compound, are unsuitable.

A COUSEN.

Manufacture of chromium. J. H. Beaumont. From Metal Research Corp. (E.P. 256,43314.10.25).—See U.S.P. 1,581,698; B., 1926, 548.

Applying protective layers on metls or other electric conductors. J. H. Melloist (U.S.P. 1,595,675, 10.8.26. Appl., 1.8.24. Con., 23.8.23).—See E.P. 220,944; B., 1925, 886.

Heat treatment of metal castins. H. Baron. From E. Will (E.P. 239,218, 27.8.25).

Construction of open-hearth furnce walls. Open-Hearth Combustion Co., Assees. of § Naismith (E.P. 242,607, 13.10.25. Conv., 10.11.24).

Furnaces [for reheating etc.] A. SMALLWOOD and J. FALLON (E.P. 255,938, 29.4.2).

## XI.—ELECTROTECHNICS.

Graphic representation of electrolytic data.

R. Nitzschmann (Chem.-Ztg., 1926, 50, 525).—It is shown how charts can be drawn up from which the relations between voltage, electrochemical equivalent, current efficiency, and energy consumption can be read off. Examples are given for the principal processes of technical electrolysis.

W. A. Caspari.

Dielectric breaking stress of liquids [transformer oils]. P. Bary (Rev. gen. Colloid., 1926, 4, 166-173).-Microscopical examination of spark gaps at 1000— 3000 volts in transformer oils indicates that a darkcoloured filament is formed, under the dielectric stress, between the points. The filament, which is considered to consist of globules of asphalt suspended in the oil and surrounded by envelopes of water, takes a certain time to form in the briskly agitated oil lying in the gap, and a spark passes only when the gap is completely bridged. The material of the filament is then re-dispersed, but forms into line again after a few seconds, allowing another spark to pass. Defective insulating properties are attributed to the presence of asphaltic suspensions having aqueous envelopes, and the superiority of centrifuging to heating as a means of purification is adduced in support. W. A. CASPARI.

See also A., Sept., 892, Temperature scale for tungsten filaments (Jones). 893, Physical properties of tungsten at high temperatures (Zwikker). 913, Electrolysis of chromium trioxide solutions (Müller). 919, Electrochemical preparation of lead arsenates and arsenites (Ormont).

Electrolytic preparation of perborate. Stepanov.—See VII.

Determination of arsenic. Evers.—See XX.

## PATENTS: \_

Packing for electrolytic apparatus for decomposition of water. R. PECHKRANZ (E.P. 237,903, 23.7.25. Conv., 2.8.24).—A resistant insulating packing is made of asbestos of other fibre impregnated with a hydrocarbon pitch such as bitumen from petroleum.

W. A. CASPARI.

Heating coils for electric furnaces. Brit. Thomson-Houston Co., Assees. of W. Keenan (E.P. 245,089, 8.12.25. Conv., 27.12.24).—From the roof of a brick furnace, resistor coils are suspended by pins or hangers mounted in recesses in such a way that expansion or contraction is provided for. W. A. Caspari.

Electric resistance elements for incandescent lamps and thermionic devices. Gen. Electric Co., and C. J. Smithells E.P. 255,195, 26.5.25).—A support for tungsten wire resistances working up to 2200° is made of magnesia with special precautions. The magnesia is sintered at 1800°, finely ground, mixed with

a small quantity of unsintered material, moulded, and heated to 3300° or above, so that the surface is fused.

W. A. CASPARI.

Electrodeposition of organic materials [rubber, etc.] on anodes from aqueous emulsions. C. L. Beal and L. W. Eberlin, Assis. to Eastman Kodak Co. (U.S.P. 1,589,324, 15.6.26. Appl., 7.5.25).—In the application of the process of U.S.P. 1,476,374 (B., 1924, 104), the harmful effects of nascent oxygen liberated at the anode (cf. U.S.P. 1,580,795; B., 1926, 639) can be avoided by forming the anode of, or coating it with, a metal which has a greater affinity for oxygen than the material being deposited. Alternatively, the metal is finely divided and dispersed with a protective colloid such as glue in the emulsion forming the electrolyte. Zinc, cadmium, and magnesium are suitable metals. The metal is oxidised preferentially to the coating, and the oxide is deposited with the latter.

T. S. WHEELER.

Electrodepositing organic material such as rubber upon porous objects of non-conducting material such as fabrics. S. E. Sheppard and C. L. Beal, Assis. to Eastman Kodak Co. (U.S.P. 1,589,325, 15.6.26. Appl., 1.4.26).—The depth and character of the deposit of organic material (rubber) obtained by the process of U.S.P. 1,476,374 (B., 1924, 104) upon a porous non-conducting fabric (cotton) may be controlled by first treating the material with a size containing a coagulant. Suitable sizes are starch, glue, or dextrin or similar hydrophile colloids adjusted to p<sub>H</sub> 3-7 by an organic acid which acts as a coagulant. To ensure an even deposit the fabric is passed first through an alkaline degreasing bath containing a 5°/o solution of sodium carbonate, phosphate, or the like and then, after washing, led directly into the size. The sized fabric is then led around an anode formed of a roller of conducting material immersed in the electrolytic emulsion.

T. S. WHEELER.

Electrodeposition of organic materials such as rubber and cellulose compounds. S. E. Sheppard and C. L. Beal, Assis. to Eastman Kodak Co. (U.S.P. 1,589,326, 15.6.26. Appl., 22.4.25).—The deposits of organic materials on conducting surfaces obtained by the method of U.S.P. 1,476,374 (B., 1924, 104) are rendered more uniform by coating the surface of the conductor with a 3—5°/<sub>o</sub> aqueous solution of gelatin or glue, or with a dilute rubber emulsion, with which is preferably incorporated a hygroscopic substance, such as glycerol or calcium chloride, to inhibit hardening of the coating on storage. The coating forms a diffusion path for the current at the surface. T. S. Wheeler.

Electrodeposition of coatings of cellulosic compounds. Aqueous emulsions of electrodepositable cellulosic compounds and coalescing agents therefor. L. W. EBERLIN and C. L. BEAL, ASSIS. to EASTMAN KODAK CO. (U.S.P. 1,589,327—8, 15.6.26. Appl., 7.4.25).—The process of U.S.P. 1,476,374 (B., 1924, 104) can be applied to the electrodeposition of a cellulose

compound by dissolving the latter in a liquid which also functions as a coalescing agent and emulsifying the mixture in an aqueous solution with an emulsifier and a protective colloid. On passing a current through the mixture the cellulose compound is deposited on the conducting anode, the coalescing agent binding the deposited particles together. For example, cellulose nitrate or acetate, or a mixture of these compounds (500 pts.) dissolved in amyl acetate (1500 pts.), is emulsified with water (10,000 pts.) in a colloid mill in presence of an emulsifier, such as Turkey-red oil (150 pts.), and a protective colloid, e.g., gum arabic (10 pts.). In the deposition a voltage of 110, and a current density of \( \frac{1}{3} \) amp. per sq. in. of anode surface, is used for 4 min. If the substance to be coated is non-conducting, it is coated with a conductor such as graphite, or if it is porous, e.g., wood, it is saturated with an aqueous solution of an electrolyte. T. S. WHEELER.

Electrodepositing rubber upon a metal wire. S. E. Sheppard and L. W. Eberlin, Assis. to Eastman Kodak Co. (U.S.P. 1,589,329, 15.6.26. Appl., 20.11.25).

—A metal wire is passed through a bath of the type described in U.S.P. 1,476,374 (B., 1924, 104) containing an aqueous emulsion of rubber, soap, sulphonated castor oil, and sulphur and if desired, aniline sulphate, and is thus coated with a mixture of rubber and sulphur. It is then led through a heater in which the rubber coating is partially dried, and after being passed between rollers which exert a firm uniform pressure on it and remove all inequalities in the coating it is led through a second heater in which drying is completed and the coating is vulcanised.

T. S. Wheeler.

Aqueous emulsion containing electrodepositable rubber and a cellulose compound. Electrodeposition of coatings comprising rubber and a cellulosic compound. S. E. Sheppard and L. W. Eberlin, Assisto Eastman Kodar Co. (U.S.P. 1,589,330 and 1,589,332 15.6.26. Appl., 7.4.25).—An aqueous rubber emulsion of the type described in U.S.P. 1,476,374 (B., 1924, 104) and 1,589,331 (following), mixed with an emulsion of a cellulose compound prepared as described in U.S.P. 1,589,327—8 (preceding), is applied to the deposition of a mixture of rubber and a cellulose compound.

T. S. WHEELER.

Aqueous emulsion of unvulcanised rubber and sulphur. S. E. Sheppard and L. W. Eberlin, Assis. to Eastman Kodak Co. (U.S.P. 1,589,331, 15.6.26. Appl., 7.4.25).—A solution for use in the electrodeposition of rubber is formed from alkaline rubber latex, and sulphur emulsified with a protective colloid, such as glue or gum arabic. Accelerators, pigments, and fillers may be dispersed in the emulsion. The coating obtained is vulcanised after deposition. A suitable emulsion is formed from 4000 c.c. of 30% rubber latex and 1000 c.c. of a 20% emulsion of sulphur in 0.5% glue solution.

T. S. Wheeler.

Utilisation of exhausted primary batteries. Chem. Fabr. Johannisthal, and Scheller (G.P.

428,267, 11.5.22).—The manganese-containing material from exhausted Leclanché cells is ignited in absence of air and the reduced zinc is removed. By treatment with dilute acid the graphite is separated, and the manganous oxide is then re-oxidised.

W. A. CASPARI.

Recovery of lead compounds from old accumulators. F. Fessia (G.P. 428,365, 27.2.23. Conv., 27.2.22).—The gratings of positive electrodes are freed from filling and used over again. Old fillings from positive and negative electrodes are subjected to heat treatment so as to produce a mixture of lead oxide and sulphate, which is applied to the filling of fresh electrodes.

W. A. Caspari.

Temperature-controlling devices for electric furnaces. Brit. Thomson-Houston Co., Ltd., and S. Payman (E.P. 255,977, 11.5.25).

Phosphorus (Nor. P. 40,306).—See VII.

Purification of fats (F.P. 606,079).—See XII.

# XII.-FATS; OILS; WAXES.

Crystalline bromides from linseed and other drying oils. H. Toms (Analyst, 1926, 51, 387—391; cf. B., 1924, 264).—The bromination products of perilla, candlenut, and Para rubber seed oils and the hexabromostearic acids isolated therefrom are identical with those obtained from linseed oil. The crystalline bromide from linseed oil (m.p. 156°) is not decomposed by prolonged boiling with ethyl acetate. Tetralin and acetone form a better crystallisation medium than ethyl acetate. Hexabromostearic acid was found on hydrolysing the bromoglyceride with 48% hydrobromic acid.

D. G. Hewer.

Fixed oil of the seeds of "Kurrajong" (Brachychiton populneum. B.Br; syn. Sterculia diversifoia, G. Don). F. R. Morrison (J. Proc. Roy. Soc. N.S.W., 1926, 59, 267—275).—Two samples of seeds collected in 1924 and 1925 yielded, by ether extraction in a Soxhlet apparatus, 23·4% and 23·9% of oil, respectively, having m.p. 30°, below 15°;  $d^{30}_{30}$ 0·9083,  $d^{15}_{15}$ 0·9203;  $n^{20}$ 1·4676, 1·4709; acid value 65·0, 42·7; saponif. value, 198·0, 192·8; iodine value (Wijs, 2 hrs.), 101·3, 107·6, unsaponifiable matter (sitosterol), 1·1%, 1·5%. The fatty acids from the 1924 sample had m.p. 33·5°,  $d^{15}_{15}$ 0·8908,  $n^{40}$ 1·4548, iodine value (Wijs, 2 hrs.) 100·9, neutralisation value 202·4, mean mol. wt. 277, and consisted of palmitic, oleic, and linoleic acids. Alkaloids were absent from the oil.

Constants of flax wax. W. Honneyman (Pharm. J., 1926, 117, 157—159).—Flax wax occurs principally in the fibre and cortical tissues of the plant, the air-dried cortex containing as much as 10%. Samples of wax from flaxes grown under different climatic conditions are shown to have practically identical characters. The analytical constants of flax wax and hemp wax are, respectively: saponif. value, 73·4—83·7, 101; iodine

value (Hanus, 2 hrs.),  $21 \cdot 6 - 28 \cdot 8$ ,  $22 \cdot 5$ ; acid value,  $17 \cdot 5 - 23 \cdot 8$ ,  $13 \cdot 6$ ;  $d0 \cdot 963 - 0 \cdot 985$ ,  $0 \cdot 977$ ; m.p.  $67 \cdot 3 - 69 \cdot 5^{\circ}$ ,  $69 \cdot 1^{\circ}$ . C. O. Harvey.

Critical points of emulsification in oil-soap emulsions. E. V. Kyser and F. C. Vilbrandt (J. Amer. Pharm. Assoc., 1925, 14, 392—398; Chem. Abstr., 1926, 20, 2391).—Cottonseed oil, oleic acid, and various alkalis were employed. Small quantities of sodium or potassium carbonate produce permanent emulsions, separation being less likely or slower than if sodium hydroxide or soluble silicate is used. Sodium and potassium hydroxides are undesirable emulsifying agents. The viscosity of all the emulsions lay between 1 and 51·5, that of the oil used.

A. A. Eldridge.

Change of refractive index of linseed oil during drying. LAURIE.—See XIII.

Chaulmoogra oil. Gelarie and Greenbaum.—See XX.

PATENT.

Refining of olive and other edible oils. E. Foray (F.P. 605,389, 17.10.25).—Free fatty acids are neutralised at 50° by sodium or potassium carbonate, and the oil is then treated with 0.1% of manganese dioxide and heated and stirred.

H. M. Langton.

Purification of fats. G. RAVINETTI (F.P. 606,079, 9.11.25. Conv., 18.11.24).—Oxidising gases (oxides of nitrogen) produced by electrical discharges are used, in a finely dispersed condition, shortly after their formation, for the purification of fats.

H. M. LANGTON.

Manufacture of soap. M. I. AISCHE (E.P. 255,508, 17.3.25).—Soaps are made by saponifying soap-yielding materials, in presence of a hydrogenating agent, e.g., an alkali amalgam, or metallic hydrides or alloys-with or without a catalyst, e.g., reduced metals—or metal oxides, salts, or compounds. For example, a mixture of menhaden oil (566 pts.) and whale oil (565 pts.) is heated to 60° and emulsified with water at this temperature, and sodium amalgam in lumps (1348 pts.) is added gradually to the mixture. A vigorous reaction occurs, and after addition of all the amalgam the temperature is raised to 145—160° for a time. When saponification is complete the soap is remelted with addition of water, and salted out as usual; mercury from the amalgam settles out at the bottom of the vessel and is drawn off separately from the saline water and glycerol. H. M. LANGTON.

Presses for expressing oils or other liquids from materials containing the same. E. C. R. Marks. From F. S. Carver (E.P. 256,344, 16.5.25).

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

Analysis of Prussian blue. F. G. A. Enna (J. Soc. Leather Trades Chem., 1926, 10, 172—174).—Moisture—1 g. of the finely powdered substance is dried for 2 hours at 105—110°, cooled, re-weighed, and dried for 1 hour more or till the weight is constant. Prussian blue should not contain more than 2.5—3% of moisture.

Total iron—0.25 g. is treated for 15 min. with cold strong sulphuric acid, then heated over a very small flame for hour until the acid just fumes. When the mass is white-not grey-it is rinsed into a flask with cold distilled water and heated until a clear solution is obtained. After filtering through a tared filter dried at 110°, any insoluble matter is washed, dried, and weighed. The filtrate is acidified with strong sulphuric acid, treated with pure granulated zinc, heated till colourless and no coloration is given with ammonium thiocyanate, diluted to 250 c.c. with freshly boiled distilled water, cooled, and made up to 250 c.c. 100 c.c. are titrated with 0.1N-permanganate; 1 c.c. = 0.012128 g. Fe<sub>7</sub>(CN)<sub>18</sub>. Actual Prussian blue—1 g. of the sample is treated with a hot 5% solution of hydrochloric acid, filtered, and the insoluble matter washed with boiling water till the filtrate gives no coloration with ammonium thiocyanate. The solution is reduced with zinc and sulphuric acid, the iron determined as before, and the result subtracted from the total iron found. The difference is actual Prussian blue. Insoluble impurities-The residue from the total iron determination is weighed and calculated as per cent. of insoluble matter. Soluble impurities is the difference of the sum of the above D. WOODROFFE. determinations from 100.

Change of refractive index of linseed oil in the process of drying and its effect on the deterioration of oil paintings. A. P. LAURIE (Proc. Roy. Soc., 1926, A, 112, 176—181).—Possible causes of the lowering of tone of oil paintings with age are discussed, and this is shown to be due not only to the yellowing of the oil film, but also to a change in the refractive index. Considering pigments as consisting of transparent particles with the property of absorbing certain parts of the spectrum, it is clear that their apparent opacity and brilliancy will depend upon their refractive index and that of the medium with which they are mixed. By microscopical examination of a number of pigments in the three media, linseed oil, bromonaphthalene, and methylene iodide saturated with sulphur, it has been found possible to arrange the more important bright pigments in order of opacity. It has also been shown, by painting a film of linseed oil on the glass surface of a refractometer, that a considerable rise in the refractive index takes place during the drying of the oil, followed by a slow, continuous rise, which was followed for some months. As it is known that slow chemical changes, accompanied by an increase in density, continue in linseed oil for years, it is probable that the increase in refractive index will L. L. BIRCUMSHAW. also continue.

Yellowing of oil films and change of refractive index of linseed oil on drying. A. P. Laure (J. Oil and Colour Chem. Assoc., 1926, 9, 163—173; cf. preceding abstract).—White pigments ground in various oil media were allowed to dry on glass and then kept in the dark under a bell jar containing a beaker of water for a period of 8 months, when their colour was compared with freshly prepared duplicates; fairly good matches were obtained with the yellow glasses alone of the Lovibond tintometer. The suggestion that unbleached

oils do not darken, and are therefore suitable for artists' colours, is not justified, as practically the same change of tint was observed in white pigments ground in dark yellow boiled oil and in refined linseed oil. Stand oil yellows appreciably less and walnut oil slightly less than linseed oil. Addition of resin which is not decomposed by heat during the process of dissolving, diminishes the yellowing of linseed oil, e.g., the addition of mastic or dammar, but the reverse is the case for addition of copal varnish where the resin is decomposed.

S. S. WOOLF.

Micrography of oil and varnish films. E. Stern (Kolloid-Z., 1926, 39, 330—334).—An account of the microscopical examination of films of the binding media of paints and varnishes. The action of water on the films is studied. The results are to a considerable extent in agreement with the behaviour of the materials in practice, and it appears possible to develop the microscopical method to render the same service to the study of paints and varnishes that metallography renders to the study of alloys. A number of photomicrographs are given.

N. H. Hartshorne.

Examination of resins. H. Wolff and W. TOELDTE (Farben-Ztg., 1926, 31, 2503—2505; cf. Eibner and others, and Schmidinger, B., 1926, 759, 760).—Tables are given indicating the nature and intensity of the characteristic fluorescences exhibited by various varnish resins in the form of lumps, powder, solutions in butyl acetate, and residue from evaporation of these solutions. In lump form the resins give irregular results, but inferences may be drawn from the fluorescence of powdered resins and from the change in appearance under the ultra-violet lamp on dissolving the resin and on evaporating the solvent. The Albertol resins give the most intense fluorescence (blue), whilst ester gum, lime-hardened resin, and Congo copal, in decreasing intensity, show light blue fluorescence. A marked increase in intensity is noticed when the acidity of resin is neutralised either by glycerol (ester gum) or by lime (lime-hardened resin). Combination of microscopical examination (Stock, B., 1925, 770), capillary analysis (Stock, B., 1926, 679), and fluorescence analysis will distinguish resins hitherto unclassifiable.

S. S. WOOLF.

Storch-Morawski (Liebermann) reaction for detection of resin (abietic acid) or resin compounds in paints, etc. M. SCHULZ and F. KRÄMER (Farben-Ztg., 1926, 31, 2556—2558).—Earlier statements to the effect that in the Storch-Morawski test for resin, a negative reaction alone is conclusive, while a positive reaction may possibly be due to constituents other than resin, are contradicted. Specifying a positive reaction to be the immediate formation of a violet coloration at least as deep as the tint of a 0.001N-solution of potassium permanganate, with rapid change to dirty brown or green colours, the authors declare that only in the presence of resin are these conditions fulfilled on applying the test. Erroneous deductions owing to the presence of phytosterol or of hydroxy-fatty acids, which give rise to colorations similar to those due to resin, are excluded

by the above definition since the colour produced by the former is not the distinct violet required, while the latter retains a blue-violet colour for a too long period to be confused with the swiftly disappearing resin effect. As, however, it was found that oxidised resin does not give the reaction, a negative reaction does not necessarily prove the absence of resin initially. In using the gelation of abietic acid in benzene solution on addition of ammonia as a confirmatory test for resin, it must be borne in mind that fatty acids under this test give a similar although slightly less firm gel. S. S. Woolf.

Computation of colorimetric purity. I. G. Priest (J. Opt. Soc. Amer., 1926, 13, 123—132). D. B. Judd (*Ibid.*, 133—154).

Reaction of "aluminon" with hydroxides of beryllium, rare earths, zirconium, and thorium. MIDDLETON.—See A., Sept., 930.

### PATENTS.

Stable pigment colours. W. EBERLEIN, and COLLOISIL COLOUR CO. (E.P. 254,887, 16.1 and 30.4.25).—Pigments, e.g., primrose lead chromate, Prussian blue, that are of unstable nature when precipitated alone, are stabilised by mixing one or both of the components before precipitation with an organic or inorganic emulsion, colloidal solution or suspension, etc., or by precipitating or flocculating one of them with a fixing earth or clay. Typical stabilisers mentioned are bentonite, water-glass, and sodium-resin soap. S. S. Woolf.

Producing carbon black for use in the manufacture of rubber and rubber goods. H. H. Ward (E.P. 255,164, 20.4.25).—A mixture of finely-divided peat, cork, coal tar pitch, coal tar, and paraffin oil is formed into blocks and subjected to a regulated current of air in an externally heated furnace, e.g., at 315°; the carbon black is collected by passing the resulting fumes through a series of chambers into which steam is injected. If no paraffin oil is used, the mixture is carbonised in the absence of air, the resulting charcoal or carbon being subsequently removed from the furnace and cooled in air-tight vessels.

D. F. Twiss.

Vehicle for colouring matters and graphite.
V. Lange, Assee. of H. Lange (E.P. 230,858, 16.3.25.
Conv., 14.3.24).—A substitute for glue-media is obtained
by boiling an aqueous solution of sugar, molasses, or
syrup with a solution of asphaltum, gutta-percha, or
similar product in benzine, benzol, etc., and removing
the scum.
S. S. Woolf.

Lacquers, impregnating materials, and the like. Farbenfabr. vorm. F. Bayer & Co. (E.P. 243,722, 23.11.25. Conv., 26.11.24).—The stability of solutions of cellulose acetate in glycerol- and other monochlorohydrins is improved by the addition of small quantities of organic bases, e.g., carbamide or its derivatives, aliphatic amino-acids, aniline, pyridine, etc. A typical mixture is 10 pts. of cellulose acetate, 65 pts. of acetone, 20 pts. of ethylene chlorohydrin, 5 pts. of monchlorohydrin, and 0.5 pt. of glycine.

S. S. WOOLF.

Resinous bodies from a phenol, furfural, and other aldehyde. C. Ellis (U.S.P. 1,592,296, 13.7.26. Appl., 11.9.22. Renewed, 30.1.24).—Acetaldehyde is condensed with excess of a phenol in the presence of an acid catalyst. The readily fusible resin so formed is treated with sufficient furfuraldehyde to react with the excess of phenol, in the presence of a mild fixed alkali catalyst.

S. S. Woolf.

Dehydrating pine oil. R. C. Palmer, Assr. to Newport Co. (U.S.P. 1,593,030, 20.7.26. Appl., 24.8.22).

—Treatment of pine oil with a dehydrating agent yields a product more closely resembling turpentine than the original oil.

S. S. Woolf.

Manufacture of artificial resins. A. Regal (E.P. 256,394, 29.7.25).—See U.S.P. 1,584,473; B., 1926, 596

Lithopone (E.P. 255,167).—See VII.

## XIV.-INDIA-RUBBER; GUTTA-PERCHA.

Rubber—a fibrous material. F. Kirchhof (Kautschuk, 1926, 151—156).—Unvulcanised rubber which has been strongly stretched and then frozen in ice water resembles wool or "viscose" in its behaviour when further stretched; the stress-strain curve approaches the stretch-axis with increase in the degree of extension previous to freezing, or with more intense freezing. Relative to wool, the ultimate strength of the frozen stretched rubber is about one-tenth and its extension at break about three times as great. More intense freezing, e.g., in liquid air, gives a product of greater strength. Both the crystalline portion of stretched rubber and the amorphous constituent are very sensitive to change of temperature.

D. F. Twiss.

Aggregation and re-aggregation of raw rubber in the presence of other substances. M. Kröger (Gummi-Ztg., 1926, 40, 2429—2430; cf. B., 1926, 137).— The re-aggregation of raw rubber in storage is less marked in samples with a higher content of natural "resin." The presence of a small proportion of a good carbon black reduces the rate of re-aggregation but a larger proportion accelerates it. Piperidine has a marked accelerating effect on the re-aggregation, whereas magnesium oxide retards it.

D. F. Twiss.

Super-accelerators [for vulcanisation of rubber]. W. J. S. NAUNTON (Trans. Inst. Rubber Ind., 1926, 2, 18—41).—A review of the present state of knowledge with respect to super-accelerators. These can be regarded as including salts (metal or organic) and disulphides of various dithio-acids. The metal salts are those of zinc or lead. Of the organic salts at least two find commercial use, viz., diethylammonium diethyldithiocarbonate, and piperidine piperidinecarbothionolate; the former of these is more active in the production of soft rubbers but less active for vulcanite. The diphenylguanidine salts of diethyldithiocarbonic acid, a-dithionaphthoic acid (salt, m.p. 154°), isopropylxanthic acid (salt, m.p. 105°), and mercaptobenzothiazole (salt, m.p. 172°), during vulcanisation in the presence of zinc oxide give the same results as a mixture of the free base with the zinc salt of the

acid; with the third compound and corresponding mixture the effect is surprisingly less than that of an equivalent amount of zinc isopropylxanthate. Superaccelerators are not affected so adversely as less active substances, e.g., diphenylguanidine, by the presence of certain other ingredients, e.g., "substitute" or antimony sulphide, in the rubber mixture under vulcanisation.

D. F. Twiss.

Production of coloured latex resistant to vulcanisation. R. Ditmar (Chem.-Ztg., 1926, 50, 528—529).— A number of dyes have been examined as to their suitability for colouring latex which is subsequently to be dried and cold-cured. The following dyes pass a stringent test: yellow, pyramine yellow G extra, stilbene yellow 3 GX; red, sorbine red X, rhodamine B extra; pink, anthosine 5B; orange, cotton orange R; violet, vulcan red BN; blue, oxamine pure blue 6B. From 0.03% to 0.06% upon the latex is required to colour the goods.

W. A. Caspari.

Organic dyes in vulcanised rubber. T. J. Drakely (Trans. Inst. Rubber Ind., 1926, 2, 42—54).— In the presence of diphenylguanidine the accelerating influence of a neutral azo-dye is masked; Bismarck Brown has a more marked effect which, however, is not enhanced by zinc oxide. Organic dyes for introduction into rubber are most conveniently used after deposition on a filler such as clay. The dye should be sufficiently soluble in rubber to ensure even distribution but not so soluble as to cause transfer of the colour to adjacent rubber. The effect of vulcanisation, in moulds and in open steam, on the colour of rubber containing various dyes, is recorded.

D. F. Twiss.

## PATENTS.

Direct production of rubber goods from rubber emulsions. Anode Rubber Co. From P. Klein and A. Szegvári (E.P. 254,765, 13.2.25).—In the production of rubber goods from suspensions such as latex, difficulties may arise from certain features of the additional substances which it is desired to incorporate by previous dispersion in the latex. Such difficulties may be obviated by first forming an intimate mixture or compound of the desired ingredient with a second substance which is capable of masking the undesirable features of the primary substance. Thus liquid hydrocarbons may be satisfactorily dispersed in rubber latex if first impregnated in kieselguhr; lead oxide intimately mixed with kieselguhr gives a more permanent dispersion than lead oxide alone; sulphur may advantageously be introduced as zinc polysulphide or by first heating with kieselguhr, clay, lampblack, or unsaturated oils, and dispersing the powdered product in the latex.

D. F. Twiss.

Accelerator for rubber vulcanisation. A. F. Hardman, Assr. to Kelly Springfield Tire Co. (U.S.P. 1,589,757, 22.6.26. Appl., 24.6.25).—A "master batch" of diphenylguanidine for vulcanisation purposes is produced by submitting a mixture of thiocarbanilide with an excess of metal oxide and a carrier to the action of ammonia under pressure.

D. F. Twiss.

Treatment of rubber with aliphatic diamines and their derivatives. S. M. Cadwell, Assr. to Naugatuck Chemical Co. (U.S.P. 1,592,820, 20.7.26. Appl., 10.7.23).—Rubber is subjected to the reaction product of ethylenediamine and an open-chain aldehyde and is then vulcanised.

D. F. Twiss.

Vulcanisation of rubber. H. O. Chute (U.S.P. 1,593,017, 20.7.26. Appl., 21.12.22).—Rubber is vulcanised with sulphur in the presence of a substance containing the characteristic carbon-nitrogen group present in calcium cyanamide. D. F. Twiss.

Rubber [vulcanisation] accelerator. G. H. Buchanan, Assr. to Amer. Cyanamid Co. (U.S.P. 1,593,385, 20.7.26. Appl., 1.3.26).—A mixture of diphenylguanidine and ditolylguanidine is used for accelerating vulcanisation. D. F. Twiss.

[Rubber] adhesives. B. F. GOODRICH Co., Assees. of W. C. GEER (E.P. 247,136, 9.4.25. Conv., 9.2.25).— Rubber is converted into less chemically unsaturated derivatives of a tough balata-like nature. 73 pts. of p-phenolsulphonic acid or similar product are mixed into 100 pts. of undissolved rubber on a mill, heated in an oven for 4-10 hrs. at 120-145°, the product worked on a warm mill to homogenise it and washed free of remaining reagent and other impurities. The balata-like product may be made by forming the sulphonic compounds in the rubber mass by adding to the raw rubber suitable organic compounds and sulphuric acid and heating the mixture. The balata-like product can be dissolved in benzene or other solvent to form a cement, or mixed with about 10% of an organic flux or softener, e.g., tung oil, castor oil, hard mineral rubber, naphthalene, or Canada balsam, or with a relatively small proportion of a nitrogenous organic age-resister such as the thiocarbamide of dimethyl-p-phenylenediamine, 1:8naphthylenediamine, aldol-α-naphthylamine, or benzidine. A suitable adhesive is prepared by mixing 86 pts. of the balata-like rubber product, 3 pts. of aldol-αnaphthylamine, 1 pt. of benzidine, and 10 pts. of tung D. WOODROFFE.

Electrodeposition of rubber (U.S.P. 1,589,324—6, 1,589,329, and 1,589,331).—See XI.

Electrodeposition of mixtures of rubber and cellulose compounds (U.S.P. 1,589,320 and 1,589,322).
—See XI.

Carbon black (E.P. 255,164).—See XIII.

## XV.-LEATHER; GLUE.

Preparation of isoelectric collagen for tannin assay. L. Meunier and P. Chambard (Rev. gén. Colloid., 1926, 4, 161—165).—Calf skin (250 g.) after treatment with lime to remove keratin, is well washed and agitated for several hours with  $2\frac{1}{2}$  litres of water containing  $1\cdot 5$  g. of acetic acid. After three washings with tap-water and a final one with distilled water, the  $p_{\rm H}$  of the hide is about 7, but there is still some lime

present. The hide is then soaked for six hours in  $2\frac{1}{2}$  litres of distilled water saturated with carbon dioxide, and the operation is repeated (about six times) until the extract no longer gives a reaction with ammonium oxalate. The hide should be preserved in water, which is saturated once a week with carbon dioxide; it contains about 0.1% (dry) of non-alkaline ash. When freed from dissolved carbon dioxide, the hide shows a point of minimum swelling, or isoelectric point, at  $p_{\rm H}$  5.4—5.6. W. A. Caspari.

Determination of the isoelectric point of hide powder by means of complex chromium salts. K. H. Gustavson (J. Soc. Leather Trades Chem., 1926, 10, 203—211).—Portions of hide powder were respectively treated with solutions of acetato-complex chromium salts and formato-chromium complex salts of different  $p_{\rm H}$  values. The  $p_{\rm H}$  value of the exhaust liquor was determined and found to be slightly greater with solutions of  $p_{\rm H} < 5.04$ , and slightly less with solutions of  $p_{\rm H} > 5.04$ . It is inferred that the isoelectric point of hide powder is 5.0, though there is evidence to indicate that it may be a function of pre-treatment with acids and alkalis.

D. Woodpoffe.

Fluorescence of the acetone extract of tanning materials. L. Meunier and A. Jamet (J. Soc. Leather Trades Chem., 1926, 10, 166—168).—Samples of coarsely ground tanning materials or the finely-powdered dry extract obtained by evaporating the aqueous infusion were shaken with 5 c.c. of acetone in a test-tube. Quebracho wood and Tizerah wood, as well as their dried extracts whether sulphited or non-sulphited, gave a characteristic yellow fluorescence, whilst chestnut wood, oakwood, (myrobalans, valonia, divi-divi, and different kinds of galls showed a characteristic violet fluorescence when the acetone solution was examined in light from a Wood lamp. Mixed pyrogallol and pyrocatechol tans, e.g., oak bark and gonakie, gave a bluish-white fluorescence. D. WOODROFFE.

Fluorescence of sulphite-cellulose extracts and its applications. L. Meunier and A. Jamet (J. Soc. Leather Trades Chem., 1926, 10, 212-213).—The pale violet fluorescence shown by solutions of sulphite-cellulose extracts can be used to detect adulteration in tannin extracts. 25 g. of sodium potassium tartrate are dissolved in 100 c.c. of water, and a 10°/ solution of normal lead acetate is added, drop by drop, with shaking until the precipitate at first formed re-dissolves. 15 g. of the tannin extract (d 1.190) are dissolved in 1 litre of water, 10 c.c. of the solution are mixed with 10 c.c. of the lead solution, a pinch of kaolin is added, and after filtering the clear filtrate is examined with a Wood lamp. A pale violet fluorescence will show with  $> 5^{\circ}/_{\circ}$  of sulphite-cellulose extract. Synthetic tannins cannot be distinguished from sulphite-cellulose extract by this means, but synthetic tannins are too expensive to be used as adulterants. D. Woodroffe.

Properties of shoe leather. V. Area change with relative humidity. J. A. Wilson and E. J. Kern

(J. Amer. Leather Chem. Assoc., 1926, 21, 351—357; cf. B., 1926, 504, 556, 600).—Measurements have been made of the increase in area and water-content with increasing relative humidity of the atmosphere of the 18 leathers described previously. Vegetable-tanned leathers showed an average increase in area of 7% and chrome leathers an increase of 14.4% at 100% relative humidity as compared with zero relative humidity.

D. WOODROFFE.

See also A., Sept., 900, Adsorption and swelling of hide powder (Kubelka and Taussig). 936, Organic chromium salts (Ageno-Valla and Raposio).

Leather dyeing. Salt; Salt and Astrom. See VI.

#### PATENTS.

Production of artificial inorganic-organic tanning materials, and tanning process. F. B. Dehn. From O. Röhm (E.P. 255,313, 19.12.25; cf. U.S.P. 1,569,578, B., 1926, 206).—A tanning solution is made by adding salts of heavy metals, e.g., ferric chloride or chrome alum, and alkalis or alkaline salts, e.g., water-glass, to soaps and one or more suitable colloids, e.g., sulphite-cellulose extract, in proportions to form in an acid solvent clear or colloidal solutions. H. Holmes.

Conversion of sulphite waste liquors into tanning extracts. Preparing concentrated sulphite-cellulose extract containing magnesium compounds. W. E. B. Baker (U.S.P. 1,592,062—3, 13.7.26. Appl., [A] 25.2.22, [B] 16.3.23.—(A) Sulphite-cellulose waste liquor is sprayed into the air to remove gaseous components and to oxidise other components. The product is then treated with an alkaline-earth oxide compound sufficient to decompose the reversible sulphurous acid present, filtered, and the concentrated filtrate treated with sulphuric acid to liberate the organic acids, which are tanning agents, and precipitate the alkaline-earth metal, which is then removed by filtration. (B) A concentrated solution of magnesium sulphate is used instead of sulphuric acid to precipitate the alkaline-earth metal. D. WOODROFFE.

Manufacture of artificial horn from proteins or albuminous substances. F. Schmidt (E.P. 240,174, 18.9.25. Conv., 19.9.24).—To facilitate the kneading and pressing of the material, a neutral, readily volatile substance, such as ethylene chlorohydrin, ethyl lactate, glycol diacetate, alcohol, or acetone is added to the water used for the preliminary steeping. H. Holmes.

Extraction process (G.P. 430,087).—See XX.

## XVI.—AGRICULTURE.

Concentration of carbonates in two Minnesota soil types. P. R. McMiller (Soil Sci., 1926, 22, 75—82).—Examination of the carbonate content of soils at varying depths indicated a definite zone of high carbonate concentration varying from about 18 to 30 in. in depth. The increase in carbonate from the surface downward is abrupt until a maximum is reached, but the decrease at lower depths is more gradual. The moisture

equivalent of a particular soil is not markedly affected by changes in carbonate content. The colour of the soil and the proportion of carbonate present were closely related.

A. G. POLLARD.

Soil studies at the Missouri station. M. F. MILLER, R. BRADFIELD, and F. L. DUDLEY (Missouri Agric. Exp. Sta. Bull., 1925, [228], 77—84; Chem. Abstr., 1926, 20, 2218).—Neutral or acid salts are much more efficient flocculating agents than alkaline salts. Treatments which lowered the Sörensen value of the clay caused liberation of more equivalents of total bases than were absorbed; those isohydric with the clay caused equivalent exchange, and those raising the  $p_{\rm H}$  value of the clay caused the absorption of more equivalents of bases than were liberated. Experimental evidence indicates that the good effects on crops of liming are largely due to the soluble calcium available to the plant rather than to the neutralisation of acidity.

A. A. ELDRIDGE.

Soil studies at the Wisconsin Experiment Station. Anon. (Wisconsin Exp. Sta. Bull., 1925, [373], 41—51; Chem. Abstr., 1926, 20, 2218).—The decomposition products of decaying manure assist in rendering the soil potash-soluble. To obtain the maximum atmospheric nitrogen fixation from lucerne, more phosphate is required than most soils contain. An appreciable amount of aluminium is present in the soil solution only when it is strongly acid or alkaline; the amount could be determined from the reaction. Aluminium ions are no more toxic than the corresponding amount of hydrogen ions. With barley, maize, and soya beans, part of the favourable effect of liming acid soils was due to the precipitation of aluminium.

A. A. Eldridge.

Soil and plant relationships. M. M. McCool and J. D. Romaine (Soil Sci., 1926, 22, 31—34).—The heat of wetting of a number of plant materials (seeds, leaves, roots) was determined, and found to vary with the nature of the plant, season, and previous fertiliser treatment of the soil. The possibility and value of correlating the heat of wetting of plants with soil fertility are indicated.

A. G. Pollard.

Sweet clover in relation to the accumulation, loss, and conservation of nitrates in soil. A. L. Whiting and T. E. Richmond (Soil Sci., 1926, 22, 1—19).—The value of sweet clover as a green manuring crop is demonstrated. Rapid nitrification follows the ploughing-in of the green crop, and ample nitrate can thus be accumulated for a subsequent grain crop. Used as a catch crop, sweet clover conserves much soil nitrate as organic nitrogen, and losses by leaching during rainy seasons are thereby considerably reduced. A. G. Pollard.

Influence of form, soil zone, and fineness of lime and magnesia incorporations upon the outgo of sulphates and nitrates. W. H. MacIntire (Soil Sci., 1926, 22, 21—30; cf. B., 1926, 640).—The rate of leaching out of nitrates and sulphates from soils is used as a measure of increased biological activity following the application of lime in various forms. The records of a four-year period are discussed.

A. G. Pollard.

Residual effects of forty years continuous manurial treatment. III. Ultimate fate and some physical and chemical effects of applied lime. J. W. WHITE and F. J. HOLBEN (Soil Sci., 1926, 22, 61-74).—The rate of decomposition of limestone in field soils follows closely the changes in soil reaction. In the soil studied applications of lime maintained a strongly alkaline reaction. There was, however, a considerably increased proportion of soluble organic matter and nitrate in the limed plots. During the growing season lime tended to reduce the proportion of water-soluble potassium in the soil. The percentage of carbon and nitrogen was greater in limed plots. No measurable effect of lime on the moisture-holding capacity of soil was observed, but the plough draft was markedly reduced. A. G. POLLARD.

Homogeneous carbon disulphide emulsion. W. E. Fleming (J. Agric. Res., 1926, 33, 17—20).—See B., 1925, 940.

Humus in sewage sludge. BACH.—See XXIII.

### PATENTS.

Fertiliser. F. Tharaldsen and E. Lie (Nor. P. 39,805, 22.1.23).—Crude calcium cyanamide is treated with sufficient phosphoric acid to convert the calcium oxide present which is not combined as cyanamide, into soluble calcium phosphate.

C. T. Gimingham.

Treatment of acid-soluble minerals containing potassium. Norsk Hydro-Elektrisk-Kvaelstof-aktieselskab (Nor. P. 40,027, 29.5.23).—The finely-ground mineral, e.g., leucite, is treated with a solution of a salt of urea, preferably the phosphate. A product is obtained, containing potassium in a soluble form, which, on drying, may be used directly as a fertiliser.

C. T. GIMINGHAM.

## XVII.—SUGARS; STARCHES; GUMS.

### PATENTS.

Separating and collecting organic acids and bases from beetroot molasses. Y. Takayama (U.S.P. 1,595,529, 10.8.26. Appl., 30.6.24).—See E.P. 233,196; B., 1925, 568.

Centrifugal machines (E.P. 252,339).—See I.

## XVIII.—FERMENTATION INDUSTRIES.

Rôle of proteolytic enzymes in decomposition of the herring. ALMY.—See XIX.

### PATENT.

Manufacture of non-alcoholic beer etc. J. F. MEYER (U.S.P. 1,593,191, 20.7.26. Appl., 2.6.25. Conv., 9.3.21).—A cereal is malted for 7 or 8 days at about 19° and mashed for about 10 hrs., at 10°, to produce an acid mash. The malt wort is extracted and subjected to a short alcoholic fermentation to produce sufficient acid-reacting products to modify the sugar contents as

to flavour. The fermentation is then stopped by cooling and the product is clarified out of contact with air.

H. HOLMES.

## XIX.—FOODS.

Sweetened condensed milk. IV. Refractometric method for determining total solids. F. E. Rice and J. Miscall (J. Dairy Sci., 1926, 9, 140—152; Chem. Abstr., 1926, 20, 2211).—Formulæ and tables are derived for the determination of total solids in sweetened condensed milk (a) containing 8% of fat, and (b) skim. The method is trustworthy when the sucrose content varies between wide limits provided the fat content approximates to that for which the formula has been prepared.

A. A. Eldridge.

Colour reactions of vitamin-A. T. T. Cocking and E. A. PRICE (Pharm. J., 1926, 117, 175-178).-Of the various colour reactions and modifications for the estimation of vitamin-A in cod-liver oil, the most satisfactory was found to be that given by a solution of antimony trichloride in chloroform. The blue coloration given by a solution of trichloroacetic acid in chloroform was found to be caused by the presence of traces of phosgene in the commercial acid. The colour of the oil and the amount of unsaponifiable matter are useless as criteria of vitamin content. The vitamin activity of cod-liver oils may vary as much as 8:1, Newfoundland oils usually having a much greater activity than Norwegian oils. Details of a method of estimating vitamin activity using a solution of antimony trichloride in chloroform in conjunction with a Lovibond tintometer are given (cf. Carr and Price, A., 1926, 870). Special precautions are not necessary as the reagent is not affected by traces of moisture, but the presence of carrotene vitiates the results. C. O. HARVEY.

Sweet potato starch in cornflour and arrowroot. J. R. Stubbs (Analyst, 1926, 51, 400—402).—The granules of sweet potato starch are very varied in shape and size and often have a hilum. They resemble a mixture of tapioca, sago, maize, and rice starches, whilst some have "facets." Sweet potato starch was recently found in a sample submitted as arrowroot, and during the last seven years one sample of cornflour was found to consist entirely of sweet potato starch, whilst three samples of arrowroot contained sweet potato starch in conjunction with maranta starch.

D. G. Hewer.

Rôle of the proteolytic enzymes in the decomposition of the herring. L. H. Almy (J. Amer. Chem. Soc., 1926, 48, 2136—2146).—The rapid decomposition of immature herrings (used in sardine packing) which, when caught, have food in their stomachs, is due to the softening of the abdominal wall brought about by the trypsin of the pyloric ceca, which is more active and present in greater amount when food is present. This allows the flesh to be attacked by bacteria. The pepsin extracted from the stomach of the fish is most active at  $37^{\circ}$  and at  $p_{\rm H} \ 2.5 - 2.85$ , and is present in greater amount in the stomachs of feeding fish. The trypsin from

the pyloric ceca is most active at blood heat, and at  $p_{\rm H}$  8·5—9·5, but it acts slowly at  $p_{\rm H}$  6·85.

F. G. WILLSON.

"Blowing" of canned fruit due to chemical action. G. W. Monier-Williams (Analyst, 1926, 51, 402—403).—The gas from five blown tins of loganberries with sterile contents was found to consist chiefly of hydrogen. The lacquer on the inner surface of the cans had given way in several longitudinal streaks, and there the tin had been dissolved, leaving the iron exposed. This latter was extensively corroded, particularly along the edges of the seam, presumably by the fruit acids. The total quantities of tin (a) and iron (b) in the contents of the tins, which averaged 550 g., were (a) 16—20 mg. in four cases and 66 mg. in one, and (b) 80—175 mg.

D. G. Hewer.

Freshly ground coffee and "blown" tins. J. W. Black (Analyst, 1926, 51, 403—404).—A quantity of Costa Rica coffee was ground after keeping for 8 days from the roasting time, and the evolution of gas immediately determined. For 200 g. of coffee, 52 c.c. of gas were collected in 1 hr., 90 c.c. in 5 hrs., and 132 c.c. in 48 hrs., and this result is regarded as typical.

D. G. HEWER.

See also A., Sept., 903, Microscopical study of freezing of gel (Hardy). 904, Freezing of gelatin gel (Moran). 905, Colloid chemistry of rennin coagulation (Palmer and Richardson).

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

Use of the potentiometer in the quantitative analysis of alkaloidal solutions. J. C. Krantz, Jun. (J. Amer. Pharm. Assoc., 1925, 14, 294—299; Chem. Abstr., 1926, 20, 2391).—Since the molarity of an alkaloidal salt solution does not affect the  $p_{\rm H}$ , a definite excess of acid added to variable amounts of alkaloidal salts and diluted to a definite volume at which the ionisation of the acid is practically complete will yield solutions of which the  $p_{\rm H}$  is a function of the excess of acid present. Experimental results are interpreted by means of a curve constructed, e.g., for quinine, strychnine, or atropine in 0.1N-hydrochloric acid. The method is valueless for caffeine.

A. A. ELDRIDGE.

Dissociation and volumetric determination of the cinchona alkaloids. C. Morton (Pharm. J., 1926, 117, 168—173).—The equilibria in solutions of diacid bases and of their hydrochlorides are considered, and a determination of the basic constants of the chief cinchona alkaloids from electrometric measurements of the hydrion concentrations of solutions of the hydrochlorides is described. Though a mixture of piperidine and quinoline can be accurately titrated to the half-neutralisation point with a wide choice of indicators, in the case of quinine bromocresol-purple is the only suitable indicator, and the titration error is about 10% unless a

colour standard is used. The optimum titration conditions for the cinchona alkaloids in 0.05N-solutions are given in tabular form.

C. O. Harvey.

Determination of morphine in poppy extracts. C. T. Bennett and D. C. Garratt (Pharm. J., 1926, 117, 149).—Poppy extracts, when mixed with lime and water as in the B.P. method for the determination of morphine in opium, yield a difficultly filterable mixture, and the nature of the filtrate prevents the clean separation of morphine. Extraction with a mixture of cresol and amyl alcohol gave concordant results but was tedious. Alcohol (90%) and isopropyl alcohol were found to be satisfactory solvents, and methods based on the use of the latter are described, the morphine in the extract being determined by modifications of the B.P. process, using methyl-red as indicator in place of methyl-orange. C. O. Harvey.

Assay of extract of aconite. C. W. Cornwell and A. J. Jones (Pharm. J., 1926, 117, 197—199).—In assaying extract of aconite the extraction of the ethersoluble alkaloids in the dry material does not yield results identical with those obtained by shaking out the alkaline aqueous solution with ether, especially when the alkaloids have undergone degradation. Alcohol must be excluded from the assay process. The distribution ratio for crystalline aconitine between water and ether is practically 1:11, and the solubility of the alkaloid in ether (given in the U.S.P. as 1 in 65) is practically confirmed under assay conditions. The alkaloidal residue is easily decomposed.

C. O. Harvey.

Assay of belladonna leaves. C. M. Caines and N. Evers (Pharm. J., 1926, 117, 179—180).—The various methods in use for the assay of belladonna leaves are discussed and criticised, comparative figures being given, and it is concluded that the official B.P. method is the best. Methyl-red is recommended as the most satisfactory indicator for use in the volumetric method.

C. O. Harvey.

Detection and determination of glycerol in tobacco. A. C. Chapman (Analyst, 1926, 51, 382—386). -10 g. of the sample and a few grams of anhydrous sodium sulphate are extracted in a Soxhlet thimble with pure acetone for one day, the thimble left full overnight, and extraction continued for a few hours the next day. The solvent is evaporated, the residue dissolved as far as possible in 20 c.c. of warm alcohol, and water (about 150 c.c.) added until precipitation of the resins appears complete. These are extracted by shaking with petroleum spirit, after which the aqueous layer is run off, the spirit and funnel washed with water, and the main solution and washings are gently distilled. When the volume is reduced to about 30 c.c. the cooled liquid is filtered into a weighed carbonic acid flask. The distillate is evaporated to about 5-10 c.c. and added to the residue, and the whole evaporated to a syrup at about 80° in a current of air until a marked odour of nicotine is apparent. A portion of the crude extract is weighed on a piece of glass into a long-necked flask of the type used in Perkin's modified Zeisel apparatus.

A flask containing a suspension of red phosphorus in water is attached to the long-necked flask, and immersed in a water bath at 60—70°; this serves to retain any iodine liberated. It is connected with a flask containing alcoholic silver nitrate and a guard flask to retain the isopropyl iodide formed. 15 c.c. of hydriodic acid  $(d \ 1.71)$  are placed in the long-necked flask, which is heated in an oil bath at 135-140°, and a slow current of carbon dioxide passed through. If an appreciable amount of glycerol is present precipitation of silver iodide begins in 20 min. and, after complete precipitation, the salt is separated and weighed, and the weight multiplied by 0.392 gives the equivalent of glycerol. For the detection of glycerol, after extraction with acetone for some hours, and removal of resins, an aqueous extract of small bulk is prepared, and traces of water are removed under reduced pressure. The flask is then connected to a small Bruhl apparatus and the distillate boiling at 120—130° at 5 mm. pressure is dissolved in water, the greater part of the nicotine removed with ether, and the remainder of the base precipitated with silicotungstic acid, filtered off, and the filtrate evaporated to small bulk after neutralisation with soda. The water is evaporated, the residue distilled under 2-3 mm. pressure, and the distillate identified as glycerol. Control experiments with glycerol-free tobacco showed the very gradual formation of a very small amount of silver iodide precipitate, and a deduction of 0.06 g. from the weight of silver iodide, corresponding with 10 g. of glycerolcontaining tobacco, should be made. The results were accurate to within 0.1% of the glycerol added.

D. G. HEWER.

Chemical constituents of the oleo-resin and fatty matter of Indian valerian root. K. Bullock (Pharm. J., 1926, 117, 152—157; cf. B., 1925, 825).—On steam distillation valerian root yielded 0.35% of oil (n 1.5025, d<sup>19</sup> 0.9819), consisting principally of a sesquiterpene hydrocarbon plus small quantities of a sesquiterpene alcohol and valeric and formic acids in the combined state. Saturated acids (probably a mixture of stearic and palmitic acids) occur in the free condition, as also do a small quantity of unsaturated acids of the oleic series and a notable quantity of valeric acid. A petroleum spirit extract of the root was found to contain (in addition to the constituents of the steam-distilled oil) arachidic acid, hentriacontane, neutral and acidic resinous material. with some linoleic and linolenic acids. Terpenes or borneol were not found in any of the material examined. C. O. HARVEY.

Automatic continuous percolator [for extraction of drugs]. D. S. RATTRAY (Pharm. J., 1926, 117, 195—196).—The inner tube of a Greiner and Friedrich extraction-tube (having its drug-containing vase removed) is lengthened so that it projects below the outer tube, and both tubes dip below the surface of the extracting liquid in the container. The apparatus is connected to a filter pump via a condenser and water trap, and by carefully adjusting the pump a continuous, steady flow of the liquid through the drug in the extraction tube is maintained.

C. O. HARVEY.

Differentiation of veronal, proponal, and luminal. L. EKKERT (Pharm. Zentr., 1926, 67, 481-482).—If the substances be dropped on to molten sodium hydroxide, veronal gives an acid, rancid odour; luminal gives first a pleasant odour, which becomes piercing, the veronal becoming yellow to yellowish-red; proponal gives first a spicy odour, which becomes piercing. Any of the substances dissolved with salicylaldehyde in alcohol and covered with a layer of strong sulphuric acid gives a red coloration at the zone of contact. If luminal, even in traces, be treated with formaldehyde and strong sulphuric acid, it gives a red coloration, gradually at room temperature and in 1 min. on the boiling water-bath; veronal and proponal give only a yellowish coloration even after some minutes on the water-bath. Known tests are reviewed. B. FULLMAN.

Volatility of benzoic acid. A. F. Lerrigo (Analyst, 1926, 51, 405—406).—Benzoic acid crystals prepared by dissolving 0·5 g. of pure acid in dry methylated ether and in ether saturated with water, spontaneously evaporating the ether, and exposing for 24 hrs. to room temperature, were heated in the oven for observed periods at different temperatures. Smaller and finer crystals were obtained from the dry ether, and the rate of loss increased but slowly up to 50°, being about 0·001 g. per hr. at 40°, but at 70° the loss was more than 0·02 g. per hr.

D. G. HEWER.

Reaction between lead subacetate and phenol. G. A. Medley (Pharm J., 1926, 117, 149—150).—As the precipitate formed by adding basic lead acetate to aqueous solutions of phenol is insoluble in phenol, the conclusion is drawn that its formula is  $(C_6H_5O_2)Pb$  and not  $C_6H_5O.Pb.OH$ . The absence of a precipitate when normal lead acetate is used is probably due to the acidity developed in normal acetate solutions; precipitation with the basic acetate is prevented by adding a few drops of dilute acetic acid. Other phenols give similar precipitates. C. O. Harvey.

Micrographic test for tartaric acid in solutions containing it. M. François and C. Lormand (J. Pharm. Chim., 1926, [viii], 4, 54—61).—The presence of d-tartaric acid may be shown by adding calcium acetate, the resulting crystals of calcium d-tartrate being recognisable microscopically. These crystals form in a solution of d-tartaric acid containing more than 0·15 g. of the acid per litre and less than 1 g. of citric acid per litre if the latter be present. The presence of tartaric acid may be confirmed by testing the precipitate with resorcinol and sulphuric acid.

B. Fullman.

Chaulmoogra oil and its saponification. A. J. Gelarie and F. R. Greenbaum (Amer. J. Pharm., 1926, 98, 411—414).—If treated with the quantity of sodium hydroxide indicated by its saponification value, chaulmoogra oil is incompletely saponified. Saponification is effected by shaking 50 g. of the oil with 14·25 g. of sodium hydroxide in 50 c.c. of water for ½ hr., and leaving the mixture for at least 12 hrs. in the cold. The resulting soap shows 0·7—0·8% of free alkalinity, and

yields a 10% solution. The oil is saponified in ½ hr. if 13 c.c. be shaken with 40 c.c. of 10% alcoholic sodium hydroxide. The soap obtained has alkalinity 1·4%, and yields a 40% solution in the cold. The solution is freed from alcohol by distillation in vacuo at 40—50°, when it congeals; it re-liquefies when the alcohol is replaced by an equal quantity of water, the free alkalinity being then 0·7%. This solution is neutralised with concentrated hydrochloric acid and, on addition of water till the sodium chloride concentration is 0·8%, has a soap concentration equivalent to 7—10% of oil. This solution may be used intramuscularly or intravenously.

B. FULLMAN.

Analysis and composition of commercial glycerophosphates. G. J. W. Ferrey (Pharm. J., 1926, 117, 159—162).—Esterification of phosphoric acid by glycerol at 105-110° yields chiefly the dibasic mono-ester (OH)<sub>2</sub>C<sub>3</sub>H<sub>5</sub>·O·PO(OH)<sub>2</sub>, but at temperatures above 110° condensation occurs to the di-ester which, being monobasic, causes lowering of the proportion of metallic radical in the salts. As normal sodium glycerophosphate is slightly alkaline to phenolphthalein, in order to determine exactly the alkaline or acid impurity a more satisfactory indicator is thymolphthalein, the  $p_{\rm H}$  values of neutral glycerophosphate solutions being found to be within the range 8.5—9.5. After neutralisation to thymolphthalein, the titration of the glycerophosphate is carried out with dimethylaminoazobenzene as indicator. Titration with acid and alkali gives trustworthy results, but only in the absence of organic acids and inorganic phosphates. The products of most English manufacturers were found to be reasonably pure and free from di-ester, but considerable variations in the amounts of water of crystallisation were observed, particularly with the magnesium and the 50% sodium salts. C. O. HARVEY.

Carbon tetrachloride in pharmacy. G. E. Trease and H. Tingey (Pharm. J., 1926, 117, 150—152).—As a solvent for the preparation of certain oleo-resins carbon tetrachloride has no advantages other than that of its non-inflammability, and it appears to have little use as an extracting medium for alkaloidal drugs. Carbon tetrachloride has an advantage over chloroform and iodoform as a colour reagent for phenols in that it gives characteristic reactions with a number of o- and m-substituted phenols, but not with the p-substituted phenols examined. The colours are probably due to dyes of the aurin type.

C. O. Harvey.

Utilisation of ethylene obtained by cracking of petroleum for the production of alcohol. W. Gerr and S. Porov (Neftjanoe Chozjajstvo, 1926, 10, 88—82; Chem. Zentr., 1926, II., 667).—Gas obtained from solar oil by cracking consisted of 54% of methane and its homologues, 12·2% of hydrogen, and 33·8% of unsaturated hydrocarbons. It was passed over calcium chloride, over wood charcoal to remove higher homologues of ethylene, and through an absorption tube kept at 40° and containing glass beads or glass wool, in which a stream of a 1% solution of silver sulphate in 94%

sulphuric acid absorbed the ethylene. The ethylsulphuric acid obtained was decomposed with water and the alcohol distilled off, 33.82 g. of alcohol being obtained from 300 litres of gas. When the gas has not been previously freed from the higher olefines the yield of alcohol is practically the same, and an oil insoluble in water is also obtained. The gas freed from ethylene burns without smoke.

Determination of arsenic in chemicals by the electrolytic method. N. Evers (Pharm. J., 1926, 117, 183—184).—A modified apparatus for the electrolytic determination of arsenic is described, the method of preparing and connecting the cathode being simplified. A list of chemicals, mostly pharmaceutical products, which may be directly tested is given, and special methods of preliminary treatment are described.

C. O. HARVEY.

Genus Mentha. VI. Volatile oil of a strain of Japanese peppermint grown at Madison, Wisconsin. G. C. Jenison and R. E. Kremers (J. Amer. Pharm. Assoc., 1925, 14, 495—498; Chem. Abstr., 1926, 20, 2392).—A strain of Japanese peppermint, the oil of which is normally rich in l-menthol, produced an oil containing 85% of pulegone and a small quantity of l-limonene; neither menthol nor menthone could be found.

A. A. Eldridge.

Obtaining camomile oil. E. KARPATI (Riechstoffind., 1926, 37—39; Chem. Zentr., 1926, II, 660).— An intimate mixture of 3 pts. of camomile flowers and 2 pts. of residues is distilled with steam at a pressure of 7—8 atm. The average oil content of Hungarian camomile is 0.318%.

B. FULLMAN.

Essential orange-flower extract oil. ELZE (Riechstoffind., 1926, 29—30; Chem. Zentr., 1926, II, 660).—On extraction with light petroleum, 1000 kg. of fresh orange flowers gave a product which yielded on treatment with alcohol and distillation 0·165 kg. of oil  $(d\ 0.892, \alpha_{\rm D}-5^{\circ}, {\rm linalyl\ acetate\ 47\cdot04\%})$  containing a number of terpenes, methyl anthranilate, etc. The residues of distillation contain 0·07 kg. of oil  $(d\ 1.0049, \alpha_{\rm D}+1.5^{\circ}, 41.2\%)$  of linalyl acetate). In the residues of distillation of both oils farnesol occurred.

B. FULLMAN.

Essential oil from the flowerheads of Perovskia atriplicifolia, Benth. M. G. Rao (J. Indian Chem. Soc., 1926, 3, 141—147).—The flower heads yield 1% of a light green oil having  $d^{30}_{30}$  0.8943,  $n_p^{30}$  1.4748,  $\alpha_p^{30}$  + 8.53°, acid value 0.2, ester value 30.4 (after acetylation 49.22). The oil contains about 50% of terpenes, chiefly d- $\alpha$ -pinene,  $\beta$ -pinene, and camphene; 15—18% of alcohols and esters (mainly d-borneol and its acetate); and sesquiterpenes,  $\alpha$ -caryophyllene and aromadendrene having been identified. The oil may prove to be a valuable source of d-borneol. G. M. Bennett.

Essential oil of *Boronia citriodora* and the occurrence of citronellol. A. R. Penfold (J. Proc. Roy. Soc. N.S.W., 1925, 59, 35—40).—Steam distillation of the

leaves and terminal branchlets of this Tasmanian alpine plant yields 0.71-0.93% of an oil  $(d^{15}_{15} 0.8814-0.8822, \alpha-2.8^{\circ}$  to  $-3.8^{\circ}, n_{20} 1.4608-1.4611$ , ester value 42.10-69.88, ester value after acetylation 239.46-241.24, acid value 4—5, solubility in 70% alcohol 1:1), containing citronellol (80.38-82.31%), citronellol esters, principally the acetate, with some valerate, d- $\alpha$ -pinene, sesquiterpene, a paraffin (m.p.  $64-65^{\circ}$ ), and small quantities of a phenolic substance and free capric acid. Citral, citronellal, and geraniol are absent. E. H. Sharples.

Essential oils from the leaves of Murraya Koenigii (Spreng.), Murrayaexotica (Linn.), and M. exotica var. ovatifoliolata (Engler). A. R. PENFOLD and J. L. SIMONSEN (J. Proc. Roy. Soc. N.S.W., 1925, 59, 146—155). —In each case the leaves were steam-distilled. M. Koenigii from Dehra Dun yields 0.04% of a pale yellow oil  $(d_{30}^{30})$  0.8711,  $n_{30}^{30}$  1.478,  $\alpha - 18.2^{\circ}$ , acid value 1.1, saponif. value 11.06, saponif. value after acetylation 31.83), containing α-pinene, l-caryophyllene, esters of palmitic acid, and dl-sabinene. On oxidation with alkaline potassium permanganate, dl-sabinene yields dl-sabinenic acid (m.p. 84-85° anhyd.). from Dehra Dun yields 0.01% of a dark, unpleasant smelling oil ( $d^{15}_{15}$  0.9023,  $n_{\rm p}^{22}$  1.496, saponif. value 8.87, saponif. value after acetylation 72.53, insoluble in 10 vols. of 80% alcohol), containing l-cadinene, a second sesquiterpene, and possibly methyl anthranilate. M. exotica var. ovatifoliolata from Queensland yields 0.06-0.15% of oil  $(d^{15}_{15} \ 0.9117 - 0.9126, \ n_{D}^{20} \ 1.499 - 1.5008,$  $[\alpha]_{\rm p}$ -10° to -24.7°, saponif. value 19.37-26.18, saponif. value after acetylation 68.55), containing principally bisabolene and an unidentified sesquiterpene with small amounts of a sesquiterpene alcohol, free palmitic acid, and esters of caprylic acid.

E. H. SHARPLES.

Essential oil of Eriostemon myoporoides (de Candolle). A. R. Penfold (J. Proc. Roy. Soc. N.S.W., 1926, 59, 206—211).—Steam distillation of the leaves and terminal branchlets yields 0.71—0.77% of a mobile, bright yellow oil having  $d^{15}_{15}$  0.8580—0.8789,  $\alpha + 29.2^{\circ}$  to  $+36.75^{\circ}$ ,  $n^{20}$  1.4687—1.4740, saponif. value 4.61—6.38, saponif. value after acetylation 14.59—24.69. It is insoluble in 12 vols. of 80% alcohol (by wt.) and contains d- $\alpha$ -pinene (75—85%), ocimene, a sesquiterpene, ledum camphor, methyl anthranilate, a paraffin of m.p. 64— $65^{\circ}$ , and traces of a phenolic substance. E. H. Sharples.

Essential oils of Melaleuca linariifolia (Smith) and M. alternifolia (Cheel). A. R. Penfold (J. Proc. Roy. Soc. N.S.W., 1926, 59, 306—324).—The leaves and terminal branchlets were distilled in both cases. M. linariifolia gives an average yield of 1.5% of oil having  $d_{15}^{15}$  0.8927—0.8992,  $\alpha + 3.3^{\circ}$  to  $+6.8^{\circ}$ ,  $n_{20}^{20}$  1.4752—1.4780, ester value 1.3—2.67, ester value after acetylation 58.23—82.10. M. alternifolia yields 1.76—1.83% of oil having  $d_{15}^{15}$  0.8958—0.8961,  $\alpha + 6.8^{\circ}$  to  $7.4^{\circ}$ ,  $n_{20}^{20}$  1.4782—1.490, ester value 3.67—7.35, ester value after acetylation 79.36—83.64. The principal constituents of both oils are  $\alpha$ - and  $\gamma$ -terpinene, cymene, cincole (16—20% in the former, 6—8% in the latter),

 $\Delta^{1}$ -terpinenol-4 (naphthylurethane, m.p.  $104-105^{\circ}$ ; nitrosochloride, m.p.  $115-116^{\circ}$ ), sesquiterpenes, chiefly cadinene and the corresponding sesquiterpene alcohol. Small amounts of d- $\alpha$ -pinene and a phenol are present. M. linariifolia contains an unidentified constituent possessing the physical properties of sabinene.

E. H. SHARPLES.

Essential oil of Baeckea Gunniana, var, latifolia (F.v.M.). A. R. Penfold (J. Proc. Roy. Soc. N.S.W., 1926, 59, 351—355).—Steam distillation of the leaves and terminal branchlets yields 0.33-0.74% of a dark brown product (m.p.  $43-47^{\circ}$ ,  $d^{15}_{15}$   $0.9578-d^{26}_{15}$  0.9550, α +  $15.6^{\circ}$  to +  $23.0^{\circ}$ ,  $n^{20}$  1.5038-1.5059, ester value 11.44-23.19, ester value after acetylation 138.38-151.80, solubility in 70% alcohol (by wt.) 1:2-3), containing eudesmol (60%), eudesmene, β-pinene, and a small quantity of an unidentified phenol and valeric acid ester, and a yellow stearoptene,  $C_{13}H_{18}O_4$  (m.p.  $103.5-104^{\circ}$ ).

E. H. Sharples.

Germicidal values of some Australian essential oils and their pure constituents, together with those for some essential oil isolates and synthetics. III. A. R. Penfold and R. Grant (J. Proc. Roy. Soc. N.S.W., 1926, 59, 346—350; cf. B., 1925, 334).—Rideal—Walker coefficients are given. The influence of the degree of dispersion on the magnitude of the coefficient is discussed. For the rapid detection of small quantities of cymene in admixture with terpenes the determination of the Rideal—Walker coefficient is of considerable value, its presence raising the number out of all proportion to the percentage present. The term "biometric test" is applied to this form of analysis.

E. H. Sharples.

Polarimetric examination of cade oils. R. Massy (J. Pharm. Chim., 1926, [viii], 4, 61-65).—Polarimetric examination of oils obtained (presumably by treatment with alkali and steam distillation) from the tars yielded on carbonisation in a closed vessel of several coniferous woods—Juniperus oxycedrus, J. phænicea, J. thurifera, and Pinus halapensis-indicated that those from the trunk of J. oxycedrus, from the root, trunk, and branches of J. phænicea, and from the root and trunk of J. thurifera were lævorotatory, the other root, branch, and trunk oils being slightly dextrorotatory or almost inactive. The levorotatory nature of the product from true cade oil (from J. oxycedrus) does not therefore characterise it, as indicated by Huerre (J. Pharm. Chim., 1926, [viii], 3, 314). Only the oil derived from Cedrus atlantica was B. FULLMAN. strongly dextrorotatory.

Testing of cade oil. R. Huerre (J. Pharm. Chim., 1926, [viii], 4, 65—66).—Polemical. A reply to Massy (cf. preceding abstract). B. Fullman.

See also A., Sept., 902, Alkalinised solutions of salvarsan (Hunter and Patrick). 917, Reduction of carbon monoxide (Elvins and Nash). 918, Catalytic dehydration of methyl alcohol and properties of hydrous aluminium oxide catalyst (Howard); Catalytic actions of various types of reduced copper on alcohols (Hara). 936, Synthesis and hydrolysis

of a glycerolmonophosphoric diester (Bailly and GAUMÉ).

#### PATENTS.

Manufacture of new complex antimony compounds. W. CARPMAEL. From I. G. FARBENIND. A.-G. (E.P. 247,986, 19.2.26).—Antimony pentoxide or pentachloride reacts with a solution of thioglycollic acid to produce an acid, Sb(S·CH<sub>2</sub>·CO<sub>2</sub>H)<sub>5</sub>, the easily soluble salts of which can be isolated by neutralising the acid solution with alkalis or alkaline-earths and evaporating. On acidifying the solutions of these salts, or on evaporating the original reaction product directly, thioglycollic acid is partially split off and another new acid separates, containing 40% Sb as against 20.8% for the pentathioglycollic acid. The second acid also yields water-soluble salts. Both series of salts have specific pharmaceutical properties. A. DAVIDSON.

Preparation of perfumes. I. G. FARBENIND. A.-G., Assees. of A. Eisenhut (G.P. 428,548, 12.9.24).—The fraction boiling above 150° of the product, rich in alcohols, obtained by reducing carbon monoxide under pressure, is hydrolysed; or the reduction product, especially the fraction boiling at 150-200°, is esterified with aromatic acids. B. FULLMAN.

Process for extracting the organs of animals, plants, etc. Maschinebau-Anstalt Humboldt (G.P. 430,087, 22.7.24).—The material is brought into the disperse condition by means of rapidly running mills, a small quantity of a solvent or an indifferent liquid being added at the start, and the dispersion is extracted in the usual way. The process is applicable to the extraction of cinchona bark, wormwood, nux vomica, liver, spleen, dye and tanning woods, crude anthracene, etc. W. G. CAREY.

Pharmaceutical product. [α-Hydroxylepidine dimethylaminoethyl ether.] J. CALLSEN, Assr. to WINTHROP CHEMICAL Co. (Reissue 16,394, 27.7.26, of U.S.P. 1,572,768, 9.2.26. Appl., 18.6.26).—See B., 1926, 464.

Producing a substance adapted to tampon wounds. R. Vogel (U.S.P. 1,593,814, 27.7.26. Appl., 13.2.25. Conv., 19.2.24).—See E.P. 229,644; B., 1925,

Manufacture of [organic] sulphocyano-derivatives. A.-G. für Anilin-Fabr., Assees. of O. Spengler and W. Müller (U.S.P. 1,594,697, 3.8.26. Appl., 2.9.25. Conv., 24.9.24).—See E.P. 240,420; B., 1925, 1012.

Preparation of a solution of cyanamide from commercial calcium cyanamide. J. Breslauer, Assr. to Comp. De l'Azote et des Fertilisants S.A. (U.S.P. 1,595,754, 10.8.26. Appl., 14.11.24. Conv., 20.11.23).—See E.P. 225,206; B., 1925, 785.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Developer containing diaminophenol and pphenylenediamine in bisulphite solution. L. LOBEL and L. J. Bunel (Bull. Soc. franc. Phot., 1926, [iii], 13, 143-147).—The following developer was studied: water, 1 litre, sodium bisulphite solution (d 1.32), 75 c.c., p-phenylenediamine base, 13 g., diaminophenol hydrochloride, 5 g. This mixture gives a developer of normal rapidity, whilst diaminophenol does not develop in bisulphite solution, and p-phenylenediamine develops only in sulphite solution and then very slowly. The high development rate of the mixture is due to the formation of sodium sulphite and p-phenylenediamine sulphite by interaction of the bisulphite with the p-phenylenediamine. The mixture is far more stable in air than amidol developer; the stability approaches that of metol-quinol. It is very sensitive to the presence of bromide ions, whereas diaminophenol is scarcely affected by bromide.

W. CLARK.

See also A., Sept., 920, Influence of adrenaline on the photographic plate (Vollmer). 923, Colloidal aurous oxide. Gold toning of photographic papers. (STEIGMANN).

Dyeing processes using Indigosol O. FRIED-LÄNDER. -- See VI.

### PATENTS.

Changing the light-sensitiveness of photographic emulsions. S. E. Sheppard, Assr. to Eastman Kodak Co. (U.S.P. 1, 591, 499, 6.7.26. Appl., 2.12.25).—A silver halide photographic emulsion is incorporated, at any stage of its preparation, with an organic compound in which a sensitising substance is combined with a group which renders it latent. Subsequently, by addition of a suitable enzyme the sensitiser is released in an active form. For example, a mustard oil sensitiser is prepared by treating the seeds of black mustard with boiling alcohol, extracting the residue with cold water, evaporating the solution to a small volume, acidifying with tartaric acid, and again extracting with alcohol. The syrup obtained, after being neutralised with potassium carbonate, constitutes a latent sensitiser, the sensitising substance, allyl mustard oil, being combined with a monose radical. The syrup is mixed with the silver halide emulsion and the active sensitising agent liberated by addition of an enzyme prepared from mustard seed. R. B. CLARKE.

Preparation of photographic developers. P. Schestakoff and B. Merejkovsky (E.P. 255,925, 24.4.25).—See F.P. 600,532; B., 1926, 566.

Photographic layer. R. Schwarz (U.S.P. 1,594,470, 3.8.26. Appl., 10.9.25. Conv., 12.9.24).—See G.P. 413,217; B., 1925, 787.

## XXII-—EXPLOSIVES; MATCHES.

Chemical stability of nitroglycerin powders. M. Tonegutti (Z. ges. Schiess- u. Sprengstoffw., 1926, 21, 127—128).—In the Abel heat test, acid ballistites give the same results as normal ballistites, whereas according to the Taliani and silvered vessel tests the stability of the former is much less. Cordite M.D. and the Austrian powders M/97 and M/97A show the reverse behaviour, as acid samples of these have a low stability in the Abel test, whilst by the other methods little difference is found between acid and normal samples. This behaviour of acid cordites is attributed to the vaseline present in them. The low stability of acid cordites in the Abel test is attributed to the presence of traces of acetone, which causes the lowering of the heat test value when the powders become acid.

S. BINNING.

Nitrous esters of cellulose. F. Blechta (Z. ges. Schiess- u. Sprengstoffw., 1926, 21, 39-41).—Methods used by previous workers when investigating the possibility of producing nitrous esters of cellulose are criticised. By using Meisenheimer's method for determining nitrites in presence of nitrates it is shown that by the action of nitrous acid on cellulose nitrous esters are formed to an extent that increases with the amount of nitrous acid in the nitric acid used. Eventually, however, with a 30% content of nitrous acid, complete solution of the cellulose residue takes place so that it is impossible to produce nitrocelluloses with any desired content of nitrous ester. In the ordinary process of nitration, nitrous esters are not formed, and even if they were formed owing to abnormal conditions, they would not affect the stability of the boiled nitrocellulose as they would be destroyed during its stabilisation.

S. BINNING.

# XXIII.—SANITATION: WATER PURIFICATION.

Corrosion of iron water mains. C. F. HICKETHIER (Rev. Fac. Cien. Quím., 1925, 3, 73—92; cf. B., 1925, 333).—A discussion of the factors affecting the corrosion of iron water mains and of protective measures, with special reference to the water supply of Buenos Aires.

G. W. ROBINSON.

Modification of the Kjeldahl method for determining organic nitrogen in sewage effluents etc. J. W. H. Johnson (Analyst, 1926, 51, 405).—Large amounts of free and saline ammonia are first distilled off, as usual, followed by distillation of nitrites after acidification with 1 in 4 sulphuric acid. Nitrates are then reduced by addition of 0.5 g. of zinc dust in the form of an ammonia-free suspension and 10 c.c. of sulphuric acid, and the solution is refluxed for 15 min. so that no loss in the intermediate nitrous stage occurs. The process is then completed as usual.

D. G. Hewer.

Anaerobic decomposition of sewage sludge. K. N. Korolkov (Trav. Comm. Epur. Eaux d'Egout (Russ.), 1926, [8], 9—83).—The results are given of laboratory investigations during the period 1918—1921 on the methane fermentation of sewage sludge at the Moscow sewage disposal works. In purifying plant of the Emscher type the normal alkaline fermentation yields a sludge with properties dependent on the course followed by the biochemical decomposition. The chemical character of the process is influenced by the composition of the fermenting material and its relation to the mass of the sludge; the biological and physio-

logical peculiarities of the micro-flora present, which produce the liquefaction of insoluble organic matter and the decomposition of fatty acids into methane and carbon dioxide; and the pronounced buffering of the liquid, the value of  $p_{\rm H}$  being about 7·8. The general results are in agreement with those obtained empirically, such as the importance of thorough mixing, the influence of temperature, etc.

T. H. Pope.

Determination of humus in sewage sludge. Bach (Gesundheitsing., 1926, 49, 19; Chem. Zentr., 1926, II., 630—631).—The manurial value of the sludge does not depend only on nitrogen, potash, phosphoric acid, etc., but also on the organic matter capable of forming humus, on the physical character, and on the abundance of bacteria. The author designates as "sludge-humus" that constituent of the sludge which effects an improvement in the physical character of soils, and in order to estimate this it is necessary: (a) to remove inorganic salts, (b) to remove oils and fats, and (c) to determine the amount of cellulose, coal, and other substances useless for fertiliser purposes and to subtract this from the remaining dry material. 100 g. of the moist sludge are intimately mixed with 1 litre of distilled water, allowed to settle, and the water is decanted; these operations are repeated twice more. The sludge after drying on the water bath is then extracted with ether and the residue weighed. Of this residue, finely powdered, 5 g. are boiled in a porcelain basin for 30 min. with 250 c.c. of 1.25% sulphuric acid. The liquid, after settling, is poured off into a sedimentation vessel, the residue being twice boiled with 100 c.c. of distilled water, and the water decanted. The residue in the basin is boiled for 30 min. with 250 c.c. of 1.25% potassium hydroxide, the liquid being decanted as before. On the next day the sediment in the two vessels is collected, the residue in the basin is added, and the whole is washed thoroughly with hot water, dried, and weighed. This material can be regarded as useless for the soil, the humus organic material having been dissolved out by the acid and alkaline treatment. The content of humus in sewage sludge from the Ems district was found to be 27.4 to 49.0% in fresh sludge, 27.7 to 43.4% in decomposed W. G. CAREY. sludge.

Colorimetric determination of free chlorine in air. Porter.—See A., Sept., 927.

Germicidal value of Australian essential oils and their constituents. Penfold and Grant.—See XX.

## PATENT.

Utilisation of the effluent from fuller's earth and similar factories. E. Maag (G.P. 428,486, 3.3.25).— To the mixture of decomposed clay or earth with the effluent, or to the separated effluent, substances such as wood refuse, peat, sulphite-cellulose, etc., are added, and the mixture is boiled, under pressure if desired. Acetic acid and methyl alcohol are produced and the organic substance undergoes degradation to charcoal, which can be used as an absorbent or as a fuel. W. G. Carey.